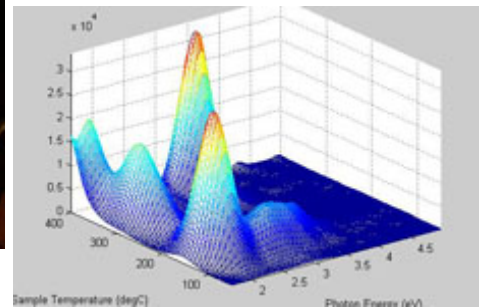
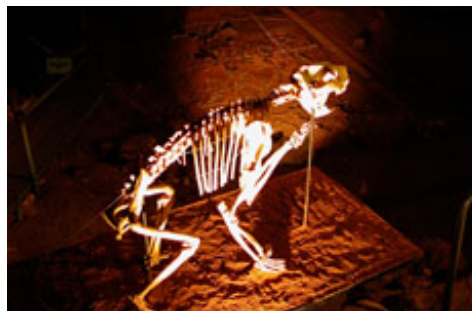


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PROGRAM and ABSTRACTS

ORAL PRESENTATIONS

35 years of struggle with ESR dating of tooth enamel

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In the early 1980s, ESR dating was easy: four additive dose points (5, 10, 20, 40 krad), a straight line fit of any ESR signal that had the audacity to increase its intensity with additional dosing and, for good measure, the dose rate was assumed to be 100 mrad/a. Since then, not only the unit for the radiation dose changed from rad to Gy, but there has also been some appreciation for some of the physical and chemical basics of ESR dating.

Firstly, it turned out that the dose response was not linear. Although this was known since the early 1950s, it was ignored for the sake of applying a ruler to one's data points. Then, for many years it was assumed that the dose response followed a single saturating exponential function. Initially, without any appreciation for the errors involved, later with some reasonable error calculations. In more recent years, it was found that the dose response is actually best described by a double saturating exponential function.

When working on powders, there has been a number of studies that demonstrated that the laboratory induced ESR signals were the same as the signals induced by natural processes: that of CO_2^- radicals (plus some interferences that could easily be removed). This concept fell apart once measurements on fragments were carried out. It turned out that at least three, if not four, different types of CO_2^- radicals were involved in the ESR signal. At least one of them is thermally unstable and at least two can change from one to another.

Dose rate estimations are a little more complicated. Bones and teeth have the unfortunate habit of accumulating uranium over time. This has repercussions for dose rate estimations. Ikeya thus formulated two possible parametric U-uptake models, early uptake and linear uptake. When scanning the literature of the previous millennium (and some significantly more recent papers), one will find that certain U-uptake models were given preference, perhaps to make ESR results conveniently fit the expected ages of the samples. If the ESR results were still too young, a recent U-uptake model could be applied, which basically ignores that there was any uranium in the sample. Then it became analytically possible to routinely measure apparent U-series ages of dental tissues. By combining U-series and ESR data, one could gain some insights into U-uptake. It turned out that neither early nor linear U-uptake were realistic approximations. Still, the combined US-ESR model still assumes a continuous U-uptake process. Using spatially resolved U-series analysis with laser ablation, it turns out that uranium migration processes in bones and teeth are fast (and complicated). Combining spatially resolved U-series analyses with insights from thermally stable ESR signals, one can actually identify domains in tooth enamel that should yield the most promising results. Then, of course, there are the usual problems with reconstructing a realistic environmental dose rate, after all, the original context was removed from the samples that are actually analysed.

There have been arguments that this is all far too complicated, that it is perfectly alright not to carry out any U-series analysis and use the above-mentioned parametric models. Well, rather than producing half-baked results, it is perhaps much better to return to the origin: a linear fit to a small number of data points compensates for the effect of unstable signals, and in spite how difficult and convoluted dose rate estimations are, one often ends up with typical value of 1000 $\mu\text{Gy/a}$ (=100 mrad/a). Such results are probably as often correct (and wrong) as the most complicated approaches.

ESR dating of alluvial quartz : New chronological data for the Intermediate sector of the Creuse river (Centre Region, France)

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Since the first prospections initiated since the early eighties, the Creuse valley appeared to be a favored spot for some of the earliest human occupations in Western Europe [1]. Since the nineties, a systematic sampling has been initiated for the electron spin resonance (ESR) dating of quartz grains extracted from alluvial formations that occasionally fossilized some of the most ancient artifact bearing sites of Western Europe. The Creuse valley belong to the Loire river system and the formation of stepped and embedded terraces systems, divided in three sectors (Massif Cental, Intermediate and Paris Basin sectors) in a strongly tectonic environment appear to take place between 1.7 Ma and 130 ka ago. The results exposed here, focus on the Intermediate system, follow the lead of several previous works [2, 3] and aim to raise our understanding of the establishment and evolution of this fluvial system.

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[3] Voinchet P., Despriée J., Tissoux H., Falguères C., Bahain J.-J., Gageonnet R., Dépont J., Dolo J.-M., 2010. ESR chronology of alluvial deposits and first human settlements of the Middle Loire Basin (Region Centre, France). Quaternary Geochronology 5, 381-384.

Are ESR dose assessments on fossil tooth enamel reliable?

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In ESR dating of fossil tooth enamel, the fitting function used for the evaluation of the D_E value is undoubtedly among the major sources of uncertainty. Although the single saturating exponential function (SSE) has been almost exclusively used since the late 1980's, several recent studies have shown that the behavior of the radiation-induced ESR signal is better described by a double saturating exponential function (DSE) [e.g. 1], whereas the SSE shows instead many limitations. This is actually more consistent with our current understanding of the nature and composition of this signal that is dominated by several types of CO_2^- radicals [2].

To address the question whether older (published) dose estimations have to be disregarded as unreliable, a series of dose recovery tests was performed using several proto-historic fossil teeth that were laboratory dosed to ~200 and ~1,500 Gy to present equivalents of Early to Late Pleistocene samples. Several fitting functions and data weighting options were tested, and results indicate that a combination of a DSE with data weighed by the inverse of the square intensities systematically provides reliable D_E results. Nevertheless, the SSE may produce consistent results as well if the D_{max} value is correctly constrained.

In order to obtain statistically significant results and determine exactly in which conditions the SSE could be used as a fair approximation of the DSE function, we ran additional computer simulations with thousands of randomly generated dose response curves. The results show that reliable doses can be obtained for preset dose values <1,000 Gy using 9 additive, exponentially distributed dose points with maximum dose values in the range of up to ten times the preset dose. Higher preset dose values (>1 kGy) require modified maximum doses and improving measurement precision is crucial.

In this work, we will present a synthetic overview of the results detailed in two recent studies [3,4] and provide as well some recommendations to ensure reliable fitting results with the SSE and DSE functions.

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[3] Duval, M. 2015. Evaluating the accuracy of ESR dose assessment of pseudo-Early Pleistocene fossil tooth enamel with dose recovery tests. *Radiation Measurements*, 79, 24-32.

[4] Duval, M. and Grün, R. submitted. Are ESR dose assessments on fossil tooth enamel reliable? Submitted to *Quaternary Geochronology*.

ESR dating of sea-floor hydrothermal barite: Contribution of ^{228}Ra to the dose rate

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Dating of submarine hydrothermal activities is an important issue in order to evaluate the sea-floor hydrothermal ore [1] and to investigate the evolution of the biological systems sustained by the chemical species arising from hydrothermal activities [2]. After a preliminary work [3], it has been shown that ESR (electron spin resonance) dating of barite (BaSO_4) is one of the promising methods for this purpose. The obtained ESR ages are roughly consistent with the U-Th ages of co-existing sulfide minerals [4], however, when comparing with the ^{226}Ra - ^{210}Pb and ^{228}Ra - ^{228}Th ages, the ESR ages are larger than these ages [5]. Although it is possible that the age differences are due to repeated cycles of formation of the deposits as already discussed [5], investigating the contribution of the doses from the extinct ^{228}Ra would still be necessary.

The equivalent dose to be obtained by the ESR measurements with the additive dose method is expressed by

$$D_E = \int_0^T D(t) dt$$

When both the parent and daughter nuclei are radioactive, the formula is written as

$$D_E = \lambda_1 N_{1p} e^{\lambda_1 T} \left\{ \frac{1}{\lambda_1} (Q_1 + Q_2 \frac{\lambda_2}{\lambda_2 - \lambda_1}) (1 - e^{-\lambda_1 T}) - \frac{Q_2}{\lambda_2} (1 - e^{-\lambda_2 T}) \right\}$$

where the Q_i is the dose rate given by nuclide i , and T is the age to be obtained. Considering the contribution of ^{226}Ra and ^{228}Ra , the ESR ages of barite were obtained for such young samples having ^{228}Ra . In the presentation, from such examples, the contributions to the doses from ^{228}Ra and daughter nuclides will be estimated for older samples which currently having no ^{228}Ra .

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Single-grain optical dating of K-feldspar: investigating the existence of post-IR IRSL standardised growth curves

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The post-IR IRSL (pIRIR) signals from individual K-feldspar grains were investigated using samples from different regions of Asia (China and Russia), Europe (France) and Africa (Kenya). The dose response curves (DRCs), or growth curves, for individual grains were measured using a multiple elevated temperature (MET) pIRIR procedure [1] with successive IR stimulations made at 50, 100, 150, 200 and 275 °C, and a two-step pIRIR procedure with the initial IR stimulation made at 200 °C and the pIRIR stimulation made at 275 °C. The MET-pIRIR and pIRIR 275 °C signals yielded indistinguishable results, but we observed a large variation in the shape of the DRCs for the sensitivity-corrected signals (L_x/T_x) of individual grains. Such grain-to-grain variation can be largely reduced, but not completely eliminated, if the DRCs are normalised using the signals from one of the regenerative doses—the so-called regenerative-dose normalisation or ‘re-normalisation’ method [2, 3]. We found that the remaining grain-to-grain variability in re-normalised DRCs can be explained by the irreproducibility of the single-grain IR laser measurements and, potentially, some unknown factors associated with the application of the pIRIR measurement procedure to individual grains. Based on these results, we propose that a standardised growth curve (SGC), and possibly even a global SGC (G-SGC), can be constructed for individual grains of K-feldspar. To test this proposition, we compared the D_e values obtained for individual grains using a full single-aliquot regenerative-dose (SAR) DRC sequence and the SGC method (on the same set of grains), and found that the two approaches yielded statistically consistent D_e estimates. The establishment of a single-grain SGC would greatly reduce the time required to measure the D_e values for individual grains of K-feldspar from old samples, as many grains are measured individually and a large range of regenerative doses are typically delivered in each SAR cycle.

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Investigating the effect of test dose and stimulation temperature on post-IR IRSL SAR protocol for rock slices

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Optically stimulated luminescence (OSL) is increasingly applied to the dating of rock surfaces. This technique has been predominantly developed for quartz and K-feldspar as target minerals. However, there is no practical way to separate these minerals from consolidated rocks without losing the grain-size dependent dosimetric information. There is little information about the performance of the post-IR IRSL (pIRIR) SAR measurement protocol for rock slices. In this study, we investigate the influence of different size of test dose and the first stimulation temperature on pIRIR₂₉₀ signal from granitic rock slices.

There does not seem to be any dependence of the shape of the dose-response curve on either the size of the test dose or the sequence of the regeneration doses. The natural pIRIR signals from these saturated slices from the inner parts of the rocks lie below saturation when 50°C is used as stimulation temperature for the first IR measurement; a higher first IR stimulation temperature (e.g. 200°C or 250°C) seems to overcome this problem. The dose recovery test on the naturally bleached surface slices support this point.

The origin of the medium OSL component in West Australian quartz

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The optically stimulated luminescence (OSL) of West Australia coarse-grained sedimentary quartz was investigated. Measurements of OSL and TL after different experimental conditions were recorded and used to investigate the nature of the medium component of the OSL decay. These observations allowed us to determine that the medium component observed after heating at 260 °C is a by-product of the production of the fast component. It was concluded that both have as their original source the 325 °C TL trap. We were able to show that the 170 °C TL trap plays a surprisingly important role in the production of the medium OSL component. During a blue stimulation, when the 170 °C trap is initially empty, some of the electrons evicted from the fast OSL trap get retrapped in the 170 °C trap from where they are evicted giving rise to the medium component. In freshly irradiated sample, the medium component originates directly from the 170 °C trap and the retrapped charge provides a negligible contribution. The major conclusions from the study are that: 1) the kinetic properties of the medium component are dependent on the properties of both the 170 °C and 325 °C TL traps and 2) the 170 °C TL trap plays unexpected roles in quartz OSL production at elevated stimulation temperatures (e.g. 125 °C). The findings are described in detail in [1].

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A bleaching experiment on weathered aeolian sands with iron-rich coatings

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Optical dating of ancient burial sands relies on the assumption of full zeroing of grave infill sediment. Recent experimental grave digging in a lunette dune at Willandra Lakes that has been host to 33 Pleistocene burials found that bright sunlight was insufficient to fully bleach loose sand that had received up to 4 h exposure [1]. Single grain OSL analysis of the backfilled sediment revealed that $\leq 1\%$ of grains had been fully zeroed, which was insufficient to clearly distinguish the disturbance event from the effects of bioturbation, biological mixing, or other sources of D_e variation. Here, we test the effect of iron-rich coatings on the zeroing of sediments from the same weathered, Pleistocene sediments with an environmental dose of 26 Gy, and from an adjacent aeolian dune dating to the Last Glacial Maximum (18 Gy) which exhibited less weathered sediment. Samples from both locations were divided, with one half cleaned of its iron-rich coatings by a 48 h treatment of HCl. Both sets of samples were then exposed to natural sunlight for 10 s, 100 s, 1000 s, and 10,000 s, and processed using standard procedures; i.e. treatments were applied to remove contaminant clays, carbonates, feldspars, organics, heavy minerals and acid soluble fluorides. Single-grain equivalent dose (D_e) values were determined using the modified single-aliquot-regenerative dose (SAR) protocol. The effect of exposure time on D_e in both clean and natural sands is examined for each location.

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Testing TT-OSL single-aliquot protocols and their applicability to long-range and high-dose sediment dating

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Seven quartz samples were dated using TT-OSL single-aliquot protocols. Different data processing was used to find dates using the TT-OSL signal, the commonly named Basic Transfer (BT-OSL) signal, and the TT-OSL minus the BT-OSL signal. Different normalisation, signal integral, and background subtraction methods were also tested against known independently gained ages.

Six of the samples used came from the south-east of South Australia (SESA) stranded dune sequence situated from Robe to Naracoorte, a series of parallel stranded dunes made of calcium carbonate and quartz [1], aging from 0 to 800 ka. The dunes have been dated a number of times by independent dating methods, including luminescence and non-luminescence methods. Due to their similar origin and deposition type, samples from these dunes were used to test TT-OSL age determination on progressively older samples.

The seventh sample was from Baldina Creek, near Burra, South Australia. This sample was taken from a megafaunal layer thought to be 40-100 ka, and electron-spin resonance (ESR) dating of diprotodon teeth in the area found them to be 50-70 ka old [2]. This sample was chosen as it is relatively young, but due to the area's natural dose rate of 2.5 Gy/ka cannot be dated using conventional OSL.

Analysis of the SESA samples proved that the TT-OSL protocol used was not suitable for dating samples beyond 200 ka, as although the regenerative dose curve did not saturate at higher equivalent doses, the age was not accurate beyond this point. Using the TT-OSL minus BT-OSL signal provided dates that most accurately corresponded to the independent dates gained previously. Using normalisation and background subtraction methods that maximised the amount of signal used increased the accuracy and precision of the result. Using only the first 0.1 s of signal provided more accurate ages than using the first 0.5 s of signal. Using the data processing method that gave the best result in the SESA samples provided an age for the Baldina Creek site that corresponded well to the ESR age within errors.

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Potential of 2D-modeling for beta dose rate characterization in heterogeneous samples

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The beta dose rate characterization can be difficult in the cases of samples presenting heterogeneity at the millimeter scale, like poly-mineral rocks or sediments containing significant coarse fractions. Modeling these samples should require analysis of the composition and structure in the three dimensions, using tomography or separation of the size fractions. The 2D-modeling allows extrapolating dose rate average value and distribution in heterogeneous isotropic samples directly from 2D mapping and analysis. This modeling is available via new features of the DosiVox software [1] and could be a convenient solution for usual dose rate modeling of complex samples of luminescence and ESR dating.

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Measuring beta-source heterogeneity in sediment using the Medipix2 pixelated semiconductor detector

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Optically stimulated luminescence (OSL) measurements are commonly performed on individual grains of a disaggregated sample, allowing for the evaluation of grain-to-grain variability in equivalent dose. In contrast, dose rate measurements are by necessity performed on bulk samples. However, without grain-specific dose rates, or an appropriate single-grain dose rate distribution, equivalent doses derived from single grains can be difficult to interpret. Establishing grain-specific beta dose rates on intact sediment samples would lead to OSL ages of greater accuracy and precision.

Here we explore the use of the Medipix2 detector to estimate the source distribution for 2D mapping of ionising radiation emitters in intact samples. The Medipix2 detector contains an array of 256x256 pixels, each 50x50 μm^2 with its own preamplifier, discriminator and digital counter, and a total sensitive area of about 2 cm^2 . When operated in the "Time-over-Threshold" mode, the time for which the pulse exceeds a set threshold is recorded and used to estimate the total deposited energy of the incident particle, such that an energy spectrum can be acquired. Particle types, their deposited energies and spatial distributions can be identified by applying a simple clustering algorithm to the recorded image frames and classifying the recorded tracks. Initial measurements have been performed on a relatively radioactive biotite slice, with successful detection and 2D mapping of the beta and gamma emissions from the decay of potassium-40. We will present some of our preliminary results, discuss the challenges of high spatial and energy resolution spectroscopy in low-activity sediment slices, and assess the feasibility of making grain-specific dose rate determinations on intact archaeological and geological sediments. The latter requires measurements at the scale of single grains preserved in their natural context to permit the evaluation of the beta particle energy and spatial distribution of radioactivity at the same spatial resolution, and on the same intact samples, as the single grains used for equivalent dose estimation. The data obtained will be used for accurate modelling of beta dose rates in intact sediment samples using Monte Carlo simulations and interpretation of OSL measurements.

Potential applications of the DosiVox software for dosimetry.

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Over the last two decades, there was an increasing interest for modeling particle-matter interactions in situations relevant to paleodosimetric dating methods. These numerical calculations followed the pioneering works of Kirkegaard & Løvborg (1980), and made use of large code libraries like MCNP or Geant4.

However, up to now, such simulations remained confined to a few laboratories, and only researchers having skills in programming dealt with these problems. Moreover, these calculations were most of the time limited to « classical » cases (for instance, to compute the absorbed dose fraction in grains) whereas many situations would require numerical simulations.

The objective behind the development of the DosiVox software is to provide to the dating community an easy-to-use tool for tackling a large variety of dosimetric questions. This software is based on a C++ code and uses the Geant4 library functionalities. A user-friendly interface allows to model numerous situations and produces a text file (specific to the case study modelled by the user) used to feed the previously-compiled DosiVox code. No skill in programming is then required.

The DosiVox software is freely downloadable on the website of the IRAMAT-CRP2A laboratory at : <<http://www.iramatcrp2a.cnrs.fr/spip/spip.php?article144>> and comes with an extensive user's manual.

We will present the basics of the DosiVox software as well as a series of applications.

Kirkegaard, P. & Løvborg, L. (1980) Transport of terrestrial gamma irradiation in plane semi-infinite geometry. *Journal of Computational Physics* 36, 20-24.

A new approach for beta dose rate evaluation in heterogeneous rocks: implications for ESR and Luminescence thermochronology

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Accurate beta dose rate evaluation is crucial in electron spin resonance (ESR) and luminescence dating. Beta particles are emitted by minerals containing radioactive elements (U, Th and K), while their attenuation factors decrease exponentially with increasing quartz grain size [1].

The averaged concentrations of radioactive elements are used for beta dose rate evaluation in most ESR/luminescence dating studies on sedimentary quartz where grains are considered spherical. However, dating of rocks is more challenging, as quartz crystals in rocks can have any shape and size and the spatial distribution of radioactive elements may be very heterogeneous depending on the rock's texture. For example, quartz surrounded by K-feldspars or micas will receive higher beta doses than those located close to Na-feldspars. After sample preparation it is impossible to gain any information about the heterogeneity of the original average beta dose rate. Therefore, detailed analyses of mineral distribution and initial quartz grain size are required to ensure a realistic beta dose rate evaluation.

We employed a series of different techniques on several granitic rocks from Northwestern Indian Himalaya. First, X-ray computed tomography (CT) was used to visualise and quantify the internal structure of the samples in three dimensions (3D), while QEMSCAN provided a direct way to identify and quantify minerals in two dimensions (2D). High resolution in situ LA-ICP-MS analysis was then performed to evaluate the radioactive elements concentrations in the different mineral phases. Finally, beta dose rates were calculated with "DosiVox" program [2]. Our preliminary results indicate that a 2D simulation from QEMSCAN data provides the best solution for obtaining realistic beta dose rate assessments.

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Distributed fibre dosimetry using optically stimulated luminescence

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An optical fibre device has been developed for the purpose of detecting ionising radiation using optically stimulated luminescence. Characterisation of glass materials has been performed, after which optical fibres were fabricated for experiments to demonstrate sensing of ionising radiation.

Fluoride phosphate glass was tested for its capability to sense ionising radiation, primarily using the mechanism of optically stimulated luminescence. The characteristics of the material were determined using a combination of spectroscopy, and thermally and optically stimulated luminescence tests. The sensitivity to ionising radiation was improved by introducing dopant ions into the glass; doping of fluoride phosphate glass with Tb^{3+} was found to increase the intensity of the optically stimulated luminescence response by an order of magnitude, from 7.56×10^6 counts/g/Gy to 100.7×10^6 counts/g/Gy.

Optical fibres were fabricated from fluoride phosphate glass using the extrusion method for fibre preform manufacture. The fabrication process was optimised in each of the extrusion, preform processing and fibre drawing stages to achieve optical fibres with loss of between 0.5 - 1 dB/m for undoped fibres, and between 1 - 4 dB/m for Tb^{3+} -doped fibres. Optical fibres were used for ionising radiation sensing experiments, where the optically stimulated luminescence response was measured following both beta and X-ray irradiation. Following a dose of 14.6 ± 0.5 Gy, optically stimulated luminescence signals were observable using optical fibre lengths of up to 2.6 m, with an integrated OSL intensity of 44.1 ± 13.0 counts.

Silica glass was also tested as an alternative material to perform optical fibre measurements using optically stimulated luminescence. The material was characterised and optical fibres were fabricated with a loss of 0.5 dB/m. Following a dose of 15.5 ± 0.5 Gy, optically stimulated luminescence signals were observable using optical fibre lengths of up to 8.6 m, with an integrated OSL intensity of 385.7 ± 43.4 counts.

The general characterisation and applicability of NaCl for retrospective dosimetry purposes.

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Since the terrorist attacks of 9/11 there has been a heightened emphasis on National Security in general and increased interest in 'as found' materials for retrospective dosimetry purposes. The results of the observations of 20 samples of NaCl were presented at LED2011 [1] and were focussed on the suitability of the 590nm emission band for retrospective dosimetry purposes. This current study is an extension of the initial results, including samples from Africa and increasing the scope of samples over those than initially covered.

Having assessed samples 1-20 as generally suitable for retrospective dosimetry purposes, the performance of all 46 samples was compared by analysing the resultant luminescence after an applied dose of 5 Gy. Individual 3DTL plots (showing TL intensity as a function of both temperature and wavelength) are presented for all 46 samples. Additional information relating to source (country of production, source and form of NaCl), production process (direct information or native crystal shape and by deduction the production temperature), chemical composition (admitted additives such as anti-cake materials and assays for a limited number – both published on packaging and performed directly on samples) and the OSL intensity and light sums of the residual 590nm and 340nm TL emissions are also included.

The samples were initially grouped according to the prevalence and relative intensity of the 590nm vs 340nm emission bands and a further sub-group formed from those samples manifesting a dominant broad peak at ~100C centred at 400nm.

In general what was found is that the 340nm emission is present in all samples that had measurable TL emissions at 5 Gy dose level and that the range of intensities varied over an order of magnitude. On the other hand, 590nm emissions were not present in all samples but where present ranged over 4 orders of magnitude and often completely swamped the 340nm emissions in many samples. They were most likely to be produced in sunny locations (Australia, northern Africa, southern USA, Mediterranean sea) using solar crystallisation pans rather than mined rock salt or production processes using higher level heat (Vacuum Pan, Grainer processes) favoured in cooler climate countries. Samples formed in ephemeral salt pans (#46) and efflorescence deposits on building materials (#11, #17) were seen to be lower in luminescent intensity due in part to the size and number of grains suitable for luminescent testing.

Using a series of samples from one supplier (Cheetham & Co, Australia) where the production parameters were known it is shown that the 590nm emission may be enhanced by a factor of 6 at least by the kiln drying of solar crystallisation pan salt crystals for ~2 hours @ 300C. This may account for the brightness of some samples in the 590nm band produced by this method. As noted by Spooner et al 2009, the 590nm emission is desensitised with subsequent heating cycles above 200C. This hints at structural features which enhance the 590nm emission being created in the initial kiln drying process but which are subsequently destroyed in later TL related heating above 200C.

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Alpha and beta radiation sensing using scintillating polymer fibres

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Detection of alpha particles emitted by radioactive isotopes is a challenging task, with a range of diverse applications, from monitoring radiation fields in the mining and medical processing industries, to dose validation for cancer therapies in the medical field. The use of optical fibres made from scintillating plastic as a sensing platform enable a miniaturized device capable of performing direct, real time small-scale and in-vivo measurements and operates on the phenomenon of radiation induced scintillation within the material. In addition, such a dosimeter would be cheap to produce, safe, tissue equivalent and is not subject to interference from electrical or magnetic fields. Preliminary results show the response of the scintillating plastic to alpha and beta radiation and outcomes of initial fibre fabrication trials.

Site formation processes and their influence on chronological records from cave deposits: examples from Naracoorte Caves

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Interpreting site chronologies from cave deposits can be challenging due to complex sedimentation processes and re-working. A useful approach is to treat datable materials as taphonomic entities in the same way as bones and other preserved materials. Unraveling the full range of processes that have contributed to site formation is important to understanding inherent biases and limitations in the preserved record.

This paper will explore the various taphonomic pathways for organic and inorganic materials preserved in Quaternary cave deposits, using examples from Naracoorte in South Australia. Specifically, the importance of cave structure, entrance type and within-cave environment to reconstructing site chronologies will be discussed.

Luminescence dating of Neolithic hearth in Loess Plateau: New age control for stone-walled settlement of Shimao

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Shimao Site is the largest stone-walled settlement site of Longshan period to Xia Dynasty in northern China. The stone-walled city site locates in the northern part of Loess Plateau, and it is on the southern margin of the Mu Us desert (Ordos Plateau). The well-preserved watchtowers, auxiliary structures, "Imperial City Terrace" and rammed earth gate and walls unearthed from the inner and outer city indicates the forming of the earliest earthen and stone-built social and military system in northern China.

Previous date of Shimao site is provided by two carbon14 samples. A wooden beam embedded in the city walls yield a radiocarbon age of 2200-2040 BC and a lime plaster at a house foundation yielded an age of 1940-1780 BC. Our research focus on determining ages of the hearths in house foundations. The abundance of available dated house foundations and high sensitivity of heated hearth samples allows temporal and spatial analysis of the site. Dates of the hearths would provide us a comprehensive chronology of prehistoric human settlement in Shimao site. The results offers interesting insights into the development of early civilization and the formation of state in China.

Koonalda Cave, Nullarbor, South Australia- Archaeology and Issues of Optical Dating

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Koonalda Cave is a deep karst cave, situated on the Nullarbor Plain of South Australia and is well known for its archaeological evidence for ancient art and flint mining. The archaeological research was initiated by Dr Alexander Gallus in the late 1950s and he continued his investigations into the 1970s.

Gallus argued for great antiquity of the site- that is approximately 30,000 years old. This suggestion was supported by a single radiometric date of 31,500 from one of his three excavation trenches. However, a series of radiometric dates on samples from this and other trenches produced a chronology for the site, of between 22,000 and 14,000 years old. Gallus found it increasingly difficult to convince the profession that an age of 30,000 years or more for the site was acceptable.

In the early 1990s, a series of sediment samples were taken from Koonalda Cave and from a nearby rockshelter, named Allen's Cave. Allen's Cave is not a deep cave, but a modest overhang exposed in the base of a collapsed doline.

Optical dating was performed on all samples. Those from Allen's Cave proved suitable but those from Koonalda Cave were found to suffer significant discrepancies between optical and calibrated ¹⁴C dates (Roberts et al 1994). The result was interpreted as evidence for the unsuitability of luminescence dating in deep karst systems. However, the dating for Allen's Cave did extend human occupation of this region back to circa 40,000 years. This paper presents an overview of the archaeology revealed at Koonalda Cave and the issues in optical dating of sites in deep karst systems.

Mountains, monsoons and megafans: Role of luminescence dating in defining human responses to climatic changes in the Nile Valley.

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The Nile is the longest river in the world and its basin contains a generous slice of global climatic history. The work we report here is based upon 167 OSL ages, 271 (mostly AMS) ¹⁴C ages, 10 cosmogenic nuclide ages and more than 110 stratigraphic sections (pits, boreholes) studied between 2005 and 2015, supplemented by analyses of stable isotopes, vertebrate and invertebrate fossils, prehistoric artefact assemblages, heavy minerals and grain size composition.

Beds of highly organic mud or sapropels seen in marine sediment cores retrieved from the floor of the eastern Mediterranean Sea are long thought to have accumulated during times of exceptional Nile fluvial discharge, but with no direct proof. Our work in the Blue Nile, White Nile and main Nile valleys has for the first time revealed a sequence of extreme flood episodes dated by OSL synchronous with sapropel units S5 (124 ka), S4 (102 ka), S3 (81 ka), S2 (55 ka) and S1 (13.5-6.5 ka). During times of extreme floods over the past 125 ka, wide distributary channels of the Blue Nile flowed across the Gezira alluvial megafan in central Sudan and transported a bed load of sand and gravel into the lower White Nile valley. This record of Nile floods shows a precessional signal and reflects episodes of stronger summer monsoon and more northerly seasonal movement of the Intertropical Convergence Zone linked to times of higher insolation in northern tropical latitudes.

The eastern Sahara is very sensitive to climate change. During the last glacial-interglacial cycle it ranged from a savanna with rivers and lakes to an extreme desert with active sand dunes. We have mapped and dated a last interglacial lake in the White Nile valley. This lake was at least 650 km long and up to 80 km wide, with an area more than 45,000 km² in area, equal to the largest freshwater lakes on Earth today. A wetter eastern Sahara would have facilitated the migration of *Homo sapiens* out of Africa over 100 ka ago.

During the prolonged drought between 25 ka and 15 ka, the climate across North Africa was cold, dry and windy. The great lakes that fed the Ugandan headwaters of the White Nile dried out, flow in that river was reduced to a trickle, dunes migrated across the floor of the White Nile, blocking its channel, the Main Nile ceased to flow all year round, and humans abandoned the Nile Valley. The abrupt return of the summer monsoon 15 ka ago brought intense summer rainfall to Uganda (where the lakes rose and overflowed into the White Nile) and to the Ethiopian headwaters of the Blue Nile. During the summer wet season, the alluvial plains along the Egyptian Nile were flooded and over the next few thousand years several metres of fertile silt and clay were deposited. At first the Nile flood plains in Egypt were too poorly drained for early farmers to use them, although plants and animals had been domesticated in the Fertile Crescent of the Near East by 11 ka. Mesolithic groups hunted, fished and gathered wild plants in the Nile Valley until about 9-8 ka, when cattle were first domesticated in Egypt. As the Nile cut down into its bed and the flood plains became better drained, Neolithic farming began in earnest, based on wheat and barley. Irrigation was well under way by 5 ka. Several major urban civilizations arose in Egypt and Nubia (northern Sudan), with the Nubian Kerma culture being one of the most powerful and wealthy. A severe drought 4.2 ka ago led to the demise of the Old Kingdom in Egypt, but farming continued to flourish in northern Sudan for a further thousand years, until climatic desiccation, partial drying out of the Nile, and invasion from Egypt brought about the final collapse of the once flourishing Kerma urban civilization.

Optically stimulated luminescence dating of sediments from Manas Lake and its paleoenvironmental implication

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Manas Lake is a closed lake basin located in western Junggar Basin in northern Xinjiang, northwestern China. At present, the regional climate is arid and is controlled by the Westerly wind system. The evolution of Manas Lake was controlled by both climate changes and tectonic activities. During the Early Quaternary, the Paleo-Manas Lake formed in a tectonic depression in the western Junggar Basin, covering a much larger area than the modern Manas Lake. Due to tectonic movements later, a few rivers from the north stopped flowing to Manas Lake. Together with the influence of climate changes, the lake level dropped and it broke up into several lakes in the Quaternary, including the modern Manas Lake.

Various studies have focused on reconstructing the Quaternary environment of Manas Lake, in order to understand the past climate changes in details. In these studies, a robust chronology is required. Recently, optically-stimulated luminescence (OSL) dating has been successfully applied to date lacustrine sediments to study the past changes in Manas Lake. In these studies, OSL ages of lacustrine sediments from northwestern Manas Lake provide evidence of high lake levels at > 20 m above the present lake bed before ~66 ka and during 38-27 ka ago. Sediments from paleoshoreline terraces at 18 and 11 m above lake bed were dated to be ~300 and <100 years respectively. However, in these studies, no evidence of a high lake level between ~27 ka and few centuries ago were found. Such sedimentary hiatus existed in both sides of the lake.

In this study, lacustrine samples from the upper paleoshoreline on the rarely studied southeastern side of the lake will be dated by the OSL method, in order to establish the size of the age gap beyond the Little Ice Age (LIA). Preliminary results suggest that the lower layer is much older than the upper layer deposited during the LIA. When compared with the northwestern side of the lake, the size of the age gap is larger in the southeastern side, despite the same elevation. This may indicate that there was uplift in the northwestern side of the lake relative to the southeastern side.

An exposed vertical sedimentary section from the northwestern Manas Lake was also studied. It consists of two fine lacustrine sand layers with a brown silty or clayey layer in between. The section provides important evidence of paleoenvironmental changes in the area. OSL dating on the two sandy layers will help us constraint the timing of such changes. OSL dating of quartz and post infrared-infrared stimulated luminescence (pIRIR) dating of K-feldspar were applied to the same samples of same grain size, in order to attempt for an isochron dating. The results will be presented and discussed.

Chronology of formation and evolution of Red Dune sands (teri sands) of South India and its palaeo-environmental implications

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Red colored coastal dunes (locally known as “teri sands”) occur in the coastal plains of Tamil Nadu, south east coast of India between latitudes and longitudes of 8°00’ to 9°30’ N; 77°18’ to 79° 00’ E. Typically the dunes in the east coast have an elevation of 25-30m a.s.l and has a thickness of aeolian accretion up to 7m. The section located in the west coast has an elevation of 75m a.s.l. and the dune sand thickness reaching up to ~12m. To understand their evolution, chronology and environment of deposition, trenches up to 12 m deep reaching up to the basal gravel/ calcareous sandstone, were excavated.

Optically stimulated luminescence (OSL) dating, geochemical and textural analyses, and magnetic susceptibility measurements were carried out. General stratigraphy of the region comprises; basal carbonate cemented sands/ unconsolidated colluvial gravel, overlain by layers of fluvial/ lacustrine origin, including gravelly sand layers with marine shells, calcrete layers with land snail fossils and rhizoliths etc. These layers indicate a phase of landscape stability and varied environmental condition that formed a paleosurface with marked unconformity. Over this paleosurface, aggradation of aeolian red sand has occurred.

Optical dating of six stratigraphic sections comprising 39 OSL ages from various stratigraphic horizons provides the chronology of sediment aggradation events. The sequence can broadly be divided into a basal, carbonate cemented sands/ gravels deposited by fluvial/ colluvial agencies during 50- 30ka and, aeolian red sands deposition during 25- 10ka, 7-4ka, and 100- 40 years. Cessation of sand deposition at 10k reflects the control of sea level by limiting the sand supply.

Climate controls the weathering of sand. The sand succession in the area has recorded two intervals of humid climate between 17 and 14 ka, as evidenced from the sand units of increased clay, higher organic matter and enhanced magnetic susceptibility values. The lower part of the section comprised finer sands with little organic matter, reduced clay content and magnetic susceptibility, indicating a drier period with reduced vegetation and absence of soil formation during ~25 ka. The paleosurface is dated at 30 ka and this marks a change of a humid interval from 50-30ka to a more arid regime. Optical ages also show that the aeolian sand aggradations in South India are contemporaneous with similar deposits occurring in the SE coast of Sri Lanka.

Using OSL dating to examine controls on channel migration, Murrumbidgee River, NSW

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In inland Australia measuring the rate of channel migration remains a challenge, as channel migration generally occurs too slowly to be accurately measured using methods employed elsewhere such as time-lagged aerial photos. Reliable historical documents generally don't span more than the past 100 years. Dateable carbon is scarce and often has an uncertain relationship to the depositional event of interest. Dendrochronological methods are also generally unreliable due to an absence of clearly defined annual growth rings in Australian riparian vegetation. Here description is provided of single grain optically stimulated luminescence dating of floodplain sediments to measure channel migration rates on the Murrumbidgee River. OSL, stratigraphic and topographic data is presented for 8 sites distributed along the ~900 km of freely meandering stream channel from near Wagga Wagga downstream to Balranald. Simple steps to ensure contamination free down-hole sampling are described. Results are interpreted with reference to the pioneering work of Hickin and Nanson on the Beatton River [1] examining fundamental controls on channel migration rate. Other potential controls are introduced as being significant in the case of the Murrumbidgee River. This work shows channel migration to be a significant contributor to allochthonous carbon in the Murrumbidgee River.

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Establishing a chronology of lacustrine sediments for Lake Eyre (South Australia) using multi luminescence dating methods

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Lakes with large catchments within the arid and semi-arid climatic zones of Australia can provide good archives of Quaternary climate change in the southern hemisphere. However, one of the greatest difficulties for fully deciphering these terrestrial records is age determination due to the often old age of units and their fine-grained nature. Luminescence dating can provide a promising tool which has a potential to extend the upper timing range for lake sediments beyond the last glacial-interglacial cycle. In this study, we aim to establish the first robust chronology for lake-floor sediments of one of the key sites of lake studies in Australia, using luminescence dating. Lake Eyre, located in the arid southern central Australia, is the largest playa on the continent and the terminus of the 1.14×10^6 km² Lake Eyre basin, which integrates the climatic influences of tropical and sub-tropical air masses, the main drivers of Australia's climate. Previous chronological studies have focused on the paleoshoreline deposits, which were used to derive extreme wet intervals and the shrinking process of the lake since Marine Isotope Stage (MIS) 5 [1, 2]. The bottom sediments of the lake document the start of lake deposition (interpreted to be 'deep-water' facies), and can provide more information for palaeoclimate reconstruction. However, these floor sediments have never been effectively dated due to the antiquity of the sediments. In this study, we report our recent work on luminescence dating of the lake-floor sediments adjacent to the most thoroughly described stratigraphic section of Lake Eyre: Williams Point in Madigan Gulf [3].

Eleven samples were separated from a ~3.5 m core from the lake-floor. Fine-grain (4-11 μm) polymineral, medium-grain (45-63 μm) quartz and coarse-grain (>63 μm) K-feldspar were extracted as chronometers. Different luminescence dating protocols were tested and applied, including thermoluminescence (TL) dating for fine-grained polymineral, traditional single-aliquot regeneration (SAR) protocol for quartz, post-infrared infrared-stimulated luminescence (post-IR IRSL) protocol for K-feldspar, and thermally transferred optically stimulated luminescence (TT-OSL) protocol for quartz. Due to the fact that most of our samples are older than the upper age limit of the traditional SAR method, we focused more on the latter two dating protocols. The suitability of different dating protocols was assessed and measurement procedures optimized based on a set of luminescence characteristic tests and inter-comparison between methods. Using combined dating results, a lake-floor chronostratigraphy is presented and correlated with paleoshoreline records [2] and will form the basis for further palaeoclimatology work. This study highlights the importance of using multi dating approaches to extend the dating range for lake sediments in Australia.

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High-resolution luminescence dating of a coastal sediment sequence from the South Yellow Sea

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The Yangtze River delta from eastern China is one of the largest tide-dominated deltas in the world. It connects the shelf-coastal sedimentary systems with the Changjiang River and is thus a natural laboratory for studying the land-sea interactions and paleoenvironmental changes. Since the 1980s, numerous research projects have been conducted to reconstruct the stratigraphic framework with aim to understand the relationships between delta evolution and sea-level change. Among these works, the chronologies were mainly based on radiocarbon dating. However, this method might be problematic due to reworked deposition or old carbon contamination. Optically stimulated luminescence (OSL) dosimetric method has shown its unique value in dating Quaternary sediments and it has been successfully applied to coastal sediments during the past decades. However, the partial resetting of OSL signal is an inevitable issue for water-laid sediments. In this study, we utilize luminescence dating technique to date sediments from the upper 50 m of a 150 m-long core (13YZ07) in the north of Yangtze River delta. Firstly, the suitability of luminescence dating was examined by a series of tests, including preheat plateau tests and dose recovery tests performed on the extracted quartz mineral of coarse-grain (CG) fraction. Secondly, the bleaching conditions of samples were detected by two methods. We employ the recently developed feldspar post-IR IRSL (pIRIR) protocol to identify the resetting of different luminescence signals with various bleaching rates. In addition, statistical approaches were also used to analyze the distribution of equivalent doses. Furthermore, the ages derived from quartz OSL signal and feldspar pIRIR signals stimulated at temperature of 150°C and 225°C (pIRIR150 and pIRIR225) were compared. The results show that our samples were generally well bleached prior to burial and suitable for luminescence dating. Finally, we build up a high resolution chronostratigraphy on the basis of 28 quartz OSL ages. The chronological framework of 13YZ07 core reveals a striking sedimentation rate, i.e., relatively successive sedimentation since the mid-Holocene, and a sedimentary hiatuses between the early Holocene and LGM (~27 ka). We speculate that the deposition history in the study site is probably linked to sea-level rise, delta initiation and incised-valley-filled process in Yangtze River delta.

OSL chronostratigraphy for loess deposits from Tyszowce - Poland

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The Polish loess record constitutes an excellent high-resolution archive of climate and environmental changes, providing evidence for the interaction between accumulation and erosion of aeolian and fluvial sediments during the Middle and Late Pleistocene. In Poland, loess and loess-like formations occur in the southern part of the country, mostly in the south Polish uplands, i.e. in the Lublin, Sandomierz, and Cracow Uplands. In addition, such deposits are found in the forelands and foothills of the Carpathians and Sudetes. Investigated loess profile is placed in the eastern part of Poland on the Volyn Upland, close to the Ukrainian border. On this area loess formation reaches 20 meters high and our profile was discovered on the last active brickyard in this area. At present, luminescence dating provides the greatest number of chronostratigraphic data concerning loess deposits. It is one of important tools for determining the age of loess from the last 100-150 ka, which in turn contributes to the creation of loess chronostratigraphy and its correlation with a continuous maritime climatostratigraphic record. In this work we present luminescence ages of loess from the last glacial cycle in SE Poland (up to about 100 ka), obtained in the Gliwice Luminescence Laboratory. 21 samples were collected from the investigated loess profile in Tyszowce (23°42'E, 50°37'N). Combined infrared (post-IR IRSL) and blue light stimulated luminescence dating were applied to the polimineral fine grains (4-11µm) and medium grained quartz fraction (45-63µm). Obtained results for both fractions are very similar for all lithostratigraphic units.

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Can we date the earthquakes of the last millennium along the Altyn Tagh Fault (ATF) via IRSL signals?

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The Altyn Tagh Fault (ATF) is one of the most prominent active strike-slip faults of the India-Eurasia collision system. Fresh features of surface rupture, which are attributed to seismic events taking place in the last millennium, were identified at several sites on the central part of ATF. Accurate chronology of these earthquakes would help us understand the spatial relationship of them. In this study, we investigated the feasibility of using post-IR IRSL (infrared stimulated luminescence) signal from potassium feldspars (KF) to date sediments related to the earthquake events of last millennium by employing modern sag pond deposits with expected equivalent dose (D_e) of 0 Gy, because the luminescence signal of coarse grain quartz is so dim that the quartz SAR dating behaves badly. The non-fading characteristic of post-IR IRSL₁₇₀ signal measured following a prior IR stimulation at 110 °C was verified by employing the D_e plateau test with respect to integration interval and IR stimulation temperature together. The post-IR IRSL₁₇₀ D_e values of 2 mm aliquots were measured for three samples with different sedimentary textures. The median of D_e distribution of well sorted, stratified sag pond deposits is consistent with the minimum D_e values inferred by minimum age model (MAM) and finite mixture model (FMM), while the median is significantly overestimated, compared with the minimum D_e values from MAM and FMM for badly sorted deposits. MAM D_e values of 0.6 – 0.8 Gy of all three samples are consistent with the unbleachable residual dose previously reported for post-IR IRSL signal at similar temperature for well-bleached samples. We'll also measure such a residual dose for our samples subjected to the laboratory and natural sunlight bleaching. Such an intrinsic unbleachable component needs to be properly corrected when events of last millennium are dated in this area. Otherwise, the post-IR IRSL₁₇₀ age would be overestimated.

Applying OSL dating for geological mapping and mineral exploration in regolith dominated terrains; examples from the Eastern Musgrave Province, South Australia, and the East Wongatha area, Western Australia

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With about 70% of Australia's land surface covered by transported regolith material a major challenge is exploring effectively and efficiently through regolith cover of various types and thickness. The Geological Surveys of South (GSSA) and Western (GSWA) Australia assist in this challenge by compiling and providing geological maps that have information about regolith materials and landforms as an integral part of their products. These maps provide a context for exploration in regolith-dominated terrains, show the distribution and abundance of various regolith materials available for soil surveys and geochemical sampling, help in designing and interpreting geochemical surveys and interpretation of results, and also provide a first step to understanding landscape evolution of an area. Therefore, regolith units have also to be put into a stratigraphic context and hence determining the age of these units is important. However, there is a distinctive lack of age control on regolith units throughout Australia. Hence, GSSA and GSWA have recently applied optically stimulated luminescence (OSL) to assist in unravelling the age of several regolith units.

Two examples are presented here: GSSA has used OSL dating to develop a chronology of aeolian and alluvial deposition in the Eastern Musgrave Province in order to understand and reconstruct the landscape evolution and regolith material dispersion during the Quaternary. GSWA has applied OSL dating to examine changes in soil geochemistry of transported regolith with depth and the age of regolith in order to better understand the formation of geochemical anomalies found in near-surface regolith, and the rate of regolith accumulation and anomaly formation in the East Wongatha area.

OSL and TL sensitivities of quartz in a marine sediment core as a source tracer and paleoclimate proxy

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Marine sediment cores are one of the best archives for climatic reconstructions. Still, defining the siliciclastic sediment source is a crucial and non-trivial issue to appropriately reconstruct continental climate based on marine cores. This is usually assessed based on radiogenic isotopes (e.g. Nd), magnetic properties (e.g. ARM/IRM) and major elements (e.g. Fe/K), but these proxies may also be influenced by the weathering type and intensity as well as sedimentary processes. Here, we propose that Optically Stimulated Luminescence (OSL) and Thermoluminescence (TL) sensitivities of quartz can be used as a proxy for sediment source. We measured the OSL and TL sensitivities of 120 samples from an 8m long marine sediment core collected 70km offshore Northeastern Brazil coast and deposited over the last 30,000 years. Additionally, 16 samples collected along the Parnaíba River, the most important river supplying sediments to the core site and three samples of coastal deposits were also analyzed. The core samples (0.5g) were treated with H₂O₂ and HCl to respectively remove organic matter and carbonates. Then, samples were diluted in 5ml of acetone after centrifugation and mounted in aluminum discs (three discs per sample) for OSL and TL measurements. Four drops of the solution (acetone + sediment) was used per disc to keep similar sample size. All measurements were performed in a Risø TL/OSL DA-20 reader equipped with a built-in beta source (dose rate of 0.136 Gy/s), bialkali PM tube and Hoya U-340 filters (290-340 nm). OSL measurements comprised bleaching using IR and blue LEDs (90% power for 100s), irradiation with a 30Gy beta dose, preheat at 200°C for 10s, IR stimulation (100s at 125°C) and blue LEDs stimulation (100s at 125°C). The sensitivity of the fast OSL component was determined through the integration of the first second of light emission, which was normalized by the total OSL sensitivity, represented by integration of the total (100s) OSL decay curve. After the OSL measurements, the aliquots were heated up to 250°C to eliminate the natural TL signal. Then, a 30Gy beta dose was given again and the TL curve was obtained heating the samples until 250°C in a 5°C/s rate. The sensitivity related to the 110°C TL peak was calculated through the integral of 80-120°C interval of the TL glow curve. Samples were not heated over this temperature to avoid sensitivity changes. The variations in sensitivity (particularly the TL 110°C peak) observed along the core correlates well with climatic changes described for Northeastern Brazil, with periods of increased continental precipitation (e.g. Heinrich Stadial 1, 2 and the Younger Dryas) showing higher quartz sensitivity. In our presentation, we will discuss the applicability of OSL and TL sensitivities of quartz from marine sediments collected off Northeastern Brazil as a source tracer and paleoclimate proxy for the last 30 ka.

Thermoluminescence geothermometry-Otway Basin revisit

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Ypma and Hochman (1991) studied samples from boreholes of the Otway Basin (Australia) thermoluminescence (TL) and found that the natural TL peak temperatures and sensitivities of TL peaks after laboratory irradiation showed a relationship with vitrinite reflectance and apatite fission track analysis.

In this study, 8 samples from borehole of Fergusons Hill were used. The samples were stored over the last few million years at stable thermal steady state conditions, thus providing a natural annealing experiment. TL signals from separated quartz were studied as geothermometers with a newly developed measurement protocol (Tang and Li, 2015). The results have demonstrated that

- (1) the apparent TL ages reflected the thermal history of the samples.
- (2) Several thermometers can be obtained from one set of TL data of a sample. Which have different 'closing' temperatures (53~85°C), but the same thermal history.
- (3) The thermal gradient can be perfectly predicted from the TL results.

[1] Ypma, P.J., Hochman, M.B.M., 1991. Thermoluminescence geothermometry – a case study of the Otway Basin. *APEA Journal*, 312-324.

[2] Tang, S.L., Li S.-H., 2015. Low temperature thermochronology using thermoluminescence signals from quartz. *Radiation Measurements*. (in press)

Study of low temperature thermochronology using Thermoluminescence signals from K-feldspar

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The thermoluminescence signal is a result of the competing effects between the trapping of electrons induced by irradiation and the decay of trapped electrons by heating. Hence, the luminescence dating method has great potential in low temperature thermochronology. For this method to be applied, the first priority is finding an appropriate protocol for equivalent dose (D_e) measurement. Although the kinetic order and mechanism were not very clear, the K-feldspar has advantages in D_e measurement due to its bright signals and excellent reproducibility.

A multiple aliquots additive thermoluminescence (MAA-TL), a multiple aliquots regenerative thermoluminescence (MAR-TL), a multiple aliquots additive isothermal thermoluminescence (MAA-ITL), a multiple aliquots regenerative isothermal thermoluminescence (MAR-ITL) and a single aliquot regenerative dose isothermal thermoluminescence (SAR-ITL) protocol have been evaluated by D_e determination. It was observed that the D_e values of ITL are higher than those from TL under same heating temperature. The results were consistent except for an underestimation of D_e value at 295 °C ITL of the MAR-ITL protocol. This is resulted by the residual dose of regenerative dose after ITL heating.

The MAR-TL is suitable because it has advantages: 1) D_e values of different TL peaks can be measured in one run. 2) Each TL peak represents a thermal chronometer. 3) TL dating methods were well established over last 50 years. 4) TL method uses shorter machine time. 5) The thermal stability of TL signals can be identified in the temperature of TL glow curve. In the MAR-TL protocol, a cutheat (preheat) of 200 °C was used to remove the low temperature peaks. D_e values at temperatures of 250-290 °C were used for thermochronological study. The SAR-ITL was suitable because it has advantages: 1) Only small amount of materials is required for D_e measurement. 2) No normalization is required between aliquots. 3) The sensitivity change can be corrected. 4) Isothermal temperature is lower than conventional TL, so that the atmosphere of TL oven is not critical. 5) Low black body radiation during the ITL measurement. 6) It is possible to conduct component analysis to the ITL signal. In SAR-ITL protocol, temperatures of 235 and 255 °C were selected for the isothermal TL measurement. A cutheat (preheat) to the temperatures of 245 and 265 °C were used to remove TL signal from lower temperature peaks, respectively. The integration of 10-20 seconds ITL counts was used for D_e calculation. The MAA-TL and MAA-ITL protocols were not suitable for the thermochronology study, because of the uncertainty caused by the extrapolation of growth curves.

POSTER PRESENTATIONS

Early to Late Holocene Fluvial Activity of the Palar River, Tamil Nadu

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Southern peninsular rivers of India such as the Palar and the Cauvery rise in the Western Ghats and reach the Bay of Bengal in the east. Unlike the Cauvery River; the Palar River has a very wide channel breadth near the coast and no delta formation before reaching the Bay of Bengal. The paleochannels of these big rivers have preserved Late Quaternary-Early Holocene sedimentary records that can be used to decipher sediment depositional shifts that have taken place in response to palaeoclimate and environmental variations. Till date no relevant and comprehensive work has been carried out to explain the occurrence of paleochannels, causes for the very wide breadth of the Palar River channel formation and river migration. In this paper we present the plausible causes for these geomorphic changes. Using Digital elevation model (DEM), we identified three major paleochannels north of the present day Palar River channel. Hill shading of SRTM DEM of the Palar basin highlighted the existence of the paleochannels due to the elevation difference between the main channel, its banks and the flood plains. Several trenches were excavated in the main Palar channel and three trenches in various paleochannels. Textural analyses revealed that the sediments are coarse grained, poorly sorted, negatively skewed indicating high velocity deposit as compared to the paleochannels that are fine to coarse grained, moderately to poorly sorted, and negatively skewed and are largely colluvial wash deposits. The OSL dates of the paleochannels lithosections of the Palar river basin ranges from 10.09 ka to 1.6 ka. OSL dating of the paleochannel indicate that the shifting of the rivers in the region since the early Holocene period driven due to the Mulki-Pulicat fault axis (MPA). This MPA marks uplift due to the sea-floor spreading along the Indian Ocean bridge system. This has produced many geomorphological changes and in the drainage pattern. Rivers in the area gradually changed their courses at various stages leaving the old river channels. The regional slope towards south east supported by the active Mulki-Pulicat fault axis (MPA) trending east west at 13°N has caused the migration of the Palar river. This process of tectonic activity further separated the north flowing rivers further northward and south flowing rivers to south. Moreover, pulses of high intense NE and SW monsoons causing paleofloods widened the Palar channel in its lower reaches.

Optical dating of Holocene sediments from the Changjiang (Yangtze) delta, China

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The Holocene sedimentary record from the Changjiang delta, eastern China provides an opportunity to document the evolution of coastal depositional systems and understand the variations of the East Asian summer monsoon. However, the chronology of the sediments is limited or questionable due to the limitation of the experimental technique and complicated sedimentary environment. In this study, two cores from Nantong and Taizhou in the Yangtze River Delta Region were taken to test the precision and accuracy in the optically stimulated luminescence (OSL) dating of sedimentary quartz. Seven samples from Taizhou and nine samples from Nantong were collected from the cores, and the SAR protocol was applied to the medium- and coarse-grained (45-63 μm and 90-125 μm) quartz. A preheat at 200 °C for 10 s and a cut heat at 160 °C for 0 s were used, followed by stimulation of the quartz OSL samples using blue light at 125 °C for 40 s. The results showed that the medium-grained quartz ages and coarse-grained quartz ages are generally consistent with each other within the limits of experimental errors and the stratigraphical order. In addition, we explored the potential of this approach by comparing OSL and ¹⁴C age estimates. Holocene sea-level changes will be discussed base on the new OSL ages.

OSL dating of an ancient runoff farming system in the Wadi Baqa', Petra, Jordan: Preliminary results

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In 2010, the Brown University Petra Archaeological Project located several agricultural terraces in the Wadi Baqa', located slightly north of the city of Petra, Jordan. With the identification of certain archaeological features, it became apparent that the wadi system had supported a large ancient runoff farming system, and features such as barrage dams, terrace walls and rock cut channels indicated significant functional integration between the many archaeological remains. A multidisciplinary approach was used to engage in a more informative and in-depth analysis of the farming system, including OSL dating, to determine the scope of the system's use and depositional history.

This poster presents preliminary OSL results of 14 sediment samples taken between 2012–13. Early analysis indicates that the system was constructed upon Palaeolithic deposits somewhere around 2500–2000 years ago (which corresponds with archaeological observations of Roman/Nabatean activity in the area), and further indicate a long use history for the system with evidence for activity spanning into the early Islamic and modern periods.

Late Quaternary deformation at Bharawain Anticline along Himalayan Frontal Foreland Fold, NW Himalaya, India

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The Bharawain Anticline (BA) represents a ~100 km long frontal foreland fold in the NW Himalayan foothills. Himalayan Frontal Fault is limiting the anticline at its southern margin, makes this area associated with the principal zone of tectonic displacement at the northern boundary of the stable Indian plate with a rapid rate of convergence. The deformation history of the area in connection with the Himalayan orogeny plays a vital role in understanding the morphological development of this landscape and its associated tectonics. Great magnitude earthquakes are reported from Himalayan Foot Hills in the recent past during 1897, 1905, 1934 and 1950 and falls within the mesoseismal zone. The chronologies of Quaternary sediment aggradations and incision cycles and their inter-relation between climate and tectonics in the area are not known. A variety of methods including structural mapping, elevation profiling and optically stimulated luminescence (OSL) dating was employed to understand the terrace formation and tectonic history of the area. Samples for OSL dating were collected from the sand units overlying the Upper Siwalik formation and terrace sediments for the determination of terrace abandonment. Geomorphologically, the anticline is bounded by River Chakki at the Northeast and Beas at the southeast with number of major channels flow across the anticline transversely. The Chakki Ghad River occurs as an antecedent stream with two levels of terraces was formed over the course of time and tectonic evolution. The stratigraphy of the area includes generally consolidated lithounits from Upper Siwalik formation, which is overlain by loose fluvial terrace sediments.

Dating the strath surface along the abandoned fluvial terrace of Chakki Ghad River has shown the bedrock incision or uplift in the area. The field mapping indicates that the axial trace of the BA strike towards NE-SW and plunging toward NW ~20. Field investigation suggests that the Chakki Ghad River has incised the nose of the plunging BA fold and subsequently the river aggraded to form terrace. OSL ages of the samples indicate that the BA initiated post 70 ka and grew episodically and activity was ceased at 4.5 ka and thus abandonment of younger river terrace occurred after 4.5 ka. OSL ages of samples collected from the hinge portion of the folded Siwalik and post Siwalik deposits indicate that the bedrock incision (due to the growth of BA) at a rate of 2 mm a⁻¹. Previously calculated uplift rate or growth rate of JA at the hinge portion shows 3.4 ±0.28 mm/yr (Thakur et al., 2014), but BA yielded less growth rate suggesting either (i) along strike variation of growth rate of fold may be related to displacement along HFT or (ii) controlled by geometry (or position) of the different fold models. To address this issue we need to quantify the brittle displacement along HFT, hence detailed study of paleoseismic investigation is warranted.

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Partitioning of convergence in Northwest Sub-Himalaya: estimation of late Quaternary uplift and convergence rates across the Kangra reentrant, North India. International Journal of Earth Sciences, 103(4), 1037-1056.

Optical dating of paleoearthquakes along the Ms7.4 1985 Wuqia earthquake surface ruptures at the NE margin of the Pamir Syntaxis

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The August 23, 1985 Wuqia Earthquake (Ms7.4) ruptured the east segment of the seismically active Pamir Front Thrust at the leading northeastern edge of the Pamir Syntaxis of the Tibetan Plateau, produced a 15-km-long surface rupture (Feng et al., 1988; 1994), and destroyed the old Wuqia town. Clear fault scarps were observed from the airphotos taken in 1970's along the 1985 surface rupture, indicating activity of this thrust fault. Data on the recurrence period of such devastating earthquakes is a vital input for seismic hazard evaluation and risk assessment in this region.

The paleoseismologic trench has been excavated across a 1.5 m high monocline scarp formed in 1985 mainshock and exposed sediments are dominated by poorly sorted, sandy muddy matrix supported pebbles, interbedded with sandy clay or muddy sand bearing with gravels, which are interpreted as proximal alluvial fan deposits, with red clay, likely derived locally from the south limb of the Quaternary Mingyaole anticline to the west of Kashgar. All the layers in the trenches are folded and faulted, north dipping with a tendency of steepening downward. Based on the onlapping structure, dip angle change, and fault cutting relation, we identified three paleoearthquake events except the 1985 event. Optical dating samples collected from both sandy clay and muddy sand are being analyzed using Single grain quartz and 2mm small aliquot quartz technique. The preliminary results indicated the quartz of the trench samples had typical TL peak, the fast component of OSL signals and ignored recuperation. But only 1.5-3.6% proportion of grains in a sample having a detectable OSL signal. Three events before 1985 event occurred before 5.2 ka, between 3.8-4.2 ka, and after 2.8 ka respectively. The recurrence period is about 1.0 ka, it seems that at least 1-2 events are missing in this trench.

Slip rate estimate on the Gyaring Co Fault in central Tibet: constraints from Late Quaternary OSL dating

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The long-term slip rates along primary faults across Tibet can give answer to a fundamental question that what is the scale of the eastward extrusion, which is a key piece of information to evaluate and improve the deformation models for Tibetan Plateau. The Gyaring Co Fault (GCF) is a major active right-lateral strike-slip fault striking $\sim 300^\circ$ for a distance of ~ 240 km in eastern central Tibet. Initial studies suggested the GCF is one of important secondary strike-slip faults along Karakorum-Jiali Fault Zone (KJFZ) which has been reported as the southern boundary to accommodate the eastward extrusion with relative high Quaternary slip rate (10-20 mm/yr) constraining from geomorphologic estimated ages [1]. However recent studies carried out on central Tibet suggested GCF is actually a normal-striking fault [2, 3], and even may with a relative low slip rate both at timescales of ten years [2, 3] and half later Holocene [4]. Since direct ages of the faulted landforms were scarcely obtained, average long-term slip rate of the GCF remains considerable controversy [1, 2, 3, 4].

In this study, we use Optically Stimulated Luminescence (OSL) dating technique to determine the ages of displaced geomorphic markers along the GCF combined with field topographic survey to make constraints on Late Quaternary slip rate on the GCF. At Aeqingsang ($N31.46^\circ$, $E87.63^\circ$), two Late Quaternary alluvial fans show a consistent right lateral offset by ~ 500 m. Our preliminary results of OSL ages for the fans both are about 60 ka. These data suggest an average slip rate of ~ 8 mm/yr along the northern central segment of GCF since the Late Quaternary. Meanwhile, we detected a series of smaller right-lateral displacements across three Holocene alluvial fans and six rills (12, 26, 36, 58, and 65 m) at Quba ($N31.52^\circ$, $E87.48^\circ$), and eight Holocene rills in alluvial fans and one Paleo-shoreline (6, 9, 12, 20, 30, 36 m) at Aruo ($N31.21^\circ$, $E88.25^\circ$). Those smaller displacements suggest the GCF was active during the Holocene. Hence, the slip rate might be relative slow during the entire Late Quaternary period compared with initial studies [1], which is consistent with the Global Positioning System (GPS) data and Synthetic Aperture Radar Interferometry (InSAR) data [2] and latest results [4].

The relative low slip rates of averaged since Late Quaternary in our study are unlikely to support the rigid models, and in contrast, to suggest a limited eastward extrusion, which turn out to back the continuum models that active deformation is distributed in central Tibet [5].

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High resolution single-grain OSL dating of Welsby Lagoon, Queensland: Bridging Australia's MIS3 chronological gap

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The perched wetland of Welsby Lagoon, North Stradbroke Island, Queensland, contains a continuous, high-resolution sedimentary record spanning at least the last 120,000 years. This unique palaeoenvironmental archive provides important insights into last interglacial climatic conditions in Australia, rapid climate variability during the last glacial cycle, and climate stability in the lead up to continental-wide megafaunal extinction between 40,000-50,000 years ago. The ability to utilise the Welsby sedimentary record for regional palaeoclimatic reconstructions is dependent on establishing accurate and precise chronologies. However, much of the preserved palaeoenvironmental record lies beyond the upper limits of radiocarbon dating. In this study, we explore the feasibility of establishing reliable age constraint on the Marine Isotope Stage 3 and basal sections of the Welsby Lagoon record using OSL dating of individual quartz grains. We present single-grain dose recovery tests, D_e datasets and depositional ages for 10 samples spanning 3.5-8.5 m of the core sequence. The single-grain D_e results are used to assess adequacy of signal resetting prior to deposition and the potential for post-depositional mixing via bioturbation. We also derive comparative OSL ages using 'synthetic aliquots' to assess the feasibility of applying traditional, multi-grain aliquot OSL dating in this lacustrine setting.

The final OSL chronologies are compared with XRF elemental (ITRAX) data to establish climatically diagnostic trends in mineral composition, dust fluxes and charcoal abundance through time. The results of this study provide new insights into the nature, timing and extent of climate change in eastern Australia during the last glacial cycle.

Luminescence dating study of a loess section in Tajikistan, Central Asia

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Loess sequences in Central Asia provide useful archives for understanding Quaternary climate history over the Asian continent. Previous absolute age determinations of loess in central Asia were obtained mainly by thermoluminescence dating and infrared stimulated luminescence dating. Given the new protocols developed in the recent years, these results are now technically considered to be neither accurate nor precise. In this study, samples taken from Khonako loess-paleosol sequence in southern Tajikistan, Central Asia were measured using a variety of protocols which involve the use of fine-grained quartz extracts and polyminerals, including single aliquot regeneration (SAR), multiple aliquot regeneration (MAR) and isothermal TL for quartz as well as polymineral post IR-IRSL. Attempts were made for selected samples to resolve components of the quartz OSL signal, and growth curves and equivalent doses were derived using different components. For samples from the upper two loess units (L1 and L2), there seems to be a dating limit of 80 ka for quartz. In contrast, luminescence ages obtained with pIR₁₅₀IR₂₉₀ signal increase with depth and are in broad agreement with the expected ages up to L2. Further work is in progress to elaborate the protocol most suitable for dating loess in this area and to investigate the upper age limits for different protocols.

Luminescence chronologies for the Dadiyuan archaeological site in the Nihewan Basin, northern China

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The Dadiyuan site in the Nihewan Basin is an important Upper Palaeolithic site with microlithic technologies, which are characterised by wedge-shaped cores. Importantly, a fragment of human skull (the posterior part of the right parietal bone) of the same thickness as that of anatomically modern humans has also been discovered at this site and is considered to belong to late or modern *Homo sapiens* [1]. We collected five sediment samples from the site and applied a single-aliquot regenerative-dose (SAR) procedure for single-grain quartz optically stimulated luminescence (OSL) measurements. The performance of the SAR procedure was tested using standard 'preheat plateau' and dose recovery tests, on the basis of which we chose a preheat at 220 °C for 10 s for the natural/regenerative doses and a cut-heat of 180 °C for the test doses. We observed that some of the samples yielded single-grain D_e values consistent with a single population, while others appeared to consist of a discrete mixture of two or more D_e populations. To estimate the D_e values for each of these populations, we applied the Finite Mixture Model [2] and obtained results which suggest that modern humans occupied the Dadiyuan site around 9,000 years ago.

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OSL to the rescue: constraining aeolian and lake sediments of the semi-arid Paroo/Warrego Region, Australia

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Palaeoenvironmental research in the Paroo/Warrego Region has uncovered a remarkable record of intermittent lakes including a 50 year (~1895-1945) period of drought caused by a decades-long suppression in annual rainfall. That is just at the surface; deeper in the sediments there is a chemical, isotope and pollen record that goes back much further.

Preliminary work by Pearson et al (2001) and Gayler (2009) has provided a tantalising record of environmental change on the arid edge of the western Murray Darling Basin. Water deficits recorded by beach and lake sediments were dated using radiocarbon and caesium but this OSL project seeks to date Lake Numalla, which led to readjustment of the beach profile. Currawinya Lakes (L. Wyara and Numalla in particular) have unique records of events that are not as well preserved, if at all, at 8 other lakes cored at that time in the area (Gayler, 2008).

The geographic location of the Currawinya Lakes between the monsoonal north and southern Westerlies belt makes them a sensitive gauge of any anomalous shifts or intensity changes in those systems. An as yet unpublished research into historical rainfall records by Gayler and Pearson has shown that rainfall trends recorded in the lakes extend well beyond their importance to the Paroo river catchment.

This work will contribute to a large gap in the north-east Australian arid zone and contribute to increasing the scientific values of the Currawinya National Park, and is part of a landscape understanding project (Pearson et al 2015).

Cross-check between OSL and ^{14}C ages in archaeological sites in the Qaidam Basin, Qinghai-Tibetan Plateau

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The timing of prehistoric human inhabitation on the Qinghai-Tibet Plateau (QTP) is a crucial issue for both palaeoclimatic and archaeological studies. The extreme altitude, severe climate, and scant resources, all served as effective constraints to early humans who tried to live on the “roof of the world”. The migration process from the lower land to the plateau might have experienced three steps: Step 1: colonization of lowland zones (<2500 m; e.g., Gansu Lowlands); Step 2: colonization of the middle-elevation zone (Qinghai Lake area and Qaidam Basin, 2500–4000 m); and Step 3: colonization of the High Plateau (>4000 m) [1-3]. The Qaidam Basin was an important transitional area for prehistoric human to access the high QTP, and this process was believed to start at ca. 15 ka BP [1-5]. Precise chronology is crucial for understanding this migration process. However, there are still conflicts on the schedule due to the lack of proper dating materials in these sites found on the ground surface for Optically Stimulated Luminescence (OSL) or ^{14}C dating.

OSL and ^{14}C dating are the chief methods for dating remains of human activity. In this study, we present 8 OSL and 16 AMS ^{14}C ages for samples retrieved from prehistoric sites in the Qaidam Basin, NE QTP. Our data show that: (1) human activities started at least from 8–9 ka, and should not earlier than the early Holocene in the Qaidam Basin; (2) for ^{14}C dating, charcoals and baked sheep dungs are ideal dating materials with ages in agreement with both stratigraphic order and OSL ages, while ages from bones and bulk sample of ash are sometimes underestimated; (3) anthropogenic post-depositional disturbance could result in age disorder. These results suggest cross-check between different dating methods is essential for chronology building in archaeological contexts. If ages fall out of stratigraphic order it is critical to examine the depositional process to determine the correct timeline of events.

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Initial results of 3D spectral measurements on handpicked sedimentary quartz grains with diverse OSL behaviours

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Analysis of OSL data from individually measured sedimentary quartz grains has revealed the presence of significant grain-to-grain signal variability and the existence of a range of quartz OSL behaviours, including differences in sensitivity, decay rates and dose-response curve shape. A type of behaviour frequently reported in the literature relates to grains where the sensitivity-corrected natural signal (L_n/T_n) does not intersect the dose-response curve (Class 3 in [1]) and for which a D_e cannot be estimated. These types of grains have been termed non-intersecting [2] and ‘over-saturating’ [3] in studies that have considered their influence on D_e estimates from single aliquots, where the bulk signal is a grain-average from this type of grains as well as a range of ‘well-behaved’ grains (those passing SAR acceptance criteria), grains that fail various SAR acceptance criteria and very dim grains.

This poster reports initial findings from research into the TL emission spectra of these problematic grain types. We aim to determine whether there are observable differences in the luminescence trapping or recombination properties of accepted grains and non-intersecting grains.

We use a Late Pleistocene quartz sample (KI14-09) from the megafaunal site of Kelly Hill Caves, Kangaroo Island, Australia, which displays a high percentage of non-intersecting grains (~6%). Individual quartz grains that had been initially measured for D_e determination were handpicked and grouped according to their OSL behaviours to undertake further luminescence signal characterisation.

In particular, the study makes use of the interferometer-based 3D TL spectrometer [4] housed at the Adelaide Environment Luminescence Facility, which measures TL peaks across a range of emission wavelengths. We also show results from TL experiments undertaken using UV and blue emission windows in a Risø TL/OSL-DA-20 reader.

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Moving from EM-CCD luminescence images to spatially resolved estimates of equivalent dose

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Luminescence imaging of intact sediment slices is potentially beneficial for age estimation. At the very least, spatial resolution in equivalent dose (D_e) values could be compared with visual images of the sediment to assess the impact of micro-stratigraphic variations (e.g., bioturbation, heavy mineral deposition). Better still, luminescence images could be compared directly with spatially resolved dose-rate measurements, allowing for more robust interpretations of D_e distributions and more accurate ages.

While imaging systems are now offered by the major manufacturers of luminescence instrumentation, the analysis of luminescence images presents some challenges. For example, the movement of aliquots between measurements, the variable focussing of the optical lens, and the cross-talk between grains must all be accounted for in the analysis. The correction for each effect also implies an increase in the uncertainty above the counting statistics (the calculation of which can also be complex), and this must be specific to each grain based on its position and in relation to its neighbours.

To simplify the problems somewhat, we have begun imaging of conventional Risø single-grain discs using a Freiberg Instruments EM-CCD system. This reduces the problems of cross-talk and motion correction, allowing for an analytical protocol to be developed for future extension to intact sediment slices. Our protocol combines empirical measurements of reproducibility with a computational model of cross-talk to produce grain-specific uncertainty estimates. Working initially with single-grain discs allows ready comparison with conventional measurements. We will outline the potential limits of the imaging systems, and discuss some further implications for conventional single-grain dating.

First insights on silt fraction OSL use in dirty speleothems dating

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Over the last decades, stable isotopes in stalagmites assumed a key role in Quaternary precipitation reconstructions, the U-Th dating method is the most used because its high precision and accuracy. But this dating method is only applicable in “clean” speleothems, once it is not possible to split the detrital Th adsorbed in clay minerals from the Th derived of U decay. Here we propose the OSL dating of silt fraction as an alternative method for dirty speleothem dating; we discuss the issues in dose rate estimation, using three different methods, and in equivalent dose determination with SAR protocol. Two stalagmites from Northeast Brazil (semiarid climate) were sawn in dark room, then samples were drilled from successive layers and treated with HCl to remove the silt from carbonate. All the measurements were performed in a Risø TL/OSL DA-20 reader equipped with a built-in beta source (dose rate of 0.1363 Gy/s), bialkali PM tube and Hoya U-340 filters (290-340 nm). In order to remove the feldspar influence in OSL signal a IRSL (during 100s at 125°C) were added before the blue LEDs stimulation on SAR protocol with a preheat of 180°C. The samples showed a good dose recovery test with less than 10% deviation between given and estimated dose. The U, Th and K content for dose rate calculation were measured with gamma ray spectrometry and ICP-MS, both methods gave similar results. The dose rate was also measured placing thermoluminescent (TL) dosimeters (LiF TLD-100) inside the holes drilled to extract samples and assembling the two stalagmites halves. The TL dosimeters were also used to measure the environmental dose rate. The dose rate measured with the TLD-100 was 3 to 4 times higher than calculated dose rate, probably because the effect of the environmental radiation. One of the stalagmites presented coherent results, with younger samples located close to the top, as the other one showed a chaotic age distribution, maybe related to a reservoir effect. The water that infiltrates the soil above the cave may carry unbleached silt, and so the age calculated for this sample does not correspond to stalagmite deposition. We conclude that OSL dating of dirty stalagmites is possible since the silt get trapped inside them right after bleaching, which is possible in caves with small soil cover or in speleothems exposed to dust from outside the cave.

Evaluation of modified SAR procedures for D_e determination

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The single-aliquot regenerative-dose (SAR) protocol has been widely applied to a large number of sedimentary samples as a means of measuring the equivalent dose (D_e) in quartz and feldspar (Murray and Wintle, 2000). The most distinguished feature of the SAR protocol is the use of a fixed test dose to correct sensitivity change. And furthermore, Murray and Wintle (2003) proposed an additional step to the SAR protocol. It is the modification of SAR protocol by the addition of a high temperature (e.g. 280°C) optical stimulation (referred as optical washing) following each measurement of the test dose signal. The aim of this stimulation is to remove the residual signal that was thermally transferred into the traps relating to the fast component. A number of previous studies (Tribolo and Stokes, 2006; Jacob et al., 2006; Porat et al., 2009; Pawley et al., 2010; Timar-Gabor et al., 2011) suggested improved dose recovery results when additional step was applied. Also, significantly reduced recuperation and improved recycling ratio have also been obtained from the modified SAR protocol. The modified SAR protocol is now conventionally used to the dose recovery test and also for natural D_e estimation.

On the other hand, the disadvantage in such an approach is that the significant sensitivity change can be caused by high temperature optical washing (>280°C). However, general applicability of modified SAR protocol has not been tested to predict sensitivity changes. It is required that how sensitivity change is affected by optical washing at different heating conditions. This study investigates the temperature dependent characteristics of sensitivity changes brought about by optical washing. To monitor the sensitivity changes in both the natural and regenerated OSL signals, dose recovery test was conducted for fine-grained quartz (coastal sediments) using normal and modified SAR protocols. The recycling ratio and recuperation were compared to check the efficiency of the SAR correction. From these results, possible dependence of sensitivity change and its effect on dose measurement is considered in more detail.

OSL dating of individual quartz 'supergrains' from the Early Middle Palaeolithic site of Cuesta de la Bajada, Spain

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The open-air site of Cuesta de la Bajada, Teruel, Spain, has yielded ~3000 lithic artefacts displaying early Middle Palaeolithic reduction techniques, as well as a multitude of late Middle Pleistocene faunal remains. The paucity of open-air Palaeolithic sites in the interior eastern sector of the Iberian Peninsula, and the relatively low number of documented early Middle Palaeolithic archives in this region, means that Cuesta de la Bajada is of key importance for understanding the coexistence/transition of Iberian Acheulean and Mousterian techno-complexes during the Middle Pleistocene period. Establishing reliable numerical chronologies at Cuesta de la Bajada is essential for placing the site within a broader archaeological and temporal context.

In this study we present results obtained using quartz single-grain optically stimulated luminescence (OSL) dating of four samples bracketing the main archaeological horizons. The measured quartz grains are characterised by relatively bright OSL signals and display dose-response curves with very high saturation limits. The large proportion of quartz 'supergrains' in these samples offers the potential to establish extended-range chronologies that exceed the traditional upper age limits of quartz OSL dating. A range of sensitivity tests and additional quality assurance criteria are used to assess potential biases in supergrain burial dose estimation over high dose ranges. The resultant OSL ages are also compared with electron spin resonance (ESR) ages obtained on quartz grains from closely associated samples. Both the laboratory suitability assessments and ESR dating comparisons provide good support for the reliability of the supergrain OSL chronologies established at Cuesta de la Bajada. We also report on experiments performed using 'synthetic aliquots' created from the single-grain D_e datasets, and discuss their implications for the reliability of multi-grain OSL dating approaches in this context.

Evaluating OSL, ITL, TT-OSL and VSL signals for dating of high sensitivity Brazilian quartz

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Brazilian quaternary sandy sediments are dominated by quartz as a result of heavy weathering under tropical climate. High OSL sensitivity quartz (fast component dominated signal) dominates eolian, fluvial, lake, coastal and marine sediments in Brazil. This study aims to establish the age limits for luminescence dating of Brazilian quartz using available protocols. Thus, we evaluate the sensitivity, bleaching characteristics and signal saturation dose ($2D_0$) of OSL, ITL, TT-OSL and VSL signals measured in quartz samples representative of Brazilian sandy sediments. The study also attempts to compare the OSL, TT-OSL, ITL and VSL signals regarding their sensitivity and saturation doses. A low sensitivity hydrothermal quartz sample was used as a standard for comparison with quartz from sediments. The sediment samples were sieved to isolate the 180-250 μ m grain size fraction, chemically etched with H₂O₂, HCl and HF respectively to eliminate organic matter, carbonates, feldspar and outer ring of quartz grains damaged of alpha particles. Afterwards, heavy liquid separation was performed to separate quartz from remnant feldspar and heavy minerals. Quartz aliquots were mounted in cups for luminescence measurements. Three Risø TL/OSL DA-20 readers equipped with sealed Sr⁹⁰/Y⁹⁰ beta sources (dose rates 0.01 Gy/s, 0.076 Gy/s and 0.110 Gy/s) were used for measurements, with one reader equipped with a violet laser attachment. Light detection was performed with Hoya U-340 and HC 340/26 detection filters. OSL signal sensitivity, represented by the percentage of the fast component (0.8s of light stimulation), ranged from 68% (50 mGy) to 83% (0.42 Gy). ITL signal sensitivity (60 Gy) varies from 8% (ITL-270°C signal, first 10s of light stimulation) to 23% (ITL-310°C signal, first 5s of light stimulation). The hydrothermal quartz sample is dim for radiation doses up to at least 10 Gy. Average saturation doses ($2D_0$) are 175 Gy (OSL), 618 Gy (ITL-270°C), 1082 Gy (ITL-310°C) and 1710 Gy (TT-OSL). The studied samples showed a weak VSL signal, unable to generate reliable dose-response curve. Doses of 30 mGy or less could be estimated using the OSL signal. Considering the average dose rate of 0.77 Gy/ka, the minimum age that could be calculated is around 40 years (OSL). Considering average $2D_0$ values, the maximum ages that could be calculated are around 220 ka (OSL), 800 ka (ITL-270°C), 1.4 Ma (ITL-310°C) and 2.2 Ma (TT-OSL). The natural doses for young (200 years) sample were 4 Gy (ITL-310°C) and 16 Gy (TT-OSL), suggesting adequate bleaching of the ITL and TT-OSL signals.

ESR dating of the Hougou Paleolithic site in the Nihewan Basin, North China

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The Nihewan Basin in northern China has been attracting the global attention of the geologists, paleontologists, paleoanthropologists and archaeologists for well-developed Late Cenozoic lacustrine strata with plentiful paleolithic sites and abundant mammal fossils [1, 2]. Since lack of suitable dating materials, many dating researches of the Early Pleistocene sites are mainly depended on magnetostratigraphy method [2]. While the ages of the Middle Pleistocene sites are still few, such as the Hougou Paleolithic site, which is far from the geomagnetic polarity reversal boundaries that it is difficult to obtain the reliable age based on magnetostratigraphy. The age of the Hougou site is valuable to explore the Paleolithic culture development history of the posterior segment of Nihewan early Paleolithic and understand the ancient human activities history in high northern latitudes of East Asia. Therefore, it is necessary to obtain the independent age of the Hougou site.

The fluviolacustrine sediment of the Nihewan Beds near the Datianwa platform (e.g. the sections of Donggutuo, Xiaochangliang, Dachangliang (Xiantai), Donggou and Haojiatai, 1-3 km away from the Hougou section) were covered by the last interglacial paleosol S1 [3]. The sedimentary level at the top of the Haojiatai section was dated of 266 ± 16 ka by recuperated optically stimulated luminescence (ReOSL) dating method. Assuming the top strata of the Nihewan Beds from Haojiatai to Donggutuo to be roughly contemporaneous, Zuo et al. [3] estimated the interpolated age of the Hougou artifact layer is 395 ka. The upper lacustrine sediment sample L1372 (the sampling location is equivalent to HG1 in this study) in Hougou site was dated of 363 ± 14 ka by OSL dating method [4]. Since several Paleolithic sites in the Nihewan Basin had been dated by the ESR dating method successfully.

In this study, three ESR samples collected from the culture layer were involved. We use both the Ti-Li center and Al center ESR signal of quartz to date the Hougou Paleolithic site, and discussed the age results obtained by the additive dose and regenerative dose methods.

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ESR and TL signals of quartz in the present river bed sediments and in possible source rocks

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ESR, TL and OSL signals have been used for the dating of samples in Quaternary [1], [2], [3]. Recently another direction has been tried, using the ESR and TL signals as indicators of sediment provenance. Sediment provenance could give important information on mountain uplift, river contention, and crustal movement. By examining the quartz crystals found in sediment and related bedrock, it may be possible to estimate the provenance of sediment. There are possible that the present river bed sediments have various possible source rocks. Therefore, it is necessary investigate that ESR and TL signals in quartz by a change in mixture ratios of possible source rocks for estimate of sediment provenance. In this study, we will report the characteristic of ESR and TL signals of quartz in the present river bed sediments and in the possible source rocks.

The crushed rocks and the present river bed sediments were sieved into 500 μm -1 mm grains in size. The samples were treated with hydrogen peroxide, hydrochloric acid, hydrofluoric acid and heavy liquid separation. Thereafter, extracted quartz grains were crushed again to 125-250 μm . All samples were irradiated by gamma ray to a dose of 2.5kGy. ESR signals were observed by ESR spectrometers (JES-X320; X-band JEOL RESONANCE Inc.). TL signals were observed by selecting the wavelength region by using the Time-Resolving Spectroscopy System.

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The alpha effectiveness of the ESR dating signal in hydrothermal barite

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The temporal change of submarine hydrothermal activities has been an important issue in the aspect of the evolution of hydrothermal systems which is related with ore formation [1] and biological systems sustained by the chemical species arising from hydrothermal activities [2]. [3] showed that ESR (Electron Spin Resonance) dating of barite is useful for investigation of history of hydrothermal activities.

Barite crystals formed by sea-floor hydrothermal activities contains large amount of Ra which replaces Ba in the crystal lattice where the internal alpha dose rate in barite contributes 40 to 60 % of total dose rate [4]. As the LET of α particles is much larger than β and γ rays, causing high-density ionization, the probability of recombination which do not contribute to the generation of the signal is larger, therefore, generating smaller amount of signals. Determination of alpha effectiveness is thus the one of the essential factors for improving the precision of dating of barite by ESR.

[5] investigated the alpha effectiveness for the ESR signal due to SO_3^- in barite by comparing the dose responses of the signal for gamma irradiation and for He ion implantation with an energy of 4 MeV, to obtain a value 0.043 ± 0.018 . However, the dose response was far from "good", where the number of points is not sufficient. The experiments of He ion implantation was repeated in the present study for several samples to determine the precise alpha effectiveness

A sample of hydrothermal barite, taken by the NT12-06 research cruise operated by JAMSTEC (Japan Agency for Marine-Earth Science and Technology) was used for the present study. A value of 0.011 ± 0.0028 was obtained for the sample from the Hatoma Knoll hydrothermal field of Okinawa Trough, Japan. Another value of 0.030 ± 0.0103 was obtained for a synthetic barite.

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ESR dating of hydrothermal anhydrite: the first trial

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Dating of submarine hydrothermal activities has been an important issue in the aspect of the ore formation [1] and biological systems sustained by the chemical species arising from hydrothermal activities [2]. For this purpose, dating methods using radioactive disequilibrium such as U-Th method of a sulfide [1], ²²⁶Ra-²¹⁰Pb and ²²⁸Ra-²²⁸Th of barite, Ra/Ba, ESR (Electron Spin Resonance) method of barite [2, 3] have been employed.

However, practical ESR dating has never been attempted anhydrite, which is also one of the main minerals produced in the hydrothermal field. Although it is soluble and unstable at low temperature in seawater, the mineral commonly occurs in active hydrothermal vents.

In this study, we tried dating of hydrothermal anhydrite by ESR and by radioactive disequilibrium. The samples used in this experiment were taken from the Hatoma knoll field and the Daiyon-Yonaguni knoll field of Okinawa Trough in KY14-02 research cruise in 2014 operated by JAMSTEC (Japan Agency for Marine-Earth Science and Technology). The bulk radium (²²⁶Ra and ²²⁸Ra) concentrations were measured with a low background pure Ge gamma ray spectrometer. ²²⁸Ra/²²⁸Th ages of the samples were obtained to be 2.0 and 2.3 years. After γ -ray irradiation at Takasaki Advanced Radiation Research Institute, Japan Atomic Energy Agency, they were measured at room temperature with an ESR spectrometer (JES-PX2300) with a microwave power of 1mW, and the magnetic field modulation amplitude of 0.1mT.

A ESR signal of the SO₃⁻ radical was observed in the anhydrite samples having the g values similar with those observed in barite. The signal intensity increased with increasing absorbed dose of γ -rays, indicating that the mineral is possibly useful for ESR dating. Their ages were calculated from the resulting equivalent doses (D_E) and the dose rates to be about less than 2.0 years, being consistent with the ²²⁸Ra/²²⁸Th ages. Discussions on thermal stability and on the lifetime of the signals will also be in the presentation.

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Feldspar IRSL dating of human-influenced barrier shoreline down the catchment of granite and volcano

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Accurate quantification of the past coastal changes is critical for better understanding human-impacted modern coasts and their fate in the coming decades and centuries. The Yumigahama barrier is located on the southern coast of the Japan Sea and has been developed in relation to the mid-Holocene sea-level stillstand and sediment supply from the catchment of Tertiary granite and Quaternary volcano, protecting the back-barrier lagoon Nakaumi from the sea. The barrier experienced an increase in the river sediment supply caused by the steel mining in granitic rocks after the Medieval Period, resulting in the rapid shoreline progradation at a rate of c. 1.5–2.0 m/yr. Assessment of the mineral composition of beach sediments suggest that the sediment supply to the barrier shoreline increased by c. 4 times. We applied the infrared-stimulated luminescence (IRSL) dating to feldspar coarse grains extracted from beach ridge sands of the barrier, and attempted to quantify the shoreline migration rate before the mining. All samples showed well-defined IRSL signal, which fades at a rate of 10–12 %/decade. Fading-corrected IRSL ages agree with the relative chronology revealed by the ridge sequence. The oldest sample, taken at the lagoon-side end, was dated 8.1 ± 0.7 ka, marking the coastal turnaround from transgression to regression after the post-glacial sea-level rise [2]. IRSL ages indicate the shoreline prograded at a rate 0.3–0.4 m/yr between 8.1 and 2.0 ka. This pre-mining rate indicates that the migration rate increased almost proportionally to the sediment supply during the mining.

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Age estimation of coral fossil by post-IR IRSL measurement of insoluble residue

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Dating of aragonitic coral is important in studies of sea level and tectonic history in the tropical and subtropical coastal area. Uranium-series (U-series) and Electron Spin Resonance (ESR) dates of coral have been providing us the detailed Quaternary sea level history [1]. Aragonite of coral fossils, however, often alters into calcite during the period of burial, interfering with ¹⁴C, U-series and ESR dating. In this study we dissolved altered corals by hydrochloric acid and investigate characteristics of luminescence signals from the residual materials to discuss possibilities for an elevated-temperature infrared stimulated luminescence (post-IR IRSL) dating. If we can estimate the age of coral from the insoluble residue, it will be of great help not only for cross checking the U-series or/and ¹⁴C age but for estimating the age of altered coral fossils which can not be dated by them.

The fossil coral samples were taken from Yonaguni Island, southwestern Ryukyus, southwestern Japan. The sampling layer is considered as middle – late Pleistocene horizon [2]. In order to get materials from the limestone for blue light stimulated luminescence (BLSL) or post-IR IRSL dating, the outer-rim of the sample, which might be bleached by sunlight, was cut off more than 5 mm under subdued red light in a darkroom. Furthermore the sample was dissolved in 6N HCl in 10 minutes to remove the outer surface because some parts of the sample surface were rugged and difficult to cut off completely. Our preliminary experiment suggested the acid treatment of 6N HCl in 10 minutes removed more than 5mm depth from the sample surface, we think this procedure made sure to extract unbleached materials from the sample. Then the sample were washed by distilled water and again soaked in 6N HCl with hydrogen peroxide (several %) till the calcite was completely dissolved. After these treatments above, we could get small amount of residual materials from the coral fossil sample.

Polymineral fine grains (2-10 μm) for BLSL, IRSL and post-IR IRSL measurement were separated from the residual materials using Stokes' law and mounted on stainless discs from ethanol suspension. Equivalent doses were tried to be estimated from BLSL signals by the 'Double-SAR' measurement protocol [5] and from IRSL50 and post-IR IRSL290 signals with the protocol of Buylaert et al. (2012) [6].

The external dose rate due to ²³⁸U, ²³²Th and ⁴⁰K concentrations was estimated from high-resolution gamma spectrometry. A cosmic-ray contribution was calculated from the equation of Prescott and Hutton (1988, 1994) [3] [4]. In our poster presentation, we discuss on estimating the annual dose rate including the internal dose one and on the age estimation.

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Thermoluminescence efficiencies induced by alpha, beta, gamma and X-ray of Mn, Mg and Fe doped calcite

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TL dating on calcite has several advantages. Calcium carbonate is one of common minerals on the earth and there are many targets which need age estimate. Calcite emits strong TL than quartz, so that younger samples can be dated.

However, thermoluminescence dating of calcite is sometimes problematic; e.g., sensitivity change occurred through repeated heating of samples, possible anomalous fading, difference in characteristics of luminescence response against different kinds of radiation (e.g., gamma-ray, beta-ray, alpha-ray, and x-ray).

By analyzing natural calcites, it was suggested that luminescence efficiency factors are a function of Mg, Mn, Fe and Sr concentrations. Thermoluminescence property of calcite may be subject to multiple chemical factors.

In this study, synthetic calcites with controlled impurity concentrations were analyzed to evaluate relationship between multiple impurity concentration and thermoluminescence properties quantitatively. Calcium carbonates with the variety of impurity were formed from mixing solutions of calcium, magnesium, manganese and iron. All synthetic samples were characterized by powder X-ray diffraction. They were mainly calcites, Mg 0.1% and Fe0.1% doped calcites were a mixture of calcite and a small amount of vaterite. Chemical composition, especially impurity concentration, was measured by LA-ICP-MS. We measured luminescence induced by alpha-ray, beta-ray, gamma-ray and X-ray, using Photon Multiplier (R649, HAMAMATSU) with filter of 600-650nm, and the relationship between luminescence characteristics and impurity concentration was examined.

As a result,

1. Doped element concentration of synthetic calcite increase with added concentration of element.
2. TL efficiency by gamma-ray radiation of Mn doped calcite, normalized against TL by the X-ray radiation, is low.
3. Mn doped calcite emitted high TL, and Fe doped calcite emitted little TL. Thermoluminescence property of calcite depends on Mn and Fe concentration.

Identification of silica minerals in meteorite using cathodoluminescence

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Cathodoluminescence (CL), the light emission induced by electron irradiation, has been widely applied in mineralogical and petrological investigations, especially for silica minerals such as quartz. Recently, a CL spectroscopy has been used to clarify the high-pressure phase of silica minerals in shock-induced lunar and Martian meteorites, whereas a CL microscopy has not employed for the studies of silica minerals in the meteorites due to their grain sizes with micro-meter order. We have confirmed a variety of silica minerals in eucrite, which corresponds to the HED meteorite derived from one of the largest asteroids (4 Vesta). In this study we have conducted to identify the polymorphs of silica minerals in eucrite using CL microscopy and spectroscopy for their mineralogical investigation.

Several eucrite (NWA1466, NWA5356, NWA7188, Juvinas) were selected for CL measurements. Their slices were fixed on a slide glass with low-luminescent resin, and coated with 20 nm carbon after a mirror polishing. CL color images were obtained using the Luminoscope (ELM-3), attached with a cooled charge-coupled device (CCD) camera, which is consisted of a cold cathode discharge tube and a vacuum chamber placed inside by the sample. It was operated stably with electron beams generated by an excitation voltage of 10 kV and a beam current of 0.5 mA. CL spectra were obtained employing a system of CL-SEM, which comprises a SEM (Jeol: JSM-5400) combined with an integral grating monochromator (Oxford: Mono CL2) over the wide wavelength range of 300 nm to 800 nm. The system was operated at 15 kV with 2 nA incident beam current in a scanning mode to prevent the surface damage of the sample by electron bombardment. All CL spectra were corrected for the total instrumental response.

Conventionally EPMA and Raman spectroscopy have been utilized to identify the silica polymorphs in the meteorites, whereas their phase mappings are so difficult to be made for a wide area. Two phases of quartz and tridymite in NWA5356 can be easily distinguished by the color of CL emissions, which show red-purple for quartz and aqua-blue for tridymite. Sub-euhedral crystals of tridymite with 400 μm size are closely associated with anhedral quartz around outer rim, which has a concave-convex surface on the boundary, suggesting later origin of quartz. CL spectrum of quartz shows an enhanced emission band at 380-390 nm in a blue region possible corresponding to defect centers related to Al-O bridge and/or Ti incorporation, and a weak emission band at 630-640 nm in a red region. Tridymite exhibits a pronounced emission at around 410 nm, which can be assigned to defect center of $[\text{AlO}_4/\text{M}^+]^0$ by referring to Kayama et al. (2009). The emission in a red region sensitively reflects deferent tone in the CL emissions between quartz and tridymite.

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He⁺ ion implantation effect on cathodoluminescence of zircon

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Cathodoluminescence (CL) imaging allows us to recognize internal zones and domains with different chemical compositions and structural disorder at high spatial resolution, e.g. for U-Pb zircon dating (SHRIMP and SIMS) as an important tool to interpret a history of the minerals at a micrometer-scale. Radiation damage to the structure by the radiation, especially alpha particles and recoils from decays of U and Th series, have been known to significantly affect the CL features of zircon, where their intensities and emission bands change depending on a radiation dose as a function. It implies that, CL emission of zircon provides valuable information on the radiation-induced defects in the structure if the CL affected by the radiation could be evaluated. In this study, He⁺ ion implantation, corresponding to alpha radiation from U and Th decays, on natural and synthetic zircon have been conducted to clarify radiation effect on the CL of zircon.

Single crystals of zircon from Malawi (MZ) and undoped synthetic zircon with impurity contents of <5 ppm except for Hf (SZ) were selected for He⁺ ion implantation. These samples were cut perpendicular to c axis, and their surfaces were implanted by He⁺ ion at 4.0 MeV with radiation doses from 8.82×10^{-6} to 1.47×10^{-3} C/cm². CL spectra were collected from the surfaces of the samples in the range from 300 to 800 nm with 1 nm step using a scanning electron microscopy-cathodoluminescence (SEM-CL). All CL spectra were corrected for the total instrumental response.

CL spectra of MZ show emission bands at ~310 and ~380 nm assigned to intrinsic centers and sharp peaks at 475, 580 and 760 nm to Dy³⁺ impurity centers. Also, the broad emission band at ~600 nm in a yellow region is also observed in MZ samples, as well as He⁺-ion implanted SZ samples. The intensities of yellow emissions from the MZ and SZ samples increase with a rising radiation dose of He⁺-ion implantation dose. The yellow bands of MZ samples are eliminated by an annealing above 700 °C for 12 hours, but reappear by He⁺ ion implantation on the annealing recovered samples. It implies that, the yellow bands may be due to a formation of SiO_mⁿ⁻ groups and/or Frenkel-type defects originated from metamictization, also supported by ESR analysis. Furthermore, the spectral deconvolution of CL spectra in the yellow region reveals an emission component specified to radiation-induced defects, of which density is closely related to radiation dose of He⁺ ion implantation. Consequently, CL spectral method employed here for the zircon, is expected to be available as a new indicator geodosimetry and geochronometry.

Quantitative determination of Mn²⁺ site occupancy in dolomite using cathodoluminescence spectroscopy

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Cathodoluminescence (CL) has been widely applied in mineralogical and petrological investigations, especially for carbonates. Dolomite, CaMg(CO₃)₂, commonly shows red CL emission related to an impurity center of Mn²⁺ as an activator, which occupies two different sites of Ca and Mg ions. Previous investigations by ESR and cathodoluminescence (CL) methods concluded that the distribution of Mn²⁺ between the two sites is related to the generation environments such as crystallization temperature. At room temperature, however, CL emission derived from Mn²⁺ exhibits a spectral band overlapping with two emissions derived by Ca and Mg sites, which is not suitable for a spectral deconvolution. At low temperature, CL emission is enhanced due to temperature quenching of Mn²⁺ activation, also accompanied by narrower width of a band peak with a decrease in thermal vibration. In this study, we have conducted to quantitatively estimate partition coefficients of Mn²⁺ ions between Ca and Mg sites by a spectral deconvolution of the CL data obtained at low temperature.

Ten samples of dolomite from various localities were selected for CL measurements. CL spectral measurements were carried out using an SEM-CL composed of an SEM (JEOL:JSM-5410) combined with a grating monochromator (Oxford: Mono CL2) from 300 to 800 nm in 1 nm steps with a temperature controlled stage from -190 to 45 °C. All CL spectra were corrected for the total instrumental response.

All samples exhibit a broad band in a red region, of which intensity is enhanced with a sharpened spectral peak at lower temperature. A spectral deconvolution using a Gaussian fitting was successfully performed for CL data in energy units obtained at low temperature. The CL consists of two emission components at around 1.82 eV (681 nm) and 2.10 eV (592 nm). The former can be assigned to the emission derived from Mn ion impurity occupied at Mg site, the latter to the emission at Ca site. The integrated intensities of these components lead to the partition ratio of Ca site to Mg site. Maximum Ca ratio is 0.42, whereas almost dolomite indicate lower than 0.2, suggesting a preference for Mg site in dolomite lattice. This CL method allows a quantitative estimation of Mn partition between Ca and Mg-sites with a high precision even for only one grain of micrometer-sized dolomite, which could be expected to be used as a geothermometer.

Cathodoluminescence of Pb-bearing carbonate minerals

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Carbonate minerals are consisted of various divalent cations, of which transition metal elements can act as an activator for luminescence emissions in carbonate structure. Occasionally, Pb ion performs an emission center as ns^2 -type activator and/or sensitizer in both aragonite-type and calcite-type carbonates, but emission mechanism still remains unexplained. We have confirmed a blue emission in Pb-bearing aragonite (tarnowitzite) and Pb-bearing calcite (plumbocalcite) as well as Pb carbonate (cerussite). In this study we have conducted to clarify the emission center caused by Pb ion in various types of carbonate minerals using cathodoluminescence (CL) microscopy and spectroscopy.

The samples employed for CL measurements are single crystals of tarnowitzite (Tsumeb mine, Namibia and Touissit, Morocco), plumbocalcite (Tsumeb mine, Namibia), and cerussite (Touissit, Morocco), which were mineralogically characterized by EPMA and Raman analyzes. Their color CL images were obtained with a cold-cathodo microscopy of the Luminoscope. CL spectra were measured by using a SEM-CL comprised of SEM combined with a grating monochromator at accelerating voltage of 15 kV and beam current of 0.1 nA in the wavelength-range of 300-800 nm. All CL spectra were corrected for the total instrumental response.

Color CL imaging reveals a bright blue emission for tarnowitzite, a weak red emission for plumbocalcite and a weak blue emission for cerussite. CL spectral analysis indicates an enhanced emission band at 390-400 nm in a blue region for tarnowitzite and an emission band at 310-330 nm in a blue-UV region with weak one activated by Mn^{2+} at 630 nm in a red region for plumbocalcite, and a weak broad band at 350-600 nm in a blue region for cerussite. The CL caused by Pb ion depends on the host structure of calcite or aragonite type, suggesting pronounced effect of crystal field by the ligands coordinated with Pb ion, whereas the CL emission in cerussite might be related to fundamental emission derived from the transition of Pb-O. Furthermore, a large difference in ionic sizes of Ca and Pb should cause a defect center in plumbocalcite.

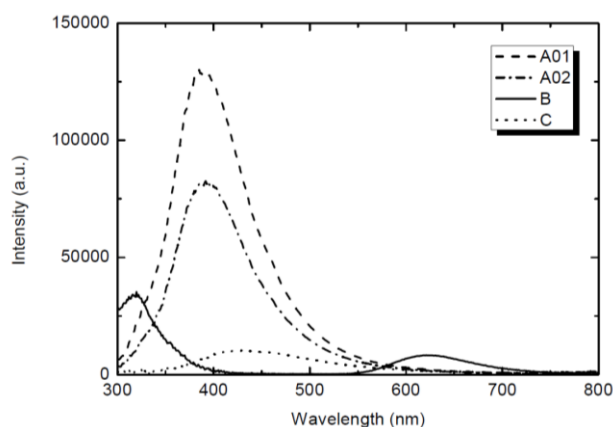


Fig. 1: CL spectra of tarnowitzite (A01, A02), plumbocalcite (B) and cerussite (C).

Cathodoluminescence zoning in the meteorite of Yamato 86004

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Enstatite is one of most important rock-forming minerals in the terrestrial and extraterrestrial materials. Luminescent enstatite has been found and investigated in enstatite chondrite (E-chondrite) and enstatite achondrite (Aubrite) without terrestrial materials. Yamato 86004 (Y-86004) is one of Antarctic meteorites, belonging to E-chondrite, of which luminescence has not been investigated so far. In this study, we have conducted to clarify luminescence characterization of the enstatite in Y-86004 using a cathodoluminescence (CL) microscopy and spectroscopy.

The sample of Y-86004 provided by the National Institute of Polar Research (NIPR), Japan has a spherical form of ca. 4 mm with a thin surface of fusion crust. The polished thin sections were used for CL measurements. Color CL images were obtained using a cold-cathode type Luminoscope with a cooled-CCD camera. CL spectroscopy was made by a SEM-CL system, which is comprised of SEM (JEOL: JSM-5410LV) combined with a grating monochromator (OXFORD: Mono CL2), where all CL spectra were corrected for total instrumental response.

By color CL imaging several CL phases were recognized as arranged in a concentric pattern, from within outward blue, bluish-purple, red, where the outer rim shows non-luminescent due to a fusion crust. Most enstatite with CL has similar chemical composition (e.g. FeO<1 wt.%), suggesting almost none of quenching effect by Fe²⁺. The CL spectra of these enstatite show a broad emission band around 390 nm in a blue region related to a defect center (intrinsic defect center) derived during crystal growth, and a broad emission band at around 670 nm in a red region assigned to impurity centers of activated ions substituted for Mg. A spectral deconvolution of the CL in energy units using a Gaussian curve fitting reveals the emission components related to impurity centers of Cr³⁺ at 1.70 eV and Mn²⁺ at 1.87 eV in a red region, and to defect centers at 2.72, 3.16 and 3.86 eV in a blue region. Conclusively, an abrupt heating of the spherical meteorite during atmospheric entry might cause the difference in peak temperatures depending on the depth from the surface due to a sudden increase in the temperature during a short time, which should eliminate the defect centers for a blue emission in response to the depth from the surface, resulting in the CL zoning in the section of the meteorite.

A GUI package for the deconvolution OSL components

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Components of an OSL decay curve can be mathematically deconvoluted with the use of curve fitting softwares. However, this deconvolution requires prior knowledge of the commercial softwares. Such softwares lack universality and work only for decay curves with high signal-to-noise ratio (SNR). Such methods are inefficient for large amount of data (e.g. a SAR analysis using components).

To overcome such problems, a user friendly, interactive and compact GUI package is developed in MATLAB. The GUI uses a non-linear fitting algorithm Levenberg-Marqardt based on χ^2 minimization to fit OSL decay parameters [1]. It isolates the OLS curve into an optimum number of components based on their decay rate. An initial guess can also be provided for the iterative, non-linear fitting algorithm.

The algorithm is critical to signal-to-noise ratio therefore the decay parameters of signals with high intensity (high dose) are used to separate the OSL components of signals with poor SNR. The GUI is interactive and works for all input data in a pre-specified format. It has the provision to accept user specified parameters like channel number, number of iteration etc..

A component specific analysis can also be done, enabling the user to understand the behaviour of the OSL curve constrained to only one of the three components. The output results, in the form of plots and data files, can be saved at user selected locations for later use. The GUI does not require any pre-installed software and works independently on any windows based operating system.

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Using DosiVox to model external dose rates for pottery from uncertain depositional contexts in Ancient Egyptian tombs

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Due to current restrictions on the exportation of archaeological samples for scientific analysis in Egypt, it is only possible to conduct OSL dating on museum material exported from the country prior to 1983. However, many ceramics held in museums were excavated prior to the introduction of rigorous excavation reporting and recording. Thus, although the finding of pottery is noted briefly in early excavation reports, they often lack any detailed or precise information about the exact deposition of ceramic material. This means that when working with museum material, the geometry of the external dose rate for ceramics can be immensely difficult to determine. For example, were the vessels upright, upside down, or on their sides? Were they propped up against the tomb wall or were they in the centre of the tomb? Were they half buried or fully buried? Recent comparative archaeological information is illustrative of the deep complexity often seen in pottery deposition, making any simplistic assumptions of burial history difficult to justify. With the development of DosiVox [1], however, we now have an excellent tool for modelling various depositional scenarios which can help us to more accurately model possible external dose rates, thus leading to a more comprehensive understanding of OSL dates for ceramics held in museum collections. This poster presents the findings of preliminary DosiVox modelling for the early Dynastic Tombs at Bet Khallaf, and the tomb of Djer at Abydos, in Egypt.

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