LUMINESCENCE IN ARCHAEOLOGY

INTERNATIONAL SYMPOSIA

L.A.I.S. 2009
9-12 September, Delphi, Greece

Dedicated to Prof. Martin Aitken (FRS)

Under the Auspices of

Ministry of National Education and Religious Affairs

Ministry of Culture
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FORWARD BY THE PRESIDENT

Nearly 50 years after the publication of the first Thermoluminescence ages, the field of Luminescence Dating has reached a level of maturity, in both research and applications in archaeology and geology.

L.A.I.S. is a new international initiative that mainly focuses on the use of luminescence dating for materials and questions of archaeological significance; in addition supports archaeological and archaeometrical communities of the World to further develop and expose luminescence issues¹.

L.A.I.S. Symposia aim at bringing together experts in the fields of luminescence, archaeology and archaeological materials from all around the world. In an exchange of knowledge, the techniques and tools available in luminescence dating and luminescence applications will be introduced to the archaeologists and archaeological/geoarcheological problems will be presented for the scientific community.

L.A.I.S. Symposia initiates a series of conferences planned to take place every two to three years; however, the next two symposia are planned for the consecutive years 2011 and 2013 while an initial planning on the next hosting countries has been made.

The 1st L.A.I.S. Symposium takes place in Greece and symbolically be hosted at the European Cultural Centre of Delphi, Greece (http://www.eccd.gr) in September 9-12, 2009.

The papers and posters presented at these conferences will be published in a special edition of a peer-reviewed international journal related with luminescence and/or archaeometry.

Topics

The topics range from fundamental studies of the physical basics and mechanisms of luminescence dating, through advances in equipment technology and analytical procedures, to sound applications and studies on archaeological material from various cultures of the World. Comparisons with other dating methods were encouraged. A few invited lectures will provide an overview on the main topics.

LAIS Organisation Details

The managing of L.A.I.S. is held by the International Standing Committee, two Advisory Committees and the Local Organising Committee.

The Int. Standing Committee is coordinated by the President of L.A.I.S. and the Members; the chairpersons of the Local Organising Committee of each symposium will be adopted as members. Meanwhile an ad hoc International Standing Committee was formed.

Prof. Ioannis Liritzis
University of the Aegean, Rhodes, Greece

¹ The other pertinent luminescence conferences are: 1) Intern. Luminescence & ESR Dating Conference, every 3 years, 2) Solid State Dosimetry Meetings, every 3 years 3) New World Luminescence Dating Workshops, 4) Asia-Pacific Conference on Luminescence Dating, 2006, and 5) the annual UK Luminescence & ESR Dating Meetings.
FORWARD BY THE CHAIRMAN

Dear Participants

On behalf of the Local Organising Committee, I'm pleased to welcome all colleagues to Delphi for the 1st LAIS - Luminescence in Archaeology International Symposium.

The Symposium has 8 sessions including 34 papers from authors oriented from all continents; the topics of the papers to be presented refer to the main fields of luminescence applications in archaeology and related material.

The Symposium could not have been carried out without the support provided by our sponsors.

We owe a great deal to all individuals who assisted us in making this Symposium possible. I would like to highlight two individual that is Nikos Laskaris and Metaxia Papageorgiou, accomplishing the painstaking work of preparing this volume.

Finally we would like to remind you that the Proceedings of the Symposium will be published following the peer-review process in a special volume of Mediterranean Archaeology and Archaeometry (MAA) Journal http://www.rhodes.aegean.gr/maa_journal/index.html.

Authors wishing to submit their paper should visit the journal’s net page for author guidelines. The deadline for full paper submission is set for October 15, 2009 and the proposed length is strongly advised to be at 6 printed pages of MAA journal’s format.

We wish you to enjoy the Symposium and the scientific work and also to have a pleasant stay at Delphi!

Assist. Prof. N. Zacharias,
University of Peloponnese, Greece
FINAL PROGRAM

September 9

WEDNESDAY

Registration - Welcome Cocktail (19:00 – 22:00)

DELPHI PALACE HOTEL
September 10
THURSDAY

REGISTRATION - OPENING CEREMONY
9:00 – 10:00

ARCHAEOLOGICAL DATING (STRUCTURES AND SETTLEMENTS) I
(10:00 – 11:50) Chair: I. Liritzis, D.A.G. Vandenberghe

(P1) A REVIEW ON LUMINESCENCE SURFACE DATING OF LITHIC MONUMENTS: 20 YEARS OF RESEARCH AND APPLICATIONS
I. LIRITZIS

(P2) FINAL PALAEOLITHIC SETTLEMENTS OF THE CAMPINE REGION (NE BELGIUM) IN THEIR ENVIRONMENTAL CONTEXT: OPTICAL AGE CONSTRAINTS
C. DERASE, D. VANDENBERGHE, M. VAN GILS, B. VANMONTFORT, E. MEIRSMAN, F. MEES, P. VAN DEN HAUTE

(P3) OPTICALLY STIMULATED LUMINESCENCE AND THERMOLUMINESCENCE DATING OF PREHISTORIC SETTLEMENT OF GÜLPINAR (SMINTHEION)
N. G. KIYAK, T. TAKAOĞLU, A. E. ERGINAL

(P4) OSL DATING OF TOMBS FROM AN IBERIAN NEOLITHIC NECROPOLIS
J. S. SÁNCHEZ, M.I. DIAS, D. F. MOSQUERA

11:50 – 12:20 coffee break

ARCHAEOLOGICAL DATING (STRUCTURES AND SETTLEMENTS) II
(12:20 – 14:00) Chair: A.K. Singhvi, N. Kiyak

(P5) LUMINESCENCE DATING: NEW IMPROVEMENTS IN SAR PROTOCOL AND NEW APPLICATIONS TO INDIAN ARCHEOLOGY (Invited)
A.K. SINGHVI, N. CHAUHAN, Y.C. NAGAR, M. JAISWAL, S. MISHRA

(P6) THE AGE OF THE ANCIENT CITY WALL SYSTEM AT THE TAYMA (NW SAUDI ARABIA) –IMPLICATIONS DERIVED FROM OPTICAL DATING

(P7) CHRONOLOGY OF CHICHEN ITZA, YUCATAN, MEXICO BY THERMOLUMINESCENCE DATING
H. CHUNG, A. RAMIREZ, P. SCHAAF, I. LEE

(P8) BULGARIAN MEgaliths – PRESENT STATE AND PERSPECTIVES FOR FURTHER DATING INVESTIGATION
L. V. TSONEV, D. Z. KOLEV
14:00 – 15:30 lunch break

September 10
THURSDAY

ARCHAEOMATERIAL DATING I
(15:30 – 17:20) Chair: M. Martini, J.S. Sánchez

(P9) LUMINESCENCE DATING OF MOSAIC GLASS (Invited)
M. MARTINI, A. GALLI

(P10) TOWARDS LUMINESCENCE DATING OF TURQUOISE GEMSTONE
USING TL AND OSL METHODS
B. SUBEDI, D. AFOUXENIDIS, G. S. POLYMERIS, N. C. TSIRLGANIS,
K.M. PARASKEVOPoulos, G. KITIS

(P11) HISTORICAL MORTAR: LUMINESCENCE FROM FINE GRAIN QUARTZ
A.M. GUELI, G. BURRAFATO, D. FONTANA, G. RISTUCCIA, G. STELLA,
S.O. TROJA, A.R. ZUCCARELLO

(P12) INDIRECT DATING OF LATE PALAEOLITHIC ROCK ART
AT QURTA (UPPER EGYPT) USING LUMINESCENCE SIGNALS
FROM ENTRAPPED SEDIMENT
D.A.G. VANDENBERGHE, M. DE DAPPER, D. HUYGE, F. MEES, G. VELGHE

(P13) OPTIMISATION OF PREPARATION AND MEASUREMENT PROTOCOLS
FOR LUMINESCENCE DATING OF SMALL SAMPLES FROM A SUITE
OF PORCELAINS AND FAIENCES
C.I. BURBIDGE, A.L. RODRIGUES, M.I. DIAS, M.I. PRUDÊNCIO, G. CARDOSO,
M.O. FIGUEIREDO, T. SILVA, M.A. MATOS, A.M. PAIS

17:20 – 17:50 coffee break

ARCHAEOMATERIAL DATING II
(17:50 – 19:30) Chair: A. Zink, C. Burbidge

(P14) 35-YEARS OF LUMINESCENCE DATING AS A TOOL
FOR FRENCH MUSEUMS
E. PORTO, A. ZINK

TERRACOTTA ARTEFACTS -
(P15) IS THERMOLUMINESCENCE ANALYSIS SUFFICIENT FOR
AUTHENTICATION?
R. NEUNTEUFEL

(P16) LUMINESCENCE DATING METHODS APPLIED TO RENAISSANCE
TERRACOTTA, THE CASE OF DELLA ROBBIA'S BOTTEGA
A. ZINK, E. PORTO
September 11
FRIDAY

**DATING PROTOCOLS IMPROVEMENTS**
(09:30 – 11:00) Chair: G. Kitis, S.H. Li

(P19) iIRSL DATING OF K-FELDSPAR AND ITS APPLICATION TO FAJIAOGOUWAN, SALAWUSU SITE IN CENTRAL CHINA (Invited)
S. H. LI, B. LI, A.G. WINTLE, H. ZHAO, G.M. YIN

(P20) TOWARDS A SINGLE-ALIQUOT REGENERATIVE DOSE PROCEDURE, SUITABLE FOR USE WITH PULSED OPTICALLY STIMULATED LUMINESCENCE (Invited)
M.L. CHITHAMBO, J.K. FEATHERS

(P21) INVESTIGATION OF THE OSL SIGNAL FROM VERY DEEP TRAPS IN QUARTZ
G. KITIS, N.G. KIYAK, G.S. POLYMERIS, V. PAGONIS

11:00 – 11:30 coffee break

**INFRASTRUCTURES AND DOSIMETRY I**
(11:30 – 13:00) Chair: I.K. Bailiff, C.T. Michael

(P22) SPATIALLY RESOLVED MEASUREMENTS OF PALAEODOSE AND DOSE RATE: ISSUES AND EXPERIMENTAL REALITIES (Invited)
I.K. BAILIFF

(P23) THICK SOURCE ALPHA PARTICLE SPECTROSCOPY: POSSIBILITIES AND PROSPECTS.
C.T. MICHAEL, N. ZACHARIAS, A. HEIN

(P24) THERMAL QUENCHING OF THERMOLUMINESCENCE IN NATURAL AND SYNTHETIC QUARTZ SAMPLES; IMPLICATIONS FOR DATING
G. S. POLYMERIS, B. SUBEDI, K. BAKOGLIDIS, D. AFOUXENIDIS, S. RAPTIS, N. C. TSIRLIGANIS, G. KITIS
(P25) ON THE SPECTRAL LUMINESCENCE EMISSION OF WHITE MARBLE AND THE CASE OF AIANI (GREECE)
J. GARCIA-GUINEA, E. CRESPO-FEO, V. CORRECHER, A. IORDANIDIS
G. CHARALAMPIDES, G. KARAMITROU-MENTESSIONDI

13:00 – 14:30 lunch

14:30 – 15:30
Round Table Discussion (Int. Standing Committee)

September 11
FRIDAY

INFRASTRUCTURES AND DOSIMETRY II
(15:30 – 17:20) Chair: N. Zacharias, M.L. Chitambo

(P26) TIME-RESOLVED LUMINESCENCE OF VOLCANIC QUARTZ
M.L. CHITHAMBO

(P27) FLASH PHOTOLYSIS OF MODERN AND FOSSIL BONE:
A POSSIBLE DATING METHOD?
A. BARTSOIKAS

(P28) PRELIMINARY TL AND OSL PROPERTIES OF NATURAL
AND ARTIFICIALLY IRRADIATED OBSIDIAN
G. S. POLYMERIS, G. KITIS, D. GOGOY, D. AFOUXENIDIS, S. RAPTI,
G. I. DALLAS, N. C. TSIRILGANIS

(P29) PRE-RESTORATION STUDY OF ETRUSCAN BUCCHERO VASE
M.G. KONONOVICH, A.Y. MAZINA, S.V. SOKOLOV, V.A. RASSUVOV

17:20 – 17:50 coffee break

GEOARCHAEOLOGICAL DATING
(17:50 – 19:40) Chair: G.W. Wagner, W. Wallinga

(P30) LUMINESCENCE DATING – KEY TECHNOLOGY IN GEOARCHAEOLOGY
(Invited) G.A. WAGNER, A. KADEREIT

(P31) THE CHIEMGAU (GERMANY) IMPACT OSL DATING PROJECT
N. ZACHARIAS, I. LIRITZIS, K. ERNSTON, D. SUDHAUS,
A. NEUMAIR, W. MAYER, M.A. RUPPENGLUCK, B. RUPPENGLUCK

(P32) UNDERSTANDING OF ARCHAEOLOGICAL DEPOSITS AND
SEDIMENTATION: OPTICAL DATING POTENTIAL OF LITHIC MICROWASTE
G.J. SUSINO

(P33) THE TIMING OF FLUVIAL LANDSCAPE EVOLUTION AROUND ARCHAEOLOGICAL SITES IN THE UPPER KHABUR BASIN (NE SYRIA): FEASIBILITY STUDIES USING OSL SIGNALS FROM FINE-GRAINED QUARTZ
D.A.G. VANDENBERGHE, K. DECKERS, T. WILKINSON, G. VELGHE

(P34) OPTICAL DATING OF A BURIAL MOUND CONSTRUCTED OF STACKED SODS – A FEASIBILITY STUDY OF NON-DESTRUCTIVE AGE PROSPECTION
J. WALLINGA, P.J. LEMMERS, D.R. FONTIJN, R. JANSEN, J. VAN DER PLICHT

19:40 – 20:00

Final Conclusions – Appointment of next LAIS

21.00 GALA DINNER
Tribune to retired Prof Gunther Wagner
This is an updated abridgement of the surface dating by luminescence. During the process of stone block preparation (cutting and carving, or sculpturing) and prior its construction, the solar radiation (UV and optical spectrum) bleaches the optically sensitive electron traps in the carved surface – much like in sediments - down to a variable depth. Solar transmission through limestones and marble can reach depths of 0.5-1 mm and up to 16 mm respectively, depending on the opaqueness (Liritzis and Galloway, 1999, 2000; Liritzis et al., 1997; Greilich et al., 2005). For quartz dominated stones the time of complete sun bleaching is in minutes, but for calcitic stones this varies between several hours to dozen of hours. In the latter case the quartz / feldspar technique for limestone monuments that contain traces of such minerals enables their dating by with single aliquot / single gain OSL techniques (Liritzis et al., 2007).

The surface dating has been developed and improved on various cases (Liritzis, 1994; Liritzis & Vafiadou, 2005; Liritzis et al., 2008; Vafiadou et al., 2005; Huntley & Richards, 1997; Theocaris et al., 1994).

Problems encountered and remedies since the birth (1991) of surface dating include:

a) high scatter of limestone TL measurements in the additive dose procedure (10-30%), propagated subsequent in age. The high scatter is due to thermal alterations of calcite in temperatures up to 400 °C where the TL of CaCO₃ induces sensitivity changes (lattice disruption) and alteration of 275 and 350 °C TL peaks.

b) the Dose rate microdosimetry in inhomogeneous rocks (e.g granites, schists). Care should be exerted for: i) K, Rb values on the actual spot of solid surface, ii) the U, Th contents from powder of the very near sampling region (μm to mm range), and iii) OSL probing of a narrow area (Bailiff, 2006).

c) incomplete bleaching of luminescence Calcites bleach slowly while quartz and feldspars quickly within minutes, but partial bleach should be investigated.

d) Growth of luminescence with irradiation (following the regeneration and additive dose techniques), as well as a dose recovery test (simulation test)

e) accidental removal of original dated surface either by earthquake, later repairs, or during initial block placement. Probing at sub-areas of ROI of surface is a satisfactory (but not definite) criterion securing a correct ED (obtained by radial plot).

f) dose rates, need particular attention due to the inhomogeneity of environmental gamma- ray dose rate, while beta particle dose-rates need a careful evaluation of the radiation field of the sandwiched dated surface.

g) ED determination for calcites follows dose plateau test (subtraction of bleached TL curves from the natural TL), while for OSL all necessary corrections and tests are applied (fading, recuperation, sensitivity changes from pre heat and shining, dose recovery).

Application examples covering the period 3rd millennium to Medieval times are presented.

References
The first Final Palaeolithic artefacts in NE Belgium were discovered at Lommel-Maatheide in the mid 1930s; since then, similar lithics have been found, spread all over the region. Their typological and technological characteristics allow attributing them to Federmesser, a tool-making tradition by groups of hunter-gatherers who populated the NW European lowlands after the last glacial. In most cases, Federmesser groups settled on (the southern flanks of) coversand ridges, as these elevated areas provided protection against groundwater and wind, and offered a higher incidence of sunlight. Typically, a depression with open water was situated to the south of these ridges.

The Federmesser artefacts are concentrated in the Usselo layer, which is intercalated between the Late Glacial coversands. The Usselo layer is a marker horizon that represents the Allerød Interstadial. Based on numerous 14C dates on Federmesser finds (e.g. burnt material from hearths, harpoons, resin) and associated organic matter (e.g. charcoal fragments from the top of the Usselo layer), the culture is generally dated to the Allerød. To improve our understanding of the cultural development of these groups of hunter-gatherers, it is necessary to consider their environmental settings. This, in turn, requires an absolute chronological framework for the regional evolution of the landscape and climate; at least for the Final Palaeolithic sites in NE Belgium, such a timeframe is virtually missing.

In this paper, we apply quartz-based optically stimulated luminescence (OSL or optical) dating to establish a chronological framework for the history of aeolian deposition at Arendonk-Korhaan and Lommel-Maatheide, two of the most extensive Federmesser sites in the northern Campine region (NE Belgium).

Samples for luminescence dating were taken from sediments over- and underlying the Usselo layer, as well as from the horizon itself; 30 samples in total were collected. At both sites, the bleached Usselo layer laterally changes into a peat layer, from which samples were taken for radiocarbon dating.

Equivalent doses were determined using the SAR protocol. The luminescence characteristics of all samples are satisfactory, and dose recovery tests (Fig. 1) indicate that the applied SAR procedure is suitable for our samples; the average measured to given dose ratios for Arendonk-Korhaan and Lommel-Maatheide are 1.05 ± 0.01 (n=42) and 1.02 ± 0.01 (n=48), respectively.

At the time of writing, dose rate determination is ongoing. Adopting a typical dose rate for the NW European coversands (~0.65 Gy/ka), however, yields optical ages between ~10-12 ka and ~13-15 ka for the sediment units immediately above and below the Usselo layer, respectively. These preliminary age estimates confirm the Late Glacial age of the coversands. Our completed chronological understanding of the environmental history of the Federmesser sites at Arendonk-Korhaan and Lommel-Maatheide will be presented and discussed at the symposium.
The site of Gülpinar (Smintheion), located on the southwestern corner of the Troad in northwestern Turkey has started to provide us with useful information about the fifth millennium B.C. in the eastern Aegean world. The site roughly falls into the Late Neolithic I phase of the Greek chronology and the vaguely understood transitional period between the Middle and Late Chalcolithic periods in western Anatolian chronology. Because sites such as Gülpinar are difficult to detect as they are represented by low level of cultural remains, its discovery is very important. A single cultural level representing this prehistoric settlement has been identified below the remains of the sacred road of the Graeco-Roman sanctuary of Apollo Smintheus.

The prehistoric site of Gülpinar (Smintheion) is often considered to be a part of the so-called “Kuntepe A/Beşik-Sivriştepe/Gülpinar” cultural horizon which defines roughly the first half of the fifth millennium B.C. in northwestern Turkey. Nevertheless, a precise dating for this archaeologically important site has not been obtained so far. Calibrated radiocarbon dates from the site of Beşik-Sivriştepe and Kumtepe A, which yielded archaeological finds identical to those of Gülpinar (Smintheion) yielded a date ranging from 4800 to 4500 B.C. A similar date is expected for the archaeological finds such as pottery from the prehistoric site of Gülpinar (Smintheion).

In the 2008 field season, pottery samples for Luminescence (optically stimulated and Thermoluminescence) dating have been collected from an undisturbed cultural level below the foundations walls of Roman structures. This prehistoric cultural level has a thickness ranging from 30 to 40 cm over the virgin soil. The top of this prehistoric cultural level is located about 150 cm below the modern surface level. Sampling was carried out at two different sites, 100 m north of the Apollo temple. The positions of sampled levels are shown in Figure 1.

Totally seven sediment and six pottery samples were evaluated by luminescence techniques. Quartz grains extracted from sediment samples were dated by conventional OSL-SAR protocol. Pottery samples collected from the same sediment layer were evaluated by both TL and OSL dating methods. The results from both sediment and pottery samples are ranging between 4.5 ka and 6.9 ka and in agreement with each other and expected dates.

Figure 1: Prehistoric sampling area (a). Sections from sampling sites 1 (b and c) and 2 (d).
Introduction: Luminescence dating has been used to date different types of archaeological and geological sediments. However, few works have been devoted to date tomb sediments (Lang and Wagner, 1997; Lang et al., 1999). These sediments were presumably deposited by human action in the past. One difficulty of dating this sediment is uncertainty about whether the residual geological signal of the sediment quartz grains has been completely reset prior to burial, as data concerning the degree of exposure to light of the sediments prior to deposition is unknown.

The necropolis of Sobreira da Cima (Beja, Portugal) is an archaeological site located in the Guadiana River basin. In this site, tombs were excavated in the ground rock and filled with sediment. Inside the bottom part of the tombs, human bones have been found. Thus, the human remains of some of the tombs have been dated by 14C (Valera et al, 2008).

Methods: Six sediment cores were taken for OSL dating of two tombs (three in each one) at different depths from the top to bottom. Coarse grain quartz sub-samples were obtained and the SAR protocol was applied to calculate equivalent doses (Murray and Wintle, 2000). The annual doses have been calculated form U, Th and K data measured by Neutron Activation Analysis (NAA). Although these technique do not allow checking disequilibrium in the 238U decay series, a geochemical characterization of the sediments by XRD and AAN has been carried out to assess if there has been any leaching of elements.

Results and Discussion: The three OSL ages calculated for each of the two tombs agree but they are different for both tombs. The first one is ~6.5 ±0.5 ka BP and the ages of second tomb are similar (~5.5-6.5 ±0.3-0.4 ka BP) although little increase of age has been observed from the top to the bottom sample. OSL ages are light younger than 14C ages obtained from bones of the first tomb (5.4 ± 0.2 ka BP). Although the second tomb was not dated by 14C, other tombs of the site have been dated (~5.1 ± 0.3 ka BP). The 14C age of most of the tombs agree with the OSL age of the top and middle samples of this second tomb.

The differences in the 14C and OSL ages have encouraged a detailed check of the equivalent and annual dose for all samples. The three equivalent doses are similar in the first tomb and different in the second one. As could be observed constructing radial plots and histograms, all equivalent doses showed a Gaussian distribution. Possible partial bleaching of the samples were also assessed by D(t) plots. A flat plateau has been observed in all the samples, indicating no evidence of partial bleaching.

Annual doses are similar in all samples, but the geochemical data suggest slight differences in the composition of the samples taken from the two tombs. These differences are due to leaching of some mobile elements. Also, the Th/U ratio slightly decreases from the top to the bottom samples of both tombs. This can be indicative of disequilibrium in the 238U decay series, a geochemical characterization of the sediments by XRD and AAN has been carried out to assess if there has been any leaching of elements.

References:
In Optically Stimulated Luminescence dating, increasingly the single aliquot/single grain regeneration (SAR) method is being used to estimate the paleodose (e.g. Vafiadou et al., 2007). In the SAR method the natural luminescence signal is interpolated on a laboratory regenerated luminescence vs. dose growth curve with due care for changes in luminescence sensitivity at each irradiation and preheat stage. The procedure assumes that during the read out and preheat of natural OSL, no sensitivity change occurs. However significant sensitivity changes (up to 20-30%) were observed, both during the natural OSL read out and preheat. Such sensitivity changes need an accounting, to avoid systematic offsets in ages. These changes can be corrected for, by using the $110^\circ$ C TL signal as a surrogate for OSL sensitivity (Stoneham and Stokes, 1991; Wintle and Murray, 1999). This eliminates possible interference from short optical stimulations for sensitivity measurements. The flow diagram details the protocol. Applications of this correction to samples from varied depositional environment of archaeological relevance will presented.

Archeological applications of luminescence in India were initiated during the early seventies with the dating of pottery. Subsequent developments include the estimation of firing temperatures via pre-dose effect; direct dating of archaeological strata and the dating of Palaeolithic implements via the dating of burial contexts. Recently Palaeolithic in the western and central India have been examined to address the problem of arrival modern humans. Genetic studies suggest that the present population of the region diversified in-situ for the past 70 ka making it important to examine sites in the age range 40-100 ka. Microliths are common in Indian sites and radiocarbon ages of > 40 ka, for these have been reported. Luminescence Dating at Mehtakheri, central India indicates older ages for this culture. Association of blade (chert and quartzites) industry with Youngest Toba tephra indicates an early age for this industry and possible association with the dispersal of modern humans in the sub continent. Some of these issues will be discussed.

References:
Since autumn 2006, the archaeological cooperation project of the German Archaeological Institute Berlin (DAI) and the General Commission for Tourism and Antiquities, Kingdom of Saudi Arabia, integrate geo-archaeological methods to examine the physical environment of former settlement periods at the ancient site of Tayma. The archaeological excavation mainly focuses on the mound of Qraya and the prominent city wall system stretching 15 km around the Tayma oasis (Eichmann et al. 2006). Archaeological evidence provided a general *terminus ante quem* of the late 2nd / early 1st millennium BC for the outer city wall. Since this date concerns only the last modification of the wall, a much earlier date for its construction was to be expected.

Near to the foundation of the western outer city wall, a systematic dating approach – combining optically stimulated luminescence (OSL) and $^{14}$C – was carried out in order to generate a reliable age for the oldest branch of the wall system nowadays covered by aeolian sand. We applied SAR technology (cf. Murray and Wintle, 2000) on coarse grains quartz samples. Before equivalent dose estimation, laboratory experiments such as preheat plateau and dose recovery tests as well as linear modulated OSL were carried out to investigate the luminescence behaviour of the samples. The dune deposit is related to the existence of the wall; therefore, dating its phase of accumulation provides minimum ages for the construction of the wall (Fig. 1, upper right photo). First OSL and radiocarbon ages indicate a construction of the wall already during the 3rd millennium BC, which is older than expected so far. Meanwhile, several sections of the city wall were sampled for optically stimulated luminescence dating (OSL) with the aim to verify the existing OSL ages.

**References:**


Ancient Mayan civilization developed in areas such as today’s Mexico, Guatemala, Honduras and Belize. The Mayan civilization development period is divided into the Preclassic, Classic, and Postclassic periods. When the most developed Classic Mayan period had decayed by 900 A.D, some Mayans moved into Chichen Itza, north of the Yucatan peninsula in Mexico. During last 60 years scholars used the chronology of Chichen Itza based on the stylistic change of architecture and pottery which reflect the intrusion of southern Mayans. However, from the very beginning archaeological evidence and the chronology of the site was not matched correctly, and it was suggested the necessity of dating by scientific technology. Pottery fragments were collected from every cultural layer of 13 test pits of Chichen Itza and related archaeological sites. From those we selected volcanic glass contained fragments by thin section petrographic analysis. And it were applied Thermoluminescence(TL) dating of fine grain technique. The Paleodose was estimated by the additive and regenerative method calculating equivalent dose and supralinearity factor respectively. The annual dose was estimated with natural radioisotopes (U, Th and K), and cosmic contribution. Once obtained the paleodose and annual dose were substituted in the age equation from Paleodose/Annualdose:

\[
\text{Age} = \frac{\text{Paleodose}(Q+I)}{\text{Annualdose}(D_\alpha + D_\beta + D_\gamma \text{ and } D_c)}
\]

\[Q = \text{equivalent dose, } I = \text{supralinearity factor, } D_\alpha, D_\beta, D_\gamma \text{ and } D_c = \text{Dose alpha, beta, gama and cosmic contribution.}\]

<table>
<thead>
<tr>
<th>No.</th>
<th>Dates of TL (A.D.)</th>
<th>Location</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ch 3</td>
<td>934± 27</td>
<td>T5, Edzná**</td>
</tr>
<tr>
<td>Ch 4</td>
<td>384 - 398</td>
<td>C.W., Chichén Itzá</td>
</tr>
<tr>
<td>Ch 5</td>
<td>306 ± 97</td>
<td>Pit T5 III, Edzná</td>
</tr>
<tr>
<td>Ch 6</td>
<td>660 ± 84</td>
<td>Pit T5 II, Edzná</td>
</tr>
<tr>
<td>Ch 7</td>
<td>1270 ± 42</td>
<td>Pit Pp II, Labna</td>
</tr>
<tr>
<td>Ch11</td>
<td>1063 ± 47</td>
<td>29 III, Mayapan</td>
</tr>
<tr>
<td>Ch13*</td>
<td>1532 ± 26</td>
<td>Pit S.C1 4 I</td>
</tr>
<tr>
<td>Ch14</td>
<td>1110 ± 53</td>
<td>Pit Buho I</td>
</tr>
<tr>
<td>Ch15</td>
<td>1132 ± 69</td>
<td>Pit Buho III</td>
</tr>
<tr>
<td>Ch16</td>
<td>1055 ± 85</td>
<td>Pit Buho III</td>
</tr>
<tr>
<td>Ch17</td>
<td>1221 ± 30</td>
<td>Pit S.I. I</td>
</tr>
<tr>
<td>Ch18</td>
<td>875 ± 88</td>
<td>Pit JJ2 I</td>
</tr>
<tr>
<td>Ch19</td>
<td>1154 ± 76</td>
<td>Pit Chac2 I</td>
</tr>
</tbody>
</table>

Table: TL Dating of samples
*didn’t show plateau
** Edzná, Chichen Itza, Mayapan: archaeological sites;
S.C14, Buho, S.L., J.J2, Chac2: name of structures.
I, II, III: cultural layer

The detailed dates obtained by TL method show the necessity to correct chronology of Chichen Itza.

References:
BULGARIAN MEGALITHS – PRESENT STATE AND PERSPECTIVES FOR FURTHER DATING INVESTIGATION

L. V. TSONEV1, D. Z. KOLEV2

Introduction: The presentation makes the first attempt to show a global picture of the megaliths in Bulgaria at a wide international forum which deals with dating. It contains a short review of the investigations up to now. Three issues are pointed out: (a) Menhirs and dolmens in Bulgaria today; (b) Necessity of more precise dating; and (c) International collaboration for creating a full, modern, precise common megalithic data base within a chronological framework for the Balkan region employing luminescence techniques.

Up to date investigations of the megaliths in Bulgaria: The scientific studies on the megaliths in Bulgaria started with the contributions of various researchers from 1880-1982, while continued in the beginning of XXI century for dolmens (Strandzha, and East Rhodope mount.) (Fig.1). Recently 2 cromlechs in East Rhodope mountain and in Thracian valley near Plovdiv were found. All the dolmens are dated by small artifacts of funeral origin (pottery, bronze fibulae etc.) in the period XII-VII cent. BC, therefore they are supposed to be rather younger than the West European and the Caucasian monuments. However the findings do not necessarily date the structure.

A new contribution to the megalithic research in Bulgaria & potential dating: A small group of physicists from the Bulgarian Academy of Sciences started in 2003 to collect new data. Two Bulgarian-Spanish expeditions were carried out and the preliminary results were published (Tsonev & Maritsas, 2009, Kolev et al., 2008). 80 dolmens are visited, localized by GPS technique, their dimensions are determined. Careful measurement of their orientation was undertaken -46 in Strandzha mountain, 21 in Sakar mountain and 13 in East Rhodope mountain. Here we try to build a new and modern data base. The orientation of the Bulgarian dolmens is distributed more or less uniformly over the whole interval between 90 and 270 degrees. Obviously, they are not built according to an astronomic criterion.

Constructively we found in Bulgaria only “ca-mera type” dolmens (XII-VII cent. BC)(Fig.1) in contrast to the situation in Western Europe (XL-XX cent. BC) where the “table type” dolmens definitively prevail. The Caucasian dolmens (XXV-XV cent. BC) also belong to the “camera type” and are technically more developed than the Balkan ones. This fact requires an additional and more precise dating of the East European dolmens, e.g. by optically stimulated luminescence or TL following current methodology (Liritzis et al., 1997; 2007). This way the various construction phases could be accurately dated.

Perspectives for further studies: A full and modern data base, which includes the megaliths in Bulgaria, Greece and Turkey, is proposed in order to investigate the Balkan megaliths as a whole. International collaboration is necessary to create a complete archaeological and chronological picture of the region. Menhirs must also be included together with the dolmens. Archaeo-astronomic research and OSL dating, potentially via a vis, could be realized on the entire multitude of objects. Last but not least such accurate results may initiate international scientific teams to make provision for preservation, restoration and tourist popularization of this kind of objects.

Acknowledgements: The participation of L.V.Tsonev is supported by Project TK 01/0404 (2008).

References:


The possibility of dating ancient mosaic glasses has been considered by analyzing the Thermoluminescent (TL) behaviour of a number of mosaic tesserae having different provenance. Optically Stimulated Luminescence (OSL) measurements have been carried on as well.

TL glow curves have been measured by means of a home made instrumentation and with a Risoe automatic apparatus, that has been used for OSL measurements as well. The wavelength of the emitted TL has been measured by means of a home made system (Martini et al, 1996)

Using specific techniques typical of the analysis of TL glow curves, like the “initial rise” following series of “partial cleanings” it has been possible to distinguish the TL emission due to the vitreous base of the tesserae from the emission given by crystalline micro-inclusions. These inclusions are rather different depending on the type of tesserae. When specific addition of opacifiers, like calcium antimonate, were made, there was evidence of their specific TL emissions both in 2D and 3D TL curves.

In many samples there appear to be presence of quartz micro-inclusions whose concentrations do not allow to be detected by XRD, but produce glow curves typical of quartz. In some cases they were probably intentionally added in order to increase the light scattering, acting as opacifiers.

Problems arose in correctly calculating the archaeological dose, because of the difficulty in separating the vitreous component, strongly affected by anomalous fading, from the dosimetric component due to the crystalline micro-inclusions.

Recently the OSL emission of the same samples has been considered, appearing to be strongly correlated to the stable component of the TL emission.

A limited number of dates obtained from the measurements of TL and OSL of tesserae coming from the San Pietro Basilica in Vatican will be presented, showing a satisfactory agreement with the know ages.

References:
Turquoise is among the first gem stones used in jewellery and possessing cultural value since 6000 BC (at least). In the past it was used as a stone of natural protection for the occupational groups who were exposed to especially high degree of risk such as pilots, air crews but now it is mainly use in jewelry and in modern gemstone therapy for those suffering from depression. The aim of this research is to identify and characterize this valuable stone scientifically by using both thermally stimulated luminescence (TL) and optically stimulated luminescence (OSL) techniques and investigate its potential use towards luminescence dating purposes. Experimental investigation includes the study of the natural TL and OSL signals, dose response curves, activation energy by initial rise method, sensitivity test of both signals and successive dose-measurement cycles. Thermal activation curves were also studied for annealed sample 1273K (900°C) which showed presence of at least three traps. The main dosimetric peaks have a peak temperature at the maximum at 339K and 514K. However, among these peaks, only the latter becomes of great interest for dating proposes. Both activation energy and frequency factor of this trap were determined and the mean trap lifetime was estimated to be in the order of 23.6ka. The OSL resulting from the same trap was also studied by performing a series of thermal annealing experiments. A series of doses were delivered, ranging from 2.5Gy up to 40Gy for both signals and showed linearity in the dose response. Small sensitization was observed at 493K (220°C) and deactivation takes place in the range of temperature 773K (500°C). The results of the present work are very promising and clearly support the possibilities of characterizing the provenance of turquoise according to geological location, of authenticity testing and of dating ancient turquoise artifacts as well as to divulge its potential dosimetric application.

References:

References:
Within the framework of a global project aimed at the chronological reconstruction of the structures belonging to the architectonic complex of San Francesco alla Collina of Paternò (Catania, Italy), a procedure was carried out using dating by thermoluminescence (TL) on terracotta bricks and optically stimulated luminescence (OSL) on mortar from the same sampling points.

The study is part of a larger research programme with the aim of extending the field of application of stimulated luminescence to mortar from historical buildings. This material is of historic-artistic interest and allows the direct dating of the building of architectonic structures.

The TL dating of the bricks or of other terracotta elements is, in fact, of the indirect type, in as much as they offer a terminus post quem: the results give the period in which they were made. The possibility that these bricks could have been reused complicates the interpretation of the dating data in terms of the period when the structure was built.

The dating of historical mortar could resolve this problem giving a more accurate date for the construction. The experimental difficulties are numerous, in particular due to the presence of unheated mineral phases added to the mortar. It is also necessary to carry out a detailed study of the mechanisms of luminescence, both thermally and optically stimulated as well as the trap centers implicated in the process of emission, above all in the case of quartz crystals, and the carbonate phases that formed during the preparation of the mortar.

In this occasion we present the results obtained during the measurements made on the enriched polymineral fine silt fraction in quartz. The bricks underwent the standard preparation while the mortar required some modifications to this protocol verifying the results step by step. A methodology was thus established to allow, by IRSL (InfraRed Stimulated Luminescence) measurements, the verification of the feldspar component reduction and the acquisition of an enriched quartz phase that underwent OSL dating.

The comparison of the age obtained by means of TL and OSL showed a good agreement thus supporting the potential of the methodology for the dating of mortar. Ongoing research is studying in detail the relationship between the signals obtained and the optical bleaching effect on the fraction of quartz extracted from the mortar.

References:
Recent Belgian archaeological missions to the surroundings of the village of Qurta (Upper Egypt) led to the discovery of petroglyphs which are quite unlike any rock art known elsewhere in Africa (Huyge et al., 2007; Huyge, 2008; Huyge and Claes, 2008). The nature and stylistic properties of the images, as well as their substantial patination and weathering suggests that they are of considerable antiquity. So far, it has been impossible to obtain numerical age constraints for the Qurta rock art; no directly-associated datable archaeological material was found, and the varnish rinds could not be dated through radiocarbon or U-series methods.

Through intensive surveying of the Nubian Sandstone cliffs near Qurta in 2008 and 2009, localities were found where the rock art is (partially) covered by coarse debris, commonly with loose sediment in the space between the rock art surface and the nearest stone block. The sedimentary infill has a homogeneous fine-sandy texture; in the field, it was interpreted as material that had been blown up from the base of the escarpment. This would make the sediment suitable for luminescence dating.

In this paper, we investigate the potential of quartz-based optically stimulated luminescence (OSL) dating to establish a reliable minimum age for the Qurta rock art. The luminescence characteristics of coarse (90-125µm) quartz grains extracted from two trapped sediment samples are investigated to some detail; intricacies of the annual dose (or dose rate) determination in this specific context are considered as well.

We show that, for both samples, the quartz OSL signal is dominated by a fast component. Dose recovery tests indicate that the single-aliquot regenerative-dose (SAR) protocol is suitable for equivalent dose (D$_e$) determination (measured to given dose ratio: 1.01±0.04; n=6). The D$_e$'s obtained using SAR are independent of preheat temperature. Analyses of large (8mm Ø) aliquots of quartz, in combination with gamma-ray spectrometric analysis of the entrapped sediment, yields optical ages of 9.1±1.0ka and 9.0±0.9ka.

We then report on a series of investigations that aim at refining these age estimates.

With respect to D$_e$-determination, we consider:
- physical and/or chemical weathering of rock fragments incorporated in the entrapped sediment (either in nature or during sample preparation), which would lead to contamination of the sedimentary quartz with grains that are significantly older;
- post-depositional mixing of the sediment (bioturbation), with specific relevance to the large-aliquot ages underestimating the true burial age.

With respect to the determination of the annual dose, we address the questions of:
- uniformity in radioactivity in the surroundings of the samples. As the sediment fill is a narrow planar structure between two Nubian Sandstone bodies, the rock will contribute to the gamma dose rate experienced by the samples;
- the cosmic dose rate to the sediment samples in this particular burial context.

At the time of writing, these investigations are ongoing. They involve luminescence analyses of small (2mm Ø) aliquots of quartz, as well as micromorphological research; both aim at unravelling the origin and resetting history of the entrapped sediment. The results, together with those of gamma-ray spectrometric analysis of the Nubian Sandstone and our evaluation of the cosmic ray contribution, will be presented at the meeting.

References:
As part of a wider study into the production and importation of porcelain and faience in Portugal, stimulated luminescence is being used to test typology-based chronologies for ceramics from various archaeological sites in Lisbon and Coimbra. To achieve this, protocols are being developed to facilitate luminescence testing of small samples (<100mg) from these different types of ceramic, in a uniform manner.

Conventional approaches to luminescence measurement of small samples from weaker ceramic fabrics such as faience use powder obtained directly by drilling using a tungsten bit. Porcelain is compact and drilling or crushing can remove luminescence signals and alter a sample’s behaviour, so it is conventionally measured in slices cut from a core drilled through the piece. Both approaches produce a polymineral sample and hence variations in luminescence behaviour depending on sample composition. A defined grain-size fraction may be separated from the powder by Stokes settling, but results from slices of porcelain may also be affected by differences in the doses absorbed by different grain sizes. Dating measurements are ideally conducted on specific mineral and grain-size fractions isolated from much larger samples (e.g. entire sherds ~20g).

In the present study cores have been drilled from the broken faces of sherds using hollow diamond tipped bits, and cleaned to remove any contamination. Initially, parts of some larger cores were coarsely crushed and unprocessed material was measured by thermoluminescence (TL) using the Multiple Aliquot Additive Dose technique. Signal levels were usable, but luminescence behaviour attributed to the presence of calcite was poor. Other crushed sub-samples were treated briefly in HCl and HF to remove carbonates and surfaces affected by crushing, and single aliquots measured using a combined sequence designed to test the signal levels and behaviour of Predose and Regenerative TL, and Regenerative optically stimulated luminescence (OSL) signals. Material losses from this preparation were unacceptably high, but the Predose TL signal was identified as most commonly present at usable levels.

Subsequent work focussed on the isolation of grains of quartz for Predose TL measurement using the simplified protocol of Galli et al. (2006). Cleaned cores were treated in 1M HCl for 4 days to weaken the ceramic by removing carbonates: this allowed both faience and porcelain to be disaggregated into powder. Predose TL signals from the residual silicates from porcelains were low but behaved well, while those from faiences were higher but subject to interference (Fig. 1). After settling to isolate the >60 and <11 μm fractions, samples were treated in fluorosilicic acid. Sufficient quartz enriched material (~5 mg) has been obtained for absorbed dose determination on a number of aliquots for each sample, to allow absolute age estimation based on Predose TL and SAR OSL from the > 60 micron fraction, combined with dose rates from INAA and a limited number of soil samples retained from the excavations.

References:
Since 1974, the Research department of the Centre for Research and Restoration of French Museum (former Laboratory of Research of French Museum) hosts a luminescence dating unit. It develops luminescence dating methods (thermoluminescence and optically stimulated luminescence) applied to cultural heritage objects, especially those from the public collections in French museums.

Founding in the 30's as a service of painting department of the Louvre museum, the laboratory became a national facility for French museums in 1968. Few time after, a specialist of ceramic, Ms J. Gautier, was recruited to develop new methods, especially TL. The first date was obtained in 1974 with the collaboration of M. Aitken and the Oxford RLAHA. In 2000, the acquisition of a Risoe TL/OSL DA15 reader gives us opportunity to extend our capacity to OSL.

Today, the luminescence dating unit staff has two permanent agents: 1 engineer and 1 technician. The other dating methods developed in our laboratory are radiocarbon, dendrochronology and archaeomagnetism. The colleagues specialists of the four methods form the so-called dating group.

Our studies can be divided into three types according to their aims and importance:

i) Short studies including investigation of art object before acquisition by museums as well as assistance to restoration.

ii) Large investigations including assistance to preparation of exhibitions (della Robbia, Paris-Nice, 2002 ; Tanagra, Paris-Montreal, 2004) and catalogues (Ancient Terracotta Lamps, Museum of national Archaeology, 2007), participation to investigation in Art history and Archaeology Project (Seille Valley’s Briquetage ; Medieval tiles of Yonnes ; Neishabur).

iii) Scientific methodological research, including research on methods and protocols and solid state investigations (characterization of jade - nephrite trap parameters ; investigations on artificial irradiation).

Except for scientific development, our investigations are made after official request of curators or archaeologists. As a public department directly supported by the Ministry of Culture, we work free of charge. Generally, sampling is made in our laboratory. The choice of sampling location is discussed with the curator in charge of the art work. It is sometime assisted by other curators. Our famous neighbour, the Louvre museum, has lot of specialists to help us. As a part of the Centre for Research and Restoration of French Museum, the luminescence dating unit works frequently in collaboration with restorers (public agents or private).

To reduce the impact of the sampling on the integrity of the object, we use mini-drill. We can work on little pieces, like terracotta stamps (poinçon). In all cases, we refuse to sample if it is not possible to obtain enough powder to made complete measurements. As destructive analytical technique, we balance between the scientific interest of dating and the aesthetic of the objects for public enjoyment.
In the last ten to twenty years, more and more ceramic objects regarded as ancient from the accompanying thermoluminescence analysis test certificates have come out of China and onto the art market. However, further testing has shown that in many cases they are recent products. The counterfeiting methods used are numerous - sculptures that consist of old and new parts, or that have been recently carved from an old, plain and worthless fired artefact are as frequently encountered as objects that have been made from a mixture of pulverized antique ceramics and modern binding agents.

Modern X-ray technology and particularly computer tomography mean that pastiches can be quickly recognized: clearly varying grey tones of different materials, straight cuts and glued areas as well as metal or plastic parts used for strengthening purposes are clear indications of manipulation and counterfeiting. Recently carved objects can be distinguished from objects modeled from unfired clay by careful microscopic examination of the surface. Ultimately, the binding agent test makes it possible to recognize products made from ‘artificial terracotta’. The binding agent most commonly found in fakes is polyvinyl acetate (also known as a wood glue with the brand name ‘Ponal’). This material can be clearly identified on the basis of its characteristic infrared spectrum and can be distinguished with certainty from materials used for surface treatment.

The presence of organic binding agents is not the only indication for manipulation of a material or the presence of ‘artificial terracotta’: in order to be able to improve workability as well as to alter colour and morphology, it is often the case that filling agents such as sand, iron oxide or ground stone are added to the above mixtures. As a consequence, proportions of elements other than in natural clay, which can be detected during chemical analysis, will obviously be found in the composition of the material of the object. Inorganic binding agents that are often used for the production of such imitations (for example water glass or cement) can be revealed by chemical analysis as a result of atypical alkali-silicon or calcium-silicon proportions.

Another good way of detecting unusual properties of terracotta objects which can indicate that they are not genuine is an examination of the material’s morphology using electron microscopy. When subjected to the appropriate magnification, fired ceramics are found to have a completely different appearance from ‘artificial terracotta’ produced from ground fragments and binding agents.

When assessing terracotta objects, the methods described above mean that it is again possible to ensure a degree of certainty that cannot be guaranteed by thermoluminescence analysis alone. Thus, material analysis with (in the event of a positive result) subsequent determination of the age by means of thermoluminescence is currently the best way of ensuring a reliable assessment of such art objects.
Six years after the exhibition *Della Robbia XV–XVI*’s (Paris–Nice, 2002), the Centre for Research and Restoration of French Museum (ministry of the culture and communication) and the Louvre museum decide to publish all laboratory investigation made on della Robbia works (Bormand, Bouquillon, Zucchiatti, in press).

In 1979, one of the first TL dating request by Mr. J.-R. Gaborit, general curator in charge of sculpture department of the Louvre museum was for a della Robbia’s piece. Since 1994, the luminescence dating unit was involved in della Robbia project.

The interest to investigate large series is to reduce the uncertainties. The project on the della Robbia’s bottega (workshop) is a good example.

The result presented in this communication is a compilation of laboratory dating since fifteen years. In fifteen years, we investigated some 70 terracotta from the Louvre and other French collections by luminescence dating (thermoluminescence and optical stimulated luminescence). During these years, our equipments as well as our protocols vary.

As collection’s pieces, only the internal concentration in radioelements could be directly measured. For some fifty pieces, radioelement content was measured by ICP-MS and ICP-AES. The others were measured by alpha counting and X-ray analysis (EDS-SEM or PIXE).

Some old TL measurement cannot be exploited, especially concerning alpha efficiency and supralinearity. As they are physical parameters due to minerals and crystal characteristics, we assum that they vary very few in an homogen set like the works of della Robbia. We have remplaced missing values by arbitral typical values: alpha efficiency = 0,130 in TL; 0,060 in OSL; supralinearity = 0,1 Gy en TL ; 0,0 Gy en OSL.

The palaeodoses are mainly about 1.8-1.9 Gy. Less than three pieces show palaeodoses close to 0.0 Gy (modern copies).

To estimate the contribution of the external environment, we reconstruct the moisture and gamma dose rate. For the gamma dose rate, we assume an average value. An average radioactive soil has a gamma dose rate of 0.9 mGy/a. But as wall decoration or church altar, della Robbia’s faiences receive less gamma rays and more cosmic rays than object stored in soil. Accordingly, we corrected the external dose rate.

Applying such values of the external dose rate to our della Robbia set, we obtain an age distribution showing a large maxima XIV–XVI c.

Luminescence dating gives valuable criterion to discriminate the fakes from the genuine. But it is not useful to discriminate between the different members of Della Robbia’s family.

Figure 1: probability density function of TL and OSL dating of della Robbia, limited to the sample dated by both methods.

Figure 2: luminescence date of artwork attributing to della Robbia family and other Renaissance artists.
(P17) OPTICAL DATING OF THE PALÄOLITHIC CAVE SITE OF XIAOGUSHAN, NORTHEASTERN LIAONING PROVINCE, CHINA

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The Xiaogushan cave site is one of the most important Late Pleistocene-Holocene sites in northern China. The site (N40°34'45.9", E122°58'26.0" 161m WGS84 elevation) is located at Liaodong Peninsula in northeastern China, ~140 km away from Shenyang City, Liaoning Province. The cave as an archaeological site was found in 1981, and began to be excavated in the summer of 1983. The cave is situated near the base of cliffs, and its entrance faces west overlooking the Haichen River from south to north. Its floor is about 1 m high above the present river bed. The cave wall consists of dolomitic marble. The cave deposits are mainly composed of soil, sand, gravel and marble fragments, and can be divided into five layers. At least 38 species of mammalian fossils including Mammuthus primigenius and Celodonta antiquitatis, and cultural remains including stone artefacts, bone and antler implements and ornaments, were excavated from the cave. The cave deposits were dated using radiocarbon dating techniques, and a total of 19 radiocarbon dates were obtained. The maximum radiocarbon age obtained is up to 46353±1179 cal. yr BP which reaches the upper limit of radiocarbon dating. In this case, other dating methods are required. In this paper, six sediment samples (Lab no.: L1091 (Layer 1, bottom), L1092 (Layer 2), L1093 (Layer 2), L1094 (Layer 2) from section A, L1095 (Layer 2) and L1096 (Layer 3) from section B) from the cave were dated using optical dating techniques.

Silty homogenous sediment samples were collected from the unexcavated sediment remains. Fine-grained and coarse-grained quartz extracted from the samples were used for luminescence measurement. The luminescence properties of the quartz extracts were investigated. The De values of these samples were determined using the single-aliquot regenerative-dose (SAR) protocol. Preheat plateau and dose recovery tests were carried out in order to determine suitable preheat conditions in the SAR procedure. All luminescence measurements were performed on a Risø TL/OSL reader. U, Th and K contents for these samples were analyzed by ICP and NAA methods.

The results shows that the fine-grained and coarse-grained quartz have different luminescence properties such as TL peaks, OSL and LM-OSL decay curve shape and dose-response curve shape, as well as preheat plateau region. The former are much brighter than the latter. The difference in luminescence properties between fine- and coarse-grained quartz extracts should be attributed to their different sources. The fine grains appear to be of eolian origin, and brought to the cave by human activities. In the nearby hills, loess deposited >10 m in thickness were found and the ages are older than about 124 ka (optical ages). The luminescence properties indicate that both fine- and coarse-grained quartz are suitable for De measurements using the SAR method.

The dose rates calculated using the results of ICP and NAA analyses are consistent within error limits, suggesting that the analyses of U, Th and K contents are reliable. The fine-grained quartz OSL ages for samples L1091-L1096 are 77.8±10.0, 56.8±4.9, 30.2±2.8, 35.1±2.4, and 53.5±4.3 and 28.2±2.2 ka, respectively. The coarse-grained quartz OSL ages for these samples are 67.7±3.7, 62.9±4.0, 26.6±1.6, 30.3±1.7, 48.7±3.9 and 19.4±1.1 ka, respectively. The latter are slightly smaller than the former except for sample L1092. We consider that the difference in OSL ages between fine and coarse quartz should be attributed to different degrees of bleaching of different size grains. Some fine grains were brought to the cave in the form of aggregates. The residual OSL signals from these grains might result in the overestimation of burial dose. For sample L1091 from the bottom (Layer 1, fluvial sediment) of the cave deposit, the coarse-grained quartz OSL ages may be affected by their luminescence properties, because some aliquots showed OSL saturation when regeneration dose is ~300 Gy, but for the fine grains, this was not observed.

Based on comparison to the calibrated radiocarbon ages, we considered the optical ages should be reliable. The cave deposits can be dated using optically stimulated luminescence dating methods.
(P18) ANALYSIS BY THERMOLUMINESCENCE (TL) OF ZAPOTEC CERAMICS OF THE CAXONOS RIVER BASIN, OAXACA, MEXICO

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Introduction: Social disciplines have long recognized the important role played by external contacts and trade on the developmental trajectories of societies. For all the theory models, understanding the degree of communication between human groups is a critical step in examining ways which diffusion or trade influenced cultural circumstances. These questions are quite important, especially when we study the exchange phenomena in Mesoamerican societies. In this case, our main interest is the connection between the valley of Oaxaca and the Gulf coast of Mexico. In the early period (1500-500 B.C), the route that has been proposed to get into contact between these two regions was the Canyon of Cuicatlán. Nevertheless, around the 500 AD there was another route through the Caxonos River Basin. This basin like the Cuicatlán Canyon is a passageway that crosscut the Sierra Madre Oriental and connected the eastern portion of the central valley of Oaxaca and the coastal plain of the Gulf of Mexico. Even that this region was quite important for the Zapotec people of the Classic period (300-800 AD), until now we did not have any serious analysis from the ceramic of this region or a chronological sequence of occupation. Since 1996 the Caxonos River Project began an exhaustive survey that covered 4000 km². Between 1998 and 2000 we started to excavate the principal sites of this region. A total of 6561 sherds were analyzed in which came to be identified 17 types, divided in three large groups. It was denominated Group to the collection of types related one to the other and present a consistency in form and color of wares that can be contemporaries. In the same manner, the category Group can be reflected its origin, local or foreign. The nomenclature is: wares that can be considered proper to the Sierra (Sierra Group), wares related to the Central valley (Valley Group) and wares similar to those found in the coast of the Gulf (Gulf Group). But now it was necessary to establish an appropriate chronology in order to prove that this was an exchange route and relationships between the Central Valley of Oaxaca and the Gulf Coast of Mexico since the Classic period until the Spanish conquest. The natural conditions of the areas is humid forest, and most of the archeological sites that we explored are in the mountain range, we could not recover to many charcoal samples. In this way, thermoluminescence analysis was the method to solve the chronological sequence. The objective of this paper, is present the result of 19 samples from different archeological sites of the Caxonos River Basin analyzed by TL. In this analysis, we also introduce the first chronological set of dates of this important area of Mesoamerica.

The thermoluminescence technique used was the fine grain technique. A 2 mm layer was first removed from the surface. The remaining sample was crushed and powdered in an agate mortar. The sample was treated with oxygen peroxide to remove the organic material and clorhidric acid to neutralize carbonates. The grains sized from 4 to 11 µm (fine grains) were separated and deposited in aluminum discs. Annual dose, for ceramics we use an alpha counter to calculate the potassium and thorium concentration and flame photometry to calculate the potassium concentration, in soil we use a gamma ray spectrometer to calculate the uranium, thorium and possassium concentration besides cosmic , to calculate the paledose were used the additive method to calculate the equivalent dose “Q” and regenerative method for supralinearity factor “P”, the plateau test was done par due to aditive method to ensure that the natural TL signal do not present a considerable fading.The samples were irradiated with a beta source of 90Sr. Once obtained the annual dose, equivalent dose and supralinearity factor the values were replaced in the age equation.

References:
Recently, a new isochron method (iIRSL) was proposed which uses the infrared stimulated luminescence signal from potassium (K) feldspar grains of different diameters (Li et al., 2008a). In this method, an isochron is constructed by plotting the natural radiation doses received by K-feldspar grains in a range of grain sizes as a function of their internal dose rates derived from the decay of $^{40}$K and $^{87}$Rb in the crystal lattice. This method was shown to have the potential to overcome the problems of anomalous fading (Li et al., 2008a) and changes in environmental dose rate (Li et al., 2008b). A method has been developed further using single K-feldspar grains. A physical mechanism is proposed for explaining the phenomena of apparent no anomalous fading shown by the internal doses. The potentials and limitations of the method are discussed.

Fossil evidence with firm chronological control for the presence of Homo sapience in central China earlier than 30,000 years has been lacking so far. Bones have been found together with *in situ* stone tools in Salawusu site. It is more than 1000 km away from the coast in Central China. Here we present an age of 55,000 years for a sediment layer containing stone tools, dated with iIRSL method. This age is consistent with the early part of the relatively warm interstadial period of Oxygen Isotope Stage 3 and is consistent with the dispersal of *H. sapiens* out of Africa, across Asia and into Australia by or before 50,000 years ago. However, the northerly location of Salawusu suggests that a single, coastal, dispersal through southern Asia was not necessarily the only or earliest route taken during the human diasporas. The age of 55,000 years at Salawusu has provided evidence for the earlier initial palaeolithic stone tool assemblage in Northeast Asia.

References:
TOWARDS A SINGLE-ALIQUOT REGENERATIVE DOSE PROCEDURE, SUITABLE FOR USE WITH PULSED OPTICALLY STIMULATED LUMINESCENCE

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The most common contemporary technique for quantifying the dose absorbed by natural minerals is the single-aliquot regenerative-dose (SAR) procedure described by Murray and Wintle (2000, 2003) and reviewed by Wintle and Murray (2006). The method was developed for use on the initial transient signal measured in continuously optically stimulated luminescence. We report on attempts to develop an analogue of SAR using pulsed optical stimulation where the luminescence is measured in the absence of stimulating light. Relying on the basic premise that the latent luminescence signal is erased during every measurement, the pulsed technique uses the dynamic luminescence throughput \( \mathcal{Z}(t_1) \), that is, the amount of luminescence detected after the pulse as a fraction of the total measured luminescence which from theoretical considerations of time-resolved luminescence (Chithambo 2007a,b) is given by

\[
\mathcal{Z}(t_1)=\frac{\tau}{t_1}[1-\exp(-t_1/\tau)]
\]

where \( \tau \) is the luminescence lifetime and \( t_1 \) the pulse-width. The method is used to develop the optimum measurement conditions to address typical problems encountered in repeated sequence of measurements such as sensitivity changes and the need to improve the accuracy attainable on the determined dose.

References:
The limitation of the fast optically stimulated luminescence signal as a chronological tool for ages up to ~ 400 ka, has led to the investigation of other luminescence signals, in particular one component of the thermally transferred OSL (TT-OSL) signal (Wang et al 2006a, 2006b). TT-OSL is the signal observed when quartz grains are optically bleached and then heated. Under the combined action of thermal activation and optical stimulation, electrons trapped at very deep traps (VDT) are liberated to the conduction band and recombine emitting photons. The aim of the present work is twofold. Firstly, to investigate the efficiency of TT-OSL form VDT of unfired quartz samples of various origin in which the VDT are occupied by electrons due to geological irradiation and secondly to investigate the properties of VDT in samples fired at very high temperatures in order to empty all the VDT. The study was performed at 8 geological quartz samples of Turkish origin. All unfired quartz samples exhibit TT-OSL signal in a wide region of OSL measuring temperatures, namely up to 375 °C. It was found that the TT-OSL signal depends strongly on the type of quartz sample. After annealing, the TT-OSL signal much more intense. The TT-OSL curves consist of at least three well defined components, for which the same nomenclature was used, namely termed fast, medium and slow components. All these well defined components exhibit an excellent behavior as a function of dose in the dose region between 0.5 and 300 Gy. The implications of all these properties on dating over 400 ka are also discussed.

Figure 2: The behaviour of the three TT-OSL components as a function of dose in the case of two different types of fired quartz samples.

References:
Wang, et al. 2006(b). Quaternary Geochronology, 1, 89-100
Recent developments in instrumentation for luminescence dating measurements have allowed a more detailed examination of issues related to the dosimetry of single grains in situ (Greilich et al., 2002; Bailiff, 2006) and the effect of heterogeneity in the sample environment on the determination of the palaeodose, $D_e$. In parallel with this experimental work the application of radiation transport modelling techniques has provided a better understanding of the likely extent of the spatial variation in dose rate (Mayya et al., 2006; Nathan and Mauz, 2008). In the work described in this paper, the spatial variation of $D_e$ and $\beta$ dose-rate was investigated experimentally for coarse grain samples using a combination of experimental and computational modelling techniques. Spatially resolved determinations of $D_e$ were performed with sliced resin-encapsulated sediment samples using an OSL scanner (Bailiff, 2006) and compared with the results of determinations of $D_e$ using multiple and single grain measurement procedures. Spatially resolved determinations of external $\beta$ dose rate were obtained using an array of external dosemeters (~1 mm dia.). The extent to which variation in $\beta$ dose rate potentially contributes to the variation in $D_e$ is examined by comparison of experimental data and also in combination with the results of modelling calculations based on simulations using the radiation transport code MCNP. The extent to which these and other experimental approaches can be employed as a tool to investigate the degree of heterogeneity in dating samples is examined using several case studies.

References:
Alpha counting techniques are widely used for dose rate determination in Luminescence and ESR dating applications. Since in many cases the most part of the natural radiation dosage absorbed by the crystals of the samples to be dated is due to the U and Th series, an increased accuracy is required. It is also known that the dose rates calculated by alpha counting are more accurate even in the case of an unknown disequilibrium. Nevertheless, an approximate knowledge of disequilibrium increases the accuracy of the calculated dose rates.

The new technique for the calculation of U and Th based on the alpha particle spectrum taken from a thick sample by using a silicon detector (PIPS) is briefly described (Michael et al., 2000).

The basic equation of this technique is the following

\[ N_m = \frac{4n_{\text{Int}}}{R_1 - R_2} \]

Where \( N_m \) (Bq/g) gives the number of alpha particles emitted by the radionuclides of the U and Th series (activity concentration, a.c.) per unit mass per unit time (s) with energies higher than a certain value \( E \) (MeV); \( n_{\text{Int}} \) is the integral of counts per unit surface (cm²) per unit time (Ks) in the energy interval between \( E_1 \) and \( E_2 \) and \( R_1, R_2 \) (mg/cm²) are the ranges in the sample corresponding to \( E_1 \) and \( E_2 \), respectively. This interval can be anywhere between \( E \) and the next lower energy of the emitted alpha-particles of the U series and the dotted of the Th series. The rapid increase of counts below 1.8 MeV is due to \( \beta \)-particles and electronic noise.

Within the present study two major advantages of the technique will also be presented: the potentiality for detecting and providing an estimation of disequilibrium in U and Th series (Michael et al., 2008), -when present- for especially young sediments, and the potential use of the technique as a new method for isotopic dating of speleothems and other materials.

References


The luminescence properties of natural quartz have attracted considerable experimental interest because of their practical use in retrospective dosimetry and in archaeology for dating ceramics and sediments. The thermoluminescence of all peaks that are associated to the $[\text{H}_2\text{O}_4]_o$ recombination centre in natural and synthetic quartz samples, namely at 95–110, 150–180, 200–220 as well as 305–325°C (depending on the heating rate) is characterized by thermal quenching. This phenomenon causes several methods of trap depth determination to give spuriously low results which erroneously imply thermal instability of the latter two peaks which are suitable for dating.

In this study the thermal quenching properties of the thermoluminescence (TL) from several synthetic and natural quartz specimens of different origins were examined, using the heating rate dependence of the TL signal, according to Nanjundaswamy et al. (2002). As the heating rate varies the TL peaks shift to different temperatures and become affected by thermal quenching to different extents. In case of artificial irradiations, all samples were previously annealed at 900°C in order to recapitulate sensitivity of the aforementioned peaks. In this work the heating rate was varied over almost two orders of magnitude (0.25 – 20 K/s). Through an analysis of the glow peak areas as a function of glow peak temperature the behaviour of the efficiency of TL production was monitored as well as compared to the theoretically expected according to the Mott-Seitz mechanism after using the values of 0.64 eV and $3 \times 10^7$ for the quenching parameters $W$ and $C$, firstly suggested by Wintle (1975). The investigations focused to unearth common characteristics in that behaviour. However, the results indicated that efficiency change due to thermal treatments are sample dependent, especially in case of the 110°C TL peak. In particular, the behaviour of the latter fails to agree with the standard thermal quenching behaviour based on thermal quenching parameters $W$ and $C$ previously reported. The work provides sufficient evidence for the need of reconstruction of the thermoluminescence signal of quartz, according to the experimental method previously suggested by Dallas et al. (2008) in the case of $\text{Al}_2\text{O}_3$:C. Implications on dating are also briefly discussed.

References:


ON THE SPECTRAL LUMINESCENCE EMISSION OF WHITE MARBLE AND THE CASE OF AIAINI (GREECE)

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Simple assumptions occur in the study of ancient marbles, such as: (1) the thermoluminescence technique (TL) provide a natural clock when a marble surface is exposed to the sunlight, setting the clock to zero, while in a burial environment the clock counts years of age (Polikreti et al., 2003) and (2) under ultraviolet (UV) light, a recently worked marble usually exhibit an even red–violet colour, whereas antique sculptures are distinguished by a spotted bluish-white colour (Rorimer, 1931). Moreover the exact causes of the fluorescence patterns and colours have not yet been related to specific marble properties. The recent advances on the modern spectrally-resolved luminescence spectrometers let us analyze and compare both, blue and red luminescence emissions from white marble. The environmental scanning electron microscopes (ESEM) joint wide chambers and electronic conduction by water vapour allowing to analyze hand artifacts without metal covering. The non-metallization improves the hot-cathodoluminescence (ESEM-CL) effectiveness from the light emission centres.

Figure 1: Spectral TL plots of White Marble Patterns (Univ. of Sussex, U.K.): (a) Fresh Marble from a quarry without blue emission; (b) Altered Marble from an Archaeological artifact.

Here we analyze spectra luminescence and provenance of archaeological and geological white marble specimens from Aiani (Greece) by ESEM-EDS-CL and learn on the spectra luminescence bands of white marbles, cluster relationships between defects, exploring potential relationships between both archaeological specimens of white marble and their geological counterparts taken from the neighbouring quarries.

Figure 2: Spectral Cathodoluminescence analyses at room temperature of archaeological (AK) and geological (Tranovaltos, Latomio, Zooodochos) white marble specimens of Aiani (Greece)

References:
Rorimer, J. J., (1931) Ultraviolet rays and their use in the examination of works of art, Metropolitan Museum of Fine Arts, Boston. US.
Time-resolved luminescence spectra from natural quartz measured using pulsed optical stimulation can be resolved into components with distinct principal and secondary lifetimes (Galloway et al., 2002; Chithambo et al., 2008). The relative importance of the principal and subsidiary lifetime components depends on combinations of measurement and annealing temperature. In particular, the lifetimes are affected by prior thermal treatment as has been discussed elsewhere (Bøtter-Jensen et al., 1995, 2003). The lifetimes, which denote the delay between stimulation and emission of luminescence, are independent of annealing up to about 500°C and then decrease thereafter with annealing temperature. Figure 1 shows an example of such behaviour in sedimentary quartz with the change of lifetimes $\tau(T)$ from the higher value $\tau_H$ to the lower one $\tau_L$ described as

$$\tau(T) = \tau_L + (\tau_H - \tau_L)/[1 + C \exp(-W/kT)]$$

where $C$ is a constant, $k$ is Boltzmann’s constant, $T$ is the absolute temperature and $W$ is the activation energy above the valence band of a non-radiative recombination centre involved in the luminescence emission process (Galloway, 2002; Chithambo et al., 2008).

In this paper, we report on the relative features of the principal and subsidiary lifetime components in volcanic quartz. The lifetimes were studied as a function of measurement, beta irradiation and preheating. Luminescence lifetimes were evaluated from time-resolved luminescence spectra measured from samples stimulated at 470 nm and studied in the ‘fast’, ‘medium’ and ‘slow’ components of the luminescence. The dependence of lifetimes on measurement temperature is described in terms of competition between radiative and nonradiative processes whereas the influence of preheating and irradiation on lifetimes is described in terms of the relative dominance of various luminescence centres brought about by changes in hole concentration at the centres.

Figure 1. The dependence of luminescence lifetimes on annealing temperature in quartz (Chithambo et al., 2008).

References


Flash photolysis is a probe laboratory technique, in which a sample is firstly excited by a strong pulse of light ($\lambda_{ex}=345$) from a laser of picosecond pulse width. This strong pulse results in an increased population of energy levels other than the ground state within a sample. Typically the absorption of light by the sample is recorded within short time intervals to monitor relaxation initiated by the pulse. The main aim of this work is to apply this technique on bone material and to examine whether it can be used for characterization or dating purposes of archaeological or paleontological bone material.

We used two specimens of bone: one modern human bone and one fossil animal bone a couple of millions years old to see whether there were any differences in their emission spectra in the laser flash photolysis spectrometer. Bone powder was used drilled from the midcortex of each bone. The specimens were measured in room temperature and at 77 K for both fluorescence and phosphorescence. At 77 K the emission is far stronger than that in room temperature. For phosphorescence there was a strong emission at ~500 nm from both specimens at 77 K (Figure 1) but very weak at room temperature. For fluorescence there was an emission at ~440 nm which increases with a decrease in temperature for the modern bone. This emission is nearly absent in fossil bone but increases on decreasing temperature (Figure 2). The intensity of fluorescence and phosphorescence at 77 K was lower in fossil bone. The resulted curves were smooth for modern bone and rough in fossil bone perhaps due to the diagenetic impurities in the fossil bone and/or its dark color. The described differences in the emission spectra between the two specimens might be due to higher contamination of fossil bone or to its lower concentration in phosphorus. More research is needed to determine whether the method could be used for characterization or dating purposes.

Acknowledgements
I thank Dr. Chris Lambert who should really have been a co-author but due to communication problems it was not possible to trace his whereabouts.

References:
Obsidian is a volcaniclastic mineral extremely hard to break. Due to this specific property, it was used in prehistoric Greece (and elsewhere in the World), in order to provide tools, weapons, knives and arrowheads. Prehistoric people traded obsidian from Melos Island of the Aegean Sea with mainland Greece, Asia Minor. The recent progress in obsidian hydration dating (Liritzis, 2007; Liritzis and Laskaris, 2009) and characterization (Delphi 2008 International Workshop) has gained an international interest. However, luminescence dating applications of obsidian are rarely reported in the literature (Göksu and Turetken, 1979). In the present work, preliminary measurements regarding basic thermoluminescent (TL) properties were obtained, using natural obsidian recovered from Melos, in order to evaluate its potential use in archaeological dating and retrospective dosimetry. This work aims to characterize this extremely precious tool stone by using both (TL) and (OSL) techniques and investigate its potential use for luminescence dating purposes. The natural TL (NTL) signal of obsidian was found to consist of one TL peak centred at about 340 °C (Fig. 1). Furthermore, NOSL is investigated in detail, and was found to consist of one component rather peculiar, whose shape is extremely flat with intensity as large as 20 times the OSL background signal. Bleaching of this NTL signal as well as natural OSL (NOSL) signal were both studied. NTL does not decrease, even after 20ks of illumination using both commercial blue LEDs as well as SOL-II simulator. The TL signal after artificial irradiations consists of several overlapping peaks, including those that are typical for quartz-dominated minerals, such as the 110 °C, 160 °C, 210 and 275 °C TL peaks. Therefore, the OSL was also studied by performing a series of thermal annealing experiment, in order to isolate the signal of interest resulting from the most appropriate for dating trap. Basic TL and OSL properties, such as TL and OSL thermal and optical stability, repeatability, TL and LM-OSL glow curve shape and mainly the linearity of the TL and OSL signals as a function of beta dose were investigated, along with preliminary fading test measurements for specific reference TL glow peaks, such as the 110 °C TL glow peak, etc. The extremely low bleaching ability of both natural TL and OSL along with the saturation of the natural TL peak, provide major difficulties in dating applications. Further work is required in order to seek for an alternative and suitable zeroing mechanism.

References:
An Etruscan vase from the Pushkin Museum of Fine Arts (Moscow, Russia) has been studied in the course of restoration work. The bucchero style vase dates back to approximately the 2nd–3rd century B.C.. Characteristically, the bucchero pottery is made utilizing a specific technique. It is most often black, sometimes gray, shiny as a result of polishing, and contains decorations usually incised or in a form of a relief.

Two available large fragments have been studied from the following parts of the vase: body (black-gray, sample 1, and black, sample 2), neck (dark-brown, sample 3), and later made restoration additions, represented by a handle (gray, sample 5) and fragments of decor (light-yellowish, sample 4a, light gray, sample 4b, yellowish-pinkish, sample 6).

As the first step, optico-petrographic analysis of all samples was carried out when samples were examined under a binocular microscope and characterized. Fragments from the original vase (samples 1-3) were studied using a photoluminescence technique. Quartz, feldspar, and calcite, present in these samples, luminesce under nitrogen laser ($\lambda_{\text{max}} = 337.1$ nm) pumping, with, respectively, light-blue, rich-blue, and red colors, characteristic for this excitation source (fig. 1).

![Figure 1: Luminescence image of sample 1; Qtz – quartz, Fsp – feldspar.](image)

The elemental composition was determined by X-ray fluorescence spectral analysis (RFSA) using microanalyser «FOCUS-M2» and emission spectral analysis (ESA) using inductively coupled plasma-atomic emission spectrometer (ICP-AES) (Baird, USA), and phase composition – by methods of microchemistry and X-ray phase analysis (RFA) on diffractometer X’Pert PRO (Philips, Holland). A summary of our results is shown in Table 1.

<table>
<thead>
<tr>
<th>Sample</th>
<th>RFSA+ESA</th>
<th>RFA+microchemistry</th>
</tr>
</thead>
<tbody>
<tr>
<td>1, 2</td>
<td>Si, Al, Ca, K, Fe, Mg, Na</td>
<td>Quartz, feldspar, clay; chalk, black organic pigment, sericite</td>
</tr>
<tr>
<td>3</td>
<td>Si, Al, Fe, Ca, K, Na</td>
<td>Quartz, feldspar, clay; organic and mineral black pigments, dehydrated white mica, iron compounds</td>
</tr>
<tr>
<td>4a, 4b</td>
<td>Ca, Fe, Si, Al, Mg, K, Na</td>
<td>Chalk, quartz, clay; Fe-oxides, sericite, black charcoal pigment</td>
</tr>
<tr>
<td>5</td>
<td>Ca, Si, Al, Fe, K, S, Mg, Na</td>
<td>Quartz, chalk, clay; gypsum, feldspar, chloride, iron compounds, sericite</td>
</tr>
<tr>
<td>6</td>
<td>Ca, Si, Al, K, Fe, Mg, Na</td>
<td>Chalk, clay, quartz; iron compounds, black mineral pigment and charcoal</td>
</tr>
</tbody>
</table>

Table 1: Chemical and phase composition of ceramics.

Our data show, that the samples under study have a similar chemical composition, but their elemental compositions are different. This is a result of specificity in the phase state of the dark ceramic of the original vase (body and neck, samples 1-3) compared with the light material of the restoration additions (other). For example, in samples 1-3 quartz, feldspar, and clay prevail, chalk is a secondary component, and iron-containing compounds were not detected. In samples 4-6, in addition to quartz and clay, we found significant amount of calcite (chalk) and traces of iron-containing phases. Notably, that ceramic mass of all restoration additions contains tiny shells Foraminifera, which were widely spread in modern (in the geological sense) seas of the Mediterranean area.

The results obtained suggest that restoration additions were made within the territory of modern Italy (Toscana).

Acknowledgment: We would like to express our deep gratitude to a colleague from Grabar Art Conservation Centre Elena Minina, who provided the samples.
Geoarchaeology deals with the application of geoscientific methods and concepts in order to understand the development of ancient landscapes as living space of past man. For the knowledge of the time-frame of depositional layers and archaeological artifacts as well as of the rates of geomorphic processes chronometric dating is essential. Commonly the radiocarbon method is used when organic materials are available, but in view of the predominance of clastic sediments the luminescence techniques are indispensable. Their latest methodological developments allow novel insights in the interaction between past cultures and their physical environment, which is demonstrated in few case studies.

The first is the Basin of Phlious in southern Greece, known for its thick colluvial and alluvial deposits from the Holocene which are well suited for optical stimulated luminescence dating (OSL). Combined with an archaeological survey, OSL dating of colluvia allowed reconstruction of the history of the Holocene soil erosion and its dependency on historical farming activities (Casselmann et al. 2004, Fuchs et al. 2004). There are periods of enhanced soil erosion which correlate with times of intensive land-use – a phenomenon which also has been observed at some OSL-dated sites in southwestern Germany (Lang et al. 1999, Kadereit et al., 2009).

Near Nasca and Palpa in the desert of southern Peru, which is renowned for its geoglyphs, a multidisciplinary project on climate change and its effects on the history of the Prehispanic cultures was performed. OSL-dating was applied to desert loess, indicating former grass vegetation and thus more humid climate (Eitel et al. 2005), and fluvial sediments. In addition, with the novel technique of spatially high resolution OSL-measurements (HR-OSL) of rock sections the age was determined, when a stone surface was for the last time exposed to daylight and, thus, when stone structures, such as the geoglyphs were built (Greilich and Wagner 2009). This technique was also applied to a diorite boulder from a debris flow and to single feldspar grains from a fluvial deposit (Kadereit et al. 2009).

Based on OSL as well as AMS-C14 ages the geomorphic and climatic history was reconstructed and correlated with the Prehispanic cultural development (Unkel et al. 2007).

References:
**THE CHIEMGAU IMPACT (GERMANY) OSL DATING PROJECT**

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**Introduction:** The samples come from the archaeological excavation site of Chieming-Stöttham, a location close to the eastern shore of Lake Chiemsee, in the very southeast of Germany. The excavation revealed a remarkable stratigraphy: a conspicuous mixed layer of up to 50 cm thickness is embedded between a layer with artifacts of the Late Bronze Age Urnfield culture (about 1200-750 BC) and a layer with a Roman pavement (about 2nd century AD). Geological and mineralogical research provided the evidence that the arguable layer is the relic of a meteorite impact, the so-called Chiemgau impact (Ernston et al. 2008). Among the Holocene meteorite impacts this impact stands out by the fact that the impact origin is well documented by shock metamorphism of rocks, by the size of the crater strewn field (about 2000 km²), the number of craters (about 80), the size of the biggest crater (600 m ø) and the variety of secondary effects (Ernston et al., 2009; Yang et al., 2008; Liritzis et al., 2009, in preparation). The stratigraphic situation of Chieming-Stöttham contributes another extraordinary aspect, because up to now worldwide no other case of an impact layer being directly embedded in an archaeological stratigraphy is known. Within the last two decades it has become a controversially disputed topic whether Holocene meteorite impacts might have influenced human civilisations. An answer to this question is very difficult for different reasons among them the very poor dating of some impacts in question. A more exact dating of the Chiemgau impact may provide the indispensable fundament for evaluating its cultural implications and thus enable an extraordinary case study (Ruppengluck & Ruppengluck, 2006, Ernstson et al. 2008).

**Samples & Dating:** OSL dating of four soil samples derived from the ~2 m deep excavated section related to the conspicuous diamicitic brecciated layer. The meteorite impact induced tsunami hypothesis presumably has bleached luminescence of quartz setting the deposited layer to zero. The SAR protocol was applied with the normal luminescence dating tests (sensitivity corrections, pre-heat tests, dose recovery). Etched quartz grains made more than two dozens and up to 80 aliquots per sample. ED ranged between 7 – 9.8 Gy in good agreement to the observed stratigraphy, provided ages at the turn of the 2nd to 3rd millennium BC (errors within 12-15%). Dose rates were measured by α-counting pairs technique, and K, Rb by SEM-EDX. Fig 1 shows an example of ED statistical evaluation.

**Figure 1.** ED determination for soil sample 5-3-M5 using the quartz extracts.

**References:**


Lithic microwaste (lithic debris <1 mm in maximum dimension) is produced during tool manufacture and rock engraving activity (Dragovich and Susino 2001). Microwaste persists initially on the ground surface and subsequently becomes incorporated into sedimentary deposits associated with human occupation (Fladmark 1982, Susino 1999, 2007). To understand the sedimentary history of archaeological deposits, the need is not only to understand the chronology of depositions and movement of artefacts (Villa 1982) but also the chronological independence of dating artefacts separately with the optical dating technique.

This research explores the application of scanning electron microscopy (SEM) to quartz microwaste, in tandem with optical dating of individual sand-sized grains of microwaste using the optically stimulated luminescence (OSL) signal. The SEM/OSL approach allows archaeological siliceous material produced during tool manufacture and rock engraving activity to be distinguished from natural sedimentary quartz. Optical dating can then be carried out separately on the two components to provide a chronology for sediment deposition and lithic manufacture – and, hence, the time of rock engraving activity – and to identify any post-depositional disturbance. Optical dating of microwaste provides a direct means of estimating the time of manufacture of lithic artefacts, and avoids the question of association between the ages obtained for the artefacts and the natural quartz sediments in which the artefacts are buried. It is, therefore, of particular benefit to mixed-age deposits. Further, it was found that the age of the sedimentary quartz and microwaste does not necessarily correlate, and that the microwaste and the sediments have different depositional histories.

Identification of microwaste was undertaken by comparing the shapes and surface features of microwaste grains with those of natural sediments (Krinsley and Doornkamp 1973). By analysing quartz grain-surface morphologies using SEM and optical stereomicroscopy, grains identified as microwaste could be distinguished from other sedimentary components of the deposit. The radiation dose absorbed by individual particles of microwaste since the time of last exposure to sunlight (taken as the time of lithic manufacture) was determined by means of the single- aliquot regenerative-dose (SAR) protocol.

This study has shown that SEM can be applied to quartz grains without any significant effect on the OSL signal, so that grains after SEM remain viable candidates for optical dating. Under subdued red illumination, 250–500 µm and 500–1000 µm microwaste particles were hand-picked for SAR analysis, taking into account their typically non-spherical shapes and medium- and coarse-sand sizes. The usual internal tests of SAR protocol performance were conducted, and the results were compared with those obtained for natural quartz grains from the same sample of sediment. In this presentation, these findings are discussed together with some of the practical problems – and solutions – regarding the extraction of sufficient microwaste grains for dose distribution analysis, and briefly describe potential new applications of the combined SEM/OSL method to archaeological material other than quartz and to other environmental situations. For example, SEM can used be identify grains with characteristically aeolian or fluvial textural features, and individual grains can then be dated by OSL to check if the water-transported grains were bleached as fully as the wind-blown grains before deposition.

References:
(P33) THE TIMING OF FLUVIAL LANDSCAPE EVOLUTION AROUND ARCHAEOLOGICAL SITES IN THE UPPER Khabur BASIN (NE SYRIA): FEASIBILITY STUDIES USING OSL SIGNALS FROM FINE-GRAINED QUARTZ

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The tributaries of the Upper Khabur in northeastern Syria played an important role in the economical and cultural development of former human societies in this region. The regional fluvial environment largely determined settlement patterns and subsistence strategies; this, in its turn, influenced how man affected the landscape (such as through deforestation and irrigation). Because of anthropogenic forcing, the present day environmental conditions do not accurately reflect those prevailing at the time of archaeological occupation. To improve our understanding of the development and functioning of these settlements, it is therefore necessary to unravel their former environmental settings. This requires a chronological framework for the regional evolution of the fluvial landscape and climate.

The Upper Khabur basin in northeastern Syria is one of the better geomorphologically studies regions of the Near East, and its settlement history is well-documented through intensive archaeological surveys and excavations. The chronology of the evolution of the landscape, however, is poorly understood; this strongly hampers the interpretation of settlement histories into their forcing environmental context.

In this paper, we investigate the potential of optically stimulated luminescence (OSL) dating to establish an absolute chronology for the riverine environmental history of several well-studied archaeological sites in this region. Our study builds on the initial work by Deckers and Vandenberghe (2007) and aims at (i) documenting the luminescence characteristics of fine silt-sized quartz extracts, and (ii) examining the reliability of the ages based on these signals.

Fluvial exposures were studied near archaeological settlement mounds (tells) along the Wadi Jaghjagh (Tell Hamidi and Tell Leilan), the Wadi Jarrah (Tell Farsuk Kabir and Tell Brak) and the Wadi Khanzir (Tell Has). The granulometric characteristics of the sediment samples imposed the use of the fine silt-sized (4-11µm) fraction, from which quartz grains were chemically isolated by etching for 5 days in H2SiF6.

All samples were analysed using the single-aliquot regenerative-dose (SAR) protocol, and we discuss the performance of the protocol in terms of recycling, recuperation and dose recovery. For all samples, recycling ratios are indistinguishable from unity and recuperation is negligibly small (less than a few % of the corrected natural OSL signal). The ratios of the measured to the given dose (dose recovery test) generally do not differ by more than 5% from unity. Two samples from one section near Tell Leilan, however, give dose recovery values of 1.29±0.16 and 2.10±0.08; interestingly, the estimated burial dose in these quartz extracts is of the order of a few hundred Gy, which is in the region of the growth curve approaching saturation. Apart from these two samples, the values for the equivalent dose ($D_e$) range from a few Gy to a few tens of Gy, and are independent of the preheat temperature in the range 160°-260°C.

Optical ages are then presented for the sedimentary sequences. For most sections, the optical ages are consistent with the stratigraphic position of the samples, as well as with the available independent age control (radiocarbon dating and TL-screening of sherds, where available). Only one of the studied sequences shows evidence for incomplete bleaching, through stratigraphically inconsistent ages.

Our study illustrates the potential of optical dating to establish a chronological framework for Holocene landscape evolution and environmental change in the Upper Khabur basin, and we briefly discuss the results with respect to the region’s settlement history. We also observe, however, some site to site variability in luminescence characteristics and bleaching history; uncertainty about the latter is the main limitation as it is difficult (or even impossible) to examine the distribution of doses within fine silt-sized (4-11µm) samples.

References:

Burial mounds are among the most striking archaeological monuments in NW-Europe. Until recently, archaeological investigation unavoidably involved excavation resulting in destruction of the monuments. Geophysical research methods (ground penetrating radar, seismic tomography) can now be used to investigate the internal structure of burial mounds without excavation. Prospection of archaeological monuments may benefit from using non-evasive methods, although we wish to stress that extreme care is needed in the interpretation to avoid destruction of valuable monuments. There are two important advantages of non-evasive methods over excavation: 1) the mound is left undisturbed for future investigation, allowing future generations to use additional and improved research methods; 2) costly excavation and archiving is avoided. So far, excavation is still needed to obtain suitable material for dating; radiocarbon dating mostly uses charcoal fragments that are sparse in the mound structure. In this study we investigate whether it is possible to determine the time of construction of burial mounds without excavating, using Optically Stimulated Luminescence (OSL) dating methods. Most important question is whether the OSL signal of sand grains in the burial mound was reset at the time of construction.

Our samples are from an Iron age burial mound at Oss Zevenbergen, in the southern Netherlands. The burial mound was partly excavated, allowing excellent control on its internal structure and providing suitable material for radiocarbon dating to validate OSL dating results. The mound is build of soil sods, which were stacked up-side-down on top of a small natural dune. We expect the surfaces of the sods to be exposed to light during construction of the mound. For equivalent dose determination we employed the Single-Aliquot Regenerative dose method on small aliquots of sand-sized quartz. First, preliminary measurements were made on a large number of samples taken in a vertical profile. Second, we sampled the sod transitions for full OSL analysis. In addition, we dated an OSL sample from an infilled Medieval grave dug into the mound.

Results from the OSL profiling study confirmed our hypothesis that sod surfaces were better bleached than the inside of the sods. In addition, results showed that the top 20 cm of the mound yielded lower OSL ages, likely due to Medieval disturbance. The palaeosurface underneath the stacked sods returned very consistent results, in line with the expected mound age. Full OSL analysis on samples from the sod transition yielded scattered equivalent dose distributions, indicating that not all grains were exposed to light during mound construction. Using advanced statistical methods we extracted the youngest results; these were internally consistent for the different samples and yielded OSL ages in excellent agreement with the radiocarbon age control. Also the OSL dating results on the Medieval grave infill were in line with radiocarbon results.

Our results suggest that OSL dating can be used to determine the time of construction of a burial mound built of stacked sods, provided that samples are taken from sod transitions. Using our methods, it is possible to determine the age of a burial mound by drilling a sediment core through the structure, and dating OSL samples from the core. Care should be taken to take samples from the most suitable intervals. Our results show that OSL methods can also be used to detect later disturbance.