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DATING BY LUMINESCENCE OF ANCIENT MEGALITHIC MASONRY

IOANNIS LIRITZIS*
ASIMINA VAFIADOU

Laboratory of Archaeometry, Dept. of Mediterranean Studies, University of the Aegean, 1 Demokratias Ave., Rhodes 85100, Greece

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e-mail: liritzis@rhodes.aegean.gr
** to whom all correspondence should be addressed*

ABSTRACT

An updated abridgement of the novel thermoluminescence (TL) technique that dates monuments made by large stone blocks, as well as stone objects, is made. 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 down to a variable depth. During exposure for a time interval the sunlight sets the light-sensitive TL clock to zero. If the stone is made of granite, basalt, sandstone rock types, the sun-exposed interval in minutes is enough to erase luminescencent traps. However, for calcitic stones this period varies between several hours to dozen of hours. In the latter case a technique is devised to estimate the bleaching time in antiquity, and thus the residual value, which serves as the 'zero time' upon which luminescence builds up until today.

The total archaeological equivalent dose or total luminescence acquired by the minerals in the particular stone is measured by Optical Stimulated Luminescence (OSL) techniques, while for calcitic stones only thermoluminescence is at present applied with involved errors of the order of 7-15% (Liritzis 2001). A new quartz / feldspar technique for limestone monuments is presented enabling their dating by established single aliquot / single gain OSL methodology.

Application examples from Greece covering the period 3rd millennium to classical time, and the potential to dating world megalithics is explored.

KEY WORDS: Thermoluminescence, optical luminescence, dating, bleaching, megalithics, granite, limestone

INTRODUCTION

The advent of the novel thermoluminescence technique that dates megalithic monuments (cyclopean walls etc) and stone objects has been well documented (Liritzis 1994; 2000; Theocaris *et al.* 1994; 1997). This paper reviews the dating of rock surfaces, and occasionally their underlying soil surfaces, by using calcite, quartz and feldspar. (Liritzis *et al.* 1996; 1997b; 1999; Liritzis 2000).

The determination of the age of stone structures and artifacts (tools, monoliths, buildings, cairns, field walls etc.) using physical methods is notoriously difficult. Age estimates almost always use material associated with the construction period, rather than material directly from the fabric of the construction, in particular by association with ^{14}C datable material, and in many cases appropriate organic debris is either not available, or the association is insecure.

Luminescence is filling (by environmental ionizing radiation) and release (by heat or optical / IR light) of electrons from electron traps.

Two methods of luminescence are employed, a) Thermoluminescence Dating, which is the thermal eviction of electrons from traps by heating to 500°C, and b) OSL Dating, i.e. the radiation eviction of electrons from traps with various wavelengths (optical and IR spectrum).

Datable materials include: ceramics, burnt soil/rocks, plasters, quietly deposited sediments, loess, palaeosols, stone buildings, statues. They date the last event, which caused release of electrons from traps within minerals.

OPTICALLY STIMULATED LUMINESCENCE (OSL)

OSL can be used to determine the time elapsed since certain minerals, such as quartz and feldspar, were last exposed to daylight. It is now widely used in the dating of geological sediments such as aeolian, marine and fluvial sand and muds, loess, and colluvial materials over the last 200 ka (recently reviewed by Murray and Olley 2002; see also Liritzis 2000; Liritzis *et al.* 2002). It also has many applications in archaeology and anthropology (Galbraith *et al.* 1999; Theocaris *et al.* 1997). As well as dating the geological sediments providing the burial context in archaeological deposits, it can be used directly to date artifacts, such as, ceramics, pot boilers, fireplaces, and even rock art, and wasp nests in caves (Liritzis and Galloway 1999; Liritzis *et al.* 1994; Roberts *et al.* 1997).

In OSL dating, the amount of trapped charge within the crystal structure is used as a measure of time; this charge accumulates as a result of the dose rate from exposure to the natural radiation flux. The trapped charge can be emptied by exposure to heat or light; exposure to daylight empties previously trapped charge, and the longer this exposure the more complete the zeroing or bleaching process (Fig.1). In the laboratory the same

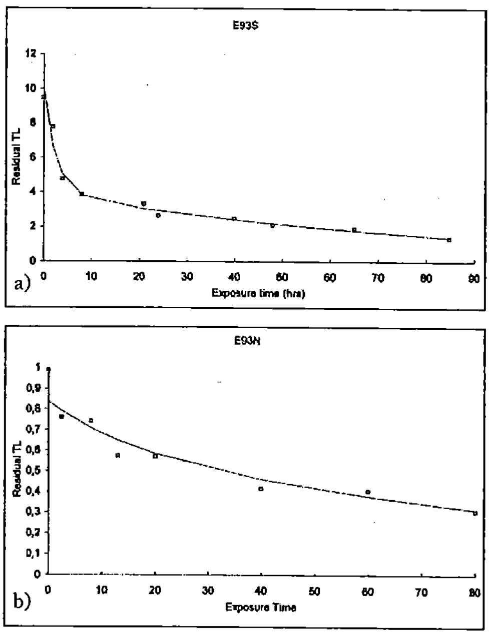


Fig. 1: Bleaching of thermoluminescence in calcites from Hellenikon pyramid (E93S, E93N) (Liritzis *et al.*, 1996).

process is used to measure the charge population – when the crystal is exposed to visible or infrared (IR) light under controlled conditions, the release of charge is accompanied by release of ultraviolet (UV) and visible light, as the charge recombines and gives up energy. Because this light (luminescence) is stimulated by exposure to light it is called optically stimulated luminescence. The trapped charge population is measured in terms of the dose absorbed by the crystal lattice, and methods for measuring this dose have undergone rapid development in the last 10 years. Almost all measurements are now based on so-called single aliquot methods (Duller 1995; Liritzis 1995; Liritzis *et al.*, 1994; 1997a; 2001; Murray *et al.* 1997a), where all the measurements needed to determine a dose are made on one sub-sample (aliquot) of the material to be dated. Though a single grain method is also valuable in case of sparse mineral content (Murray and Roberts, 1997).

Based on the above procedures an alternative strategy was introduced, which dates the last time a stone surface was exposed to daylight (Liritzis 1994; Theocaris *et al.*, 1997; Vafiadou *et al.* 2005). If the construction subsequently overlaid this surface, then this approach should provide a direct method for dating the time of construction.

The dating of large slabs of rocks refer to: Limestones / marble (calcite, $CaCO_3$), by TL, and, Granite / basalt / sandstone (quartz, feldspar), by either OSL or TL, though OSL is preferred.

None available technique is yet known to measure the ED in calcites by OSL (Galloway 2002).

In fact, granitic, basaltic and sandstone rocks contain the same minerals (quartz, feldspars) that are used for dating geological and archaeological sediments, and many rock surfaces are exposed to daylight for very long periods of time before being exploited, often for much longer than the time for which sediments were exposed during transport. Thus, it is likely that in many cases, all the mineral grains in at least some of the surfaces of stones used by ancient masons will have been exposed to daylight sufficiently to have been completely zeroed, and thus provide an accurate chronometer.

THE RATIONALE OF DATING CARVED MEGALITHIC STRUCTURES

The eviction of electrons from electron traps in crystalline materials via thermal agitation or light stimulation and their refilling from ionizing radiation is the basis for resetting of luminescence clock of TL and OSL dating methods (Liritzis 1995; 2001; Liritzis *et al.* 1997b; 2002; Aitken 1998; Murray *et al.* 1997a).

A variant of TL and OSL has already been developed for dating large carved limestone blocks (megalithic, cyclopean) of Greek monuments (Liritzis 1994; 2000; Liritzis and Galloway 1999; Theocaris *et al.* 1994; 1997).

The extension of this idea to the dating other carved rock types from ancient (particularly prehistoric) monuments, such as, granite, basalt and sandstone, has proved possible. (Liritzis *et al.* 2002).

Indeed, during the cutting and carving (or sculpturing) of a megalithic block and prior to both its emplacement in the appropriate position in a wall being overlaid by another large megalith, the solar radiation (UV and optical spectrum) bleaches the optically sensitive electron traps in the carved surface down to a variable depth (depends upon the rock type and opaqueness, but from 0.5 mm for limestones to about 5 mm in granites) (Liritzis and Galloway 1999; Vafiadou *et al.* 2005). During exposure for a time interval the sunlight sets the light-sensitive TL clock to zero. If the stone is made of granite, basalt, sandstone rock types, the sun-exposed interval in minutes is enough to erase luminescencent traps. For calcitic stones this period varies between several hours to dozens of hours. In the latter case a technique is devised to estimate the bleaching time in antiquity, and thus the residual value, which serves as the 'zero time' upon which luminescence builds up until today (Fig.2).

Thus the luminescence dating technique of megalithic structures (coined optical thermoluminescence) concerns the inter-block surfaces of building blocks and relies on the Optically Sensitive Electron Traps (OSET) responsible for TL in the surface layer of the carved limestone block having been bleached by sunlight, prior to the blocks being incorporated into the structure. Certainly, the same mechanism of solar setting applies to statues found buried under sediment. The exposure time of the surface to sunshine depends upon the efficiency and time for the stonemasons to put any block in the appropriate place overlaid by another. From the moment that any surface is no longer exposed to sunlight, then the OSET are filled by electrons produced by the ionization caused from nuclear radiation of natural uranium,

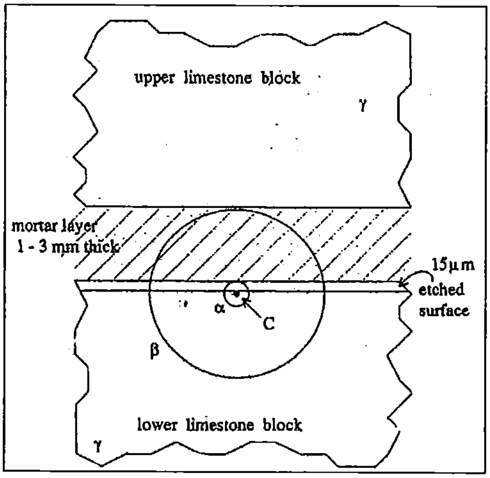


Fig. 2: Principle of dating megalithic monument. Sampling is restricted to the inner surface layer of carved block, which is exposed to alpha, beta and gamma radiation from the immediate environment (Liritzis *et al.*, 1997b).

thorium, rubidium, potassium, and the cosmic radiation. These isotopes are present in the rock slabs and the soil surrounding the sampling point.

The OSL probes the OSET (blue light probes quartz and feldspar, the IR stimulation probes only feldspar), while the TL probes the Thermally Sensitive Electron Traps (TSET) but the OSET as well. However, deconvolution of OSL decay curves allows analysis of such composite curves into individual components i.e. regarding number and type of OSET in the material (Kittis *et al.* 2002).

Age Equation-Equivalent Dose and Dose-Rate

The age of a carved rock in a monument is found from the relationship,

AGE = equivalent dose (ED) / annual dose-rate (ADR) (1)

Where, the ED Equivalent Dose (in Units of Grays), measures the total exposure to radioactivity accumulated by the sample, and the 'dose-rate' is the (assumed constant) annual rate of exposure.

The ED is determined by the Additive dose procedure, or the Regeneration procedure.

Of the most popular procedures are a) the Single Aliquot Regeneration (SAR) and b) the Single Aliquot Additive Dose (SAAD) (Fig.3).

The OSL method has been improved with the introduction of the 'single aliquot technique' and similar approaches and relevant correction procedures, which uses one disc prepared from the sample to carry out all the measurements to determine ED (Duller 1994; Galloway 1993; Liritzis *et al.* 1994; Murray *et al.* 1997b).

The ED increases with time in proportion to the number of trapped electrons. In the laboratory, the trapped electrons can be evicted by heating (TL) or by momochromatic light (OSL): usually green with quartz and infrared with feldspars, though green probes both quartz and feldspars. These electrons recombine with luminescence centers to emit

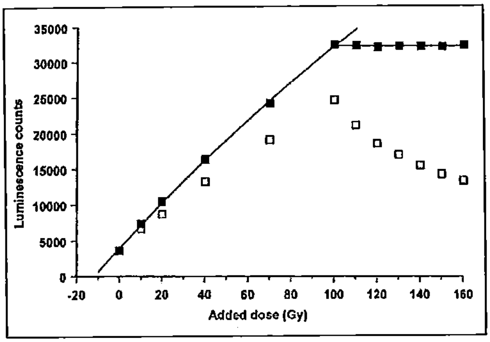


Fig. 3: Single-aliquot additive-dose procedure for the determination of equivalent dose for sample OS7 from Osirion, Abydos, Egypt. Dose against luminescence (squares) and corrected points (filled squares) (Liritzis *et al.*, 1997a; Vafiadou, 2005).

light of a characteristic wavelength, the intensity of which is measured (Liritzis 2000).

The '**dose-rate**' comprises of the alpha, beta and gamma radiation doses from the natural radioisotopes of uranium, thorium, potassium, rubidium and the cosmic radiation, as well as the environmental gamma ray dose-rate (i.e. $kD_\alpha+D_B+D_\gamma+D_c$), where k is the sensitivity ratio of beta to alpha particles in inducing luminescence.

The dosimetry here requires particular consideration regarding the three types of nuclear particles. The **alphas** deriving from within about 25 micron range deliver the full dose to the sample (the remove of the surface by dilute hydrochloric acid up to tens of microns does not affect the administered alpha dose in the powdered sample from a sampling depth of half to one mm for limestones or more for less opaque minerals). The **betas** are, a) half the total, deriving from one side, plus, b) half the total if the other side is plaster or weathered sediment, or c) zero for thin air layer. The original surface is often covered by thin layer of dust or fine sediment, and/or moss, which is removed by a few times fast dips in dilute HCl acid bath and washing with running water in a repeated manner to secure clear surface and preservation of the datable layer. The **gammas** derive from the environment, mainly from within a 35 cm radius from sampling point, but includes skyshine from air and soil. In this type belongs the cosmic radiation too. In limestone environments the gamma is very important and its accurate determination is essential. One should bear in mind that the antiquities are buried often covered by soil / sand during antiquity, where the present gamma ray dose rate reading is not representative of the gamma radiation geometry the sample has experienced during the past centuries. Collaboration with the excavators may help, whilst particular care should be exercised with the geometry of radiation (Liritzis 1989). Determination of dose rates is made via dosimetry or counting techniques and conversion of U, Th, K, Rb contents to absorbed doses (Liritzis and Kokkoris 1992; Liritzis *et al.* 2001).

SAMPLE PREPARATION AND MEASUREMENTS

Amongst the processed samples in Table 1, all but two (nos.7, 8, Table 1) are calcitic. The original surface was cleaned in red room conditions with diluted HCl acid to remove dust, and any organic residues. Then gently remove a thin layer from the surface in the form of powder, deposit in acetone bath and collect fine grains, wash in dilute acetic acid, and dry. For no.7 and 8 quartz and feldspar grains were removed by grinding down the soil and cutting sliced disks from no.7 and etching to remove alpha contribution (Vafiadou *et al.* 2005).

SOL and solar bleaching of natural and geological samples were made for various time exposures. Then the dose-plateau test (Liritzis *et al.* 1997b) was applied by subtraction of the residual bleached TL from the growth curve of additive dose procedure, for temperature region 250-400°C, and the ED was determined by extrapolation of the corrected points.

The gamma-ray dose-rate was measured by a calibrated in radioactive pads portable scintillator, and HpGe for alphas and betas of the rock.

DATING CRITERIA

The criteria applied to examine their dating suitability are fourth-fold;

a) bleaching of luminescence after exposure to sunlight or solar simulator. Total or partial bleaching by sunlight may occur; the former presumes complete zeroing of luminescence in antiquity and this occurs in statues and slabs made of quartz / feldspar; the latter presumes incomplete bleaching, thus a technique must be devised to determine the ancient residual luminescence (e.g. quick construction of limestone monuments). The latter is determined with the dose-plateau test, that is, subtraction of residual TL glow curves, after sun-exposure over variable lengths, from the natural TL, and determination of ED as a function of glow temperature, then select that residual TL glow-curve, which provides the longer dose plateau. This is the residual TL during ancient exposure to sun. The correct ED due the (unknown) incomplete bleaching prior to the construction can be determined also from radial plots (Fig.4). By plotting dozens of ED, from single grains removed from the surface, in a radial manner, the incompletely bleached grains form outliers along the trendy distribution of the bleached grains.

b) growth of luminescence with irradiation (following the regeneration and additive dose techniques).

c) fading, pre-dose and recuperation effects must be checked for,

d) effective sensitivity correction of dose recovery by OSL due to thermal heating and pre-heating and/or optical shinning.

These criteria have been investigated with detailed luminescence and radiation measurement tests on a variety of rock types (Liritzis *et al.* 2002).

The identification of mineral present can be made by XRD, while, quartz and feldspar, are also identified by probing with IR and blue LED. For some dating results, the regeneration and additive dose techniques were applied, followed by sensitivity change corrections from preheat and read, for the equivalent radiation dose determination.

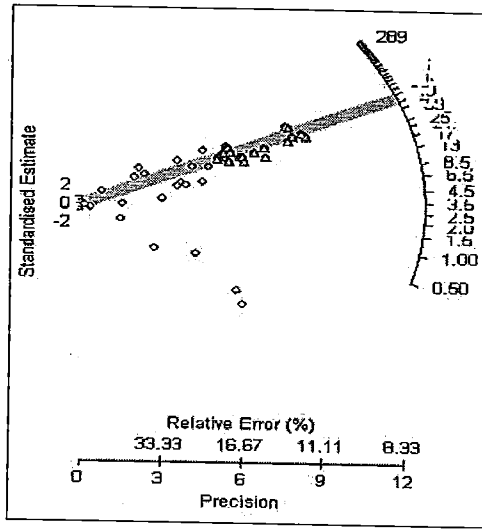


Fig. 4: Radial plot of soil sample from Ftelia Neolithic floor, Mykonos (see, also Fig.8 below) (Vafiadou *et al.*, 2005; Liritzis *et al.*, 2002) for the determination of equivalent dose and recognition of possible partial bleaching of some grains, as dispersion from main linear loci. Average dose in this case is 41±3 Grays. (Vafiadou *et al.*, 2005; Vafiadou, 2005).

The problems encountered with TL of calcites led us to development of another approach, the identification and separation of quartz grains from limestone masonries. Below this new approach is described.

QUARTZ FROM LIMESTONE: A NOVEL TECHNIQUE

XRD results on three limestone verified presence of traces of quartz (Table 2). Diluted 3.7% HCl acid was used, in a proportion of 50ml acid for 1g of powder. The powder was washed for 5-6 hours. The first attempt was made on sample BTL1 (megalithic block house in Fichtia near Mycenae, Peloponnese, Greece) in natural light. XRD analysis had shown that the sample is a calcitic sample with only 3% of quartz. The treatment to remove the calcite goes as follows: initially 20g of the sample were collected and washed in 1000ml of 3.7% HCl acid for 6 hours. The solution was centrifuged several times to retrieve the residual, which was about 1.3g. Microscopic analysis has shown that the sample consists by other mineral too, but quartz (eg. biotite, mica, feldspars etc.). In the case of mica (biotite, muscovite) and other phyllosilicic minerals treatment with $Na_2S_2O_7$ is performed (Kiely and Jackson 1964; 1965). In case of feldspars separation should be made without affecting the quartz either by concentrated HF (40%) for 40min in fraction 180-250μm, or diluted HF (5% or 10%) for 80-120min, or H_2FSi_6 (35%) for 30h or longer (Berger *et al.* 1980; Rees-Jones 1995; Prasat 2000; Robert and Wintle 2001; Stokes *et al.* 2003a,b; Mauz and Lang 2004; Syers *et al.* 1968). Confirmation is made by IRSL read out, and a proper OSL-blue measurement follows, with a single aliquot (Murray and Wintle 2000) or a single grain technique with highly accurate results. (Duller *et al.* 2000; Murray and Wintle 2000).

Two more samples (Mycenae, Greece, MTL3 and Valley Temple, Egypt, VT3) were processed under red light condition. A quantity of 0.1g from each sample was washed in 5ml of 3.7% HCl acid for 4 hours, then dried and examined by polarized light microscope. More minerals were observed than initial XRD analysis have had shown. Residues were processed and OSL readings were obtained from quartz and feldspars (Vafiadou, 2005; Liritzis *et al.* 2005).

The above technique has shown that such separation is possible and the expected single grain or single aliquot OSL ages of limestone buildings can be made more accurate. In case of feldspars presence, IR is applied, accompanied by fading test.

DATING EXAMPLES

We present some examples of monuments and archaeological cases dated with the technique of luminescence.

These include, a) Temple of Apollo, Delphi, b) Efpalinion ditch, at Samos island, c) the two Hellenic pyramidals, d) a peeble and underlying sediment at neolithic settlement Ftelia, Myconos island, e) Mycenean wall at Mycenae, Greece, and f) a polygon wall at Amfissa, Phokis, Greece.

In all but one case (the pyramidals) there is a consensus between the luminescence and archaeological dating, within the errors.

In the case of the two pyramidals the age is much higher than hitherto expected. These two structures were used for a long period Hellenistic to early Christian era, a period of about 1000 years. However, there are apparent reconstructions and repairs, and a plethora of finds during these periods. Nevertheless, prehistoric ceramic was unearthed from within the Hellenikon pyramid, as well as from test excavations of outside walls (Theocaris *et al.* 1994; 1997). Moreover, mythological reports by ancient traveler Pausanias (2^{nd}c. AD) transmit the construction to the heroic era and the twin brothers of Proetus and Acrissius, who fought and then erected this pyramid to honor the dead attaching their shields upon the walls. They were grand-grand sons of Inachus, progenitor of peoples in Argolid, during his time the homonymous deluge took place in Argolid. Geoarchaeological research at Argolid and a numerical estimation of genealogies bring this battle at around 2450 B.C. (Liritzis and Raftopoulou 1999).

Monuments worth dating include: Maltese prehistoric temples, Sardinean tombs of Nuraggi, Stonehenge in Britain, Easter Island statues, various standing stones, such as dolmen and menhirs, while Egyptian monuments from Giza and Abydos are at the moment dated as part of a Project of Historical Dating.

SOURCE OF ERRORS AND ELIMINATION

The various sources that induce errors include:

1) Scattering of additive dose points for calcitic, which can be reduced by taking many measurements. However, the new technique of quartz extraction from limestone improves considerably accuracy in ED.
2) Low radiation fields result to poor accuracies in radiation dose rates. Gamma ray dose rate most important (apply more than one method) as well as, the counting geometry, accounting for possible sand / soil cover during the past.
3) Destruction of surface datable layer due either to, friction, dilution (porous and low hardness), weathering and erosion, development of salts and secondary minerals and moss/lichens. It requires meticulous examination, e.g. remove of inappropriate surface effects, choose sampling points with plaster, or discard.

The OSL dating errors on megalithics applying the quartz technique may range between ±3-7%, while the TL of limestones are higher, around ±7 to 20%.

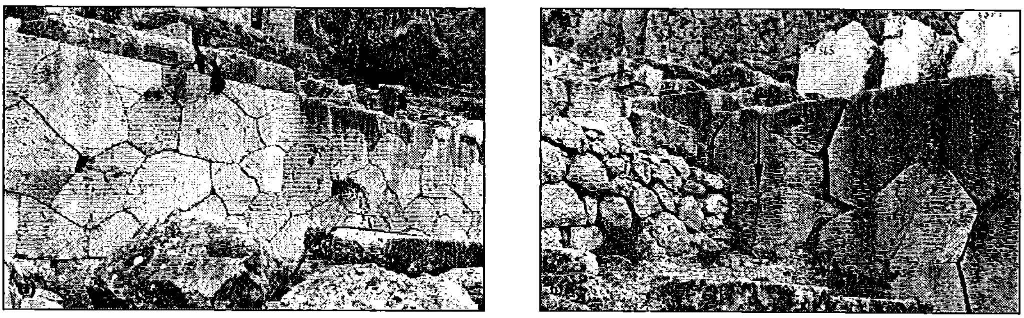


Fig. 5:Temple of Apollo, Delphi, a) polygon inscribed wall, b) western side of the wall where sampling was made (Liritzis *et al.*, 1997b)

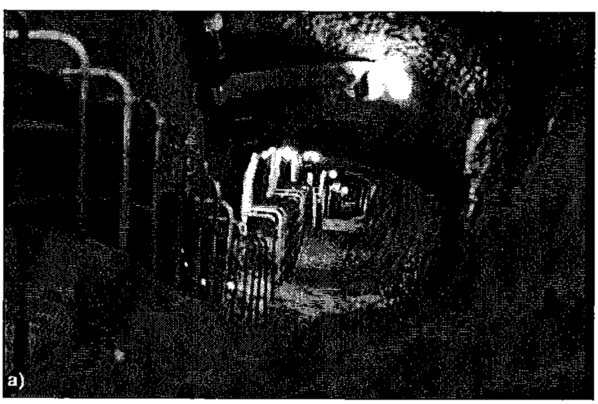
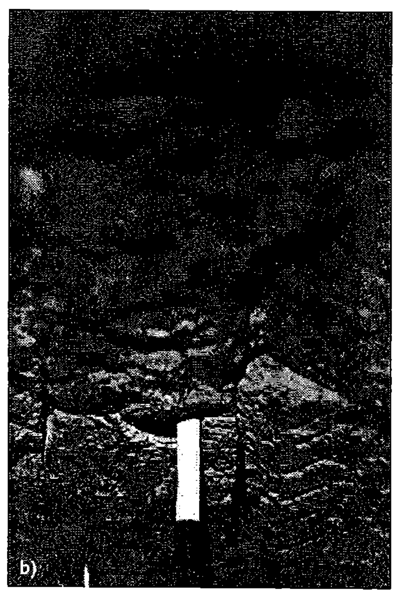


Fig. 6: Efpalinion ditch, Samos Island in the Aegean Sea. a) view of the mile long ditch from inside, b) sampling from the wall.



SITE	ARCHAEOLOGICAL AGE, years BC	LUMINESCENCE AGE, years BC
1) Temple of Apollo, Delphi (Fig.5)	550	470±200
2) Efpalinion ditch, Samos island, Greece, 245 m from entrance, SAM1(Fig.6)	530	570±300
3) Amfissa classical wall, Phokis, Greece, AMF2	5th C.	480±350
4) Hellenikon pyramidal, Peloponesse, Greece (Fig.7)	300-400?	2730±630, 720
5) Ligourio pyramidal, Peloponesse, Greece	300-400?	2260±700
6) Mycenan wall, Mycenae, Greece	1280	1110±340
7) Peeble, Ftelia Neolithic settlement, Myconos, Greece (Fig.8)	4500-5100	5800+/- 700
8) Soil floor overlied by the peeble (as above)	4500-5100	4450± 610

Table 1: Luminescence dating results of various monuments compared to their archaeological age.

NOTES per sample no. of Table 1.

(1) see, Liritzis *et al.* (1997b)

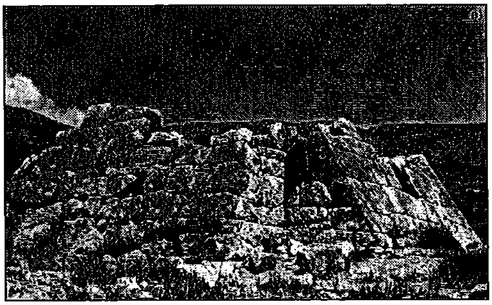
(2) Efpalinio. Bleaching times 1, 3, 7, 24 hours under SOL simulator (x7 for sun equivalent). Beta source strength at Edinburgh was 4.59 Gy/min, calibrated on quartz, for calcite a correction of 1.018 is applied. ED=6.76+/-0.3 Gy, ADR=3.80 mG/yr (Fig.9).

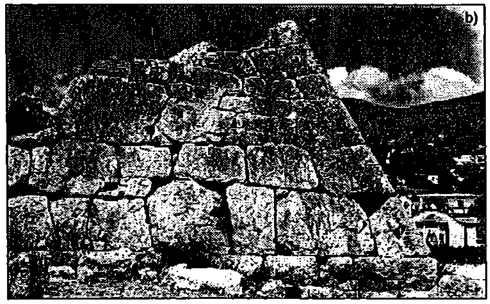
(3) Amfissa. Bleaching times 15 min, 1, 3, 5 hours under SOL simulator. (x6.3 hours for sun equivalent). The dose plateau defined an around 7 hours ancient bleaching, which provided an ED=7.70+/-0.2 Grays. From eq. (1) the ADR=3.10 mG/yr, and age=480+/-350 BC.

(4) & (5) see, Theocaris *et al.* (1994, 1997). The 630 yrs is the standard deviation of the mean ages, and 720 yrs the sd of their errors.

(6) see, Liritzis (1994). Here, the ancient exposure time to sunlight was estimated 15-20 hours, following the dose plateau test. Samples 2-6 were made at Edinburgh.

(7) & (8) See, Vafiadou *et al.* (2005) and Liritzis *et al.* (2002).





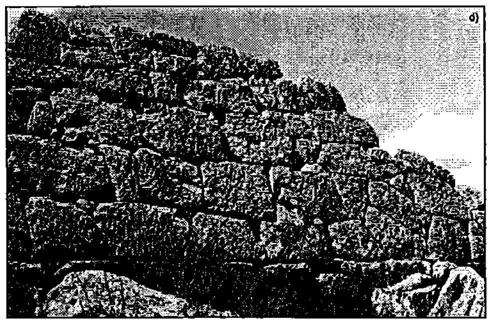


Fig. 7: Hellenikon pyramid, Argolid, Greece. a) View of the pyramid with eastern entrance, b) right corner, c) 8 m long entrance corridor, d) northern side wall.

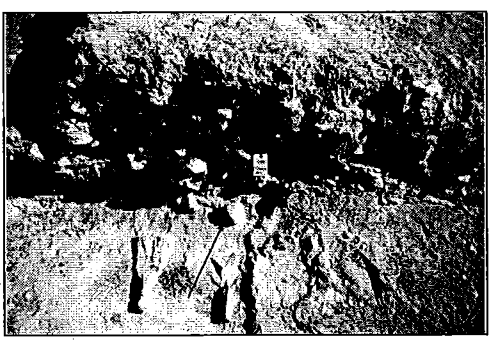


Fig. 8: Ftelia Neolithic settlement, Mykonos, Aegean Sea, Greece. Neolithic floor and sampling of sediment floor and overlying round stone.

Sample	XRD analysis*
BTL1	97% calcite, 3% quartz
VT3	100% calcite, traces quartz, dolomite, halite
MTL3	100% calcite, traces quartz
* School of GeoSciences, Geology and Geophysics, Grand Institute, University of Edinburgh	

Table 2 XRD analysis on limestones.

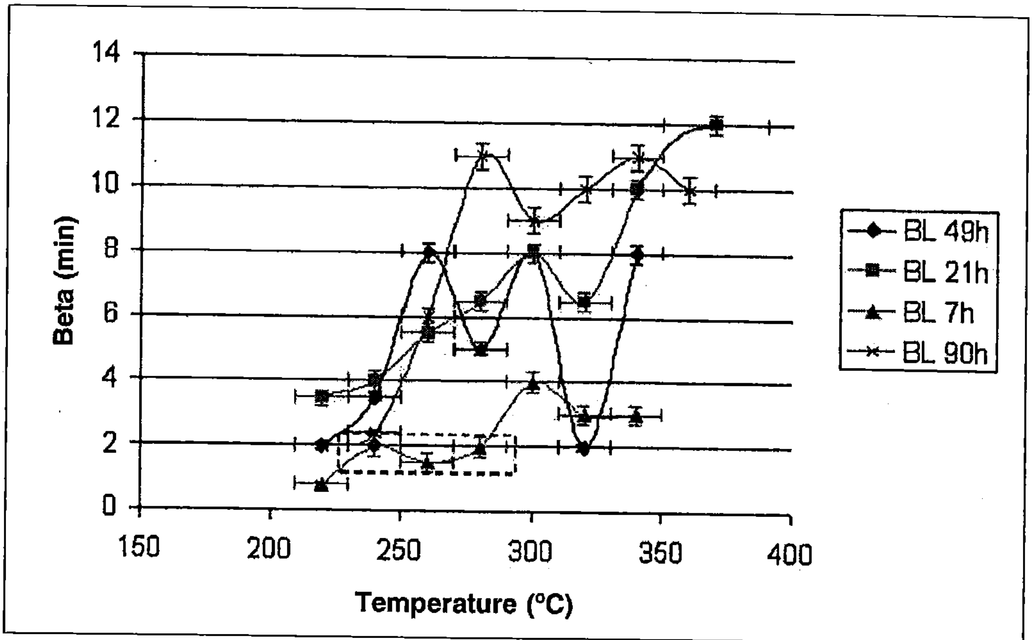


Fig. 9: Efpalinio ditch, Dose plateau test. It shows the equivalent doses (in mins of beta radiation) for different bleaching time (7, 21, 49 and 90 hours in sun) and for respective temperature ranges, as a function of the irradiation time in minutes of beta source. The best plateau is for about 7 hour exposure to sun shown enclosed in dotted frame.

CONCLUSION

The luminescence dating of megalithic monuments refers to the construction age of the masonry. As such it dates directly the construction.

It is the only direct method of dating these buildings, all other approaches are indirect (from architecture and context) and thus provide doubtful ages

Monuments made by rock types with quartz and feldspar minerals, are at present easier to date with small error (3-7%). For limestones the errors are higher (7-20%). However, in case that the limestone has quartz / feldspar, the OSL method can be used from extraction of the quartz (possibly feldspar as well) grains from the sample collected for dating.

Various monuments Greek and Egyptian (in preparation) have been dated with success, though it is recommended the dating of at least three samples per case study.

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