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SURFACE DATING BY LUMINESCENCE: AN OVERVIEW

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Abstract: Daylight radiation resets luminescence ‘clock’ to zero on rock surfaces, but transmission depends on the transparency of the rock. On burial, surfaces are no longer exposed to daylight and accumulation of trapped electrons takes place till the excavation. This reduction of luminescence as a function of depth fulfils the prerequisite criterion of daylight bleaching. Thus rock artefacts and monuments follow similar bleaching rationale as those for sediments. In limestone and marble, daylight can reach depths of 0.5-1 mm and up to 16 mm respectively, while for other igneous rocks e.g. quartz in granites, partial bleaching occurs up to 5mm depth under several hours of daylight exposures and almost complete beaching is achieved in the first 1 mm within about 1 min daylight exposure. The ‘quartz technique’ for limestone monuments containing traces of quartz enables their dating with Optically Stimulated Luminescence (OSL) techniques. The surface luminescence (thermoluminescence, TL or OSL) dating has been developed and further refined on various aspects of equivalent dose determination, complex radiation geometry, incomplete bleaching etc. A historical review of the development including important applications, along with some methodological aspects are discussed.

Keywords: luminescence, dating, artefacts, monuments, bleaching, OSL, TL, age.

1. INTRODUCTION

Determining the age of stone structures and artefacts (stone tools, monoliths, buildings, cairns, field walls etc.) using physical methods is increasingly more frequently performed, (Liritzis, *et al* 2010a; Liritzis, 2010; Greilich and Wagner, 2009). The application of thermoluminescence (TL) and OSL is used to date the most recent event when a stone surface was exposed to daylight. If soil cover subsequently buried this surface, then this approach can provide a direct method for dating the time of construction as well (**Fig. 1**).

Rocks including limestones, marbles, granites, sandstones, schists and basalts contain the same minerals 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. This is often for much longer durations, than the time for which sediments were exposed during transport. Thus, it is likely that in many rock surfaces of stones used by ancient people, all the mineral grains down a depth of some millimetres, will have been exposed to daylight sufficiently (from minutes to hours) to bleach their geological luminescence to a near zero value. Thus can provide a reasonably accurate chronometer (Liritzis, 2001; Liritzis *et al*., 1997b; Aitken, 1998; Greilich *et al*., 2005), and makes it possible to date different prehistoric monuments comprising carved rocks of varied types ranging from, granite, basalt and sandstone, (Vaz, 1983; Liritzis *et al*., 1996; Habermann *et al*., 2000; Greilich, 2004; Vafiadou *et al*., 2007; Richards, 1992; Liritzis and Galloway, 1999).

Exposure of a rock surface to daylight sets the light-sensitive TL clock to zero or to a near zero residual value. If the stone is made of granite, basalt, sandstone rock types, the sun-exposed interval in minutes is enough to

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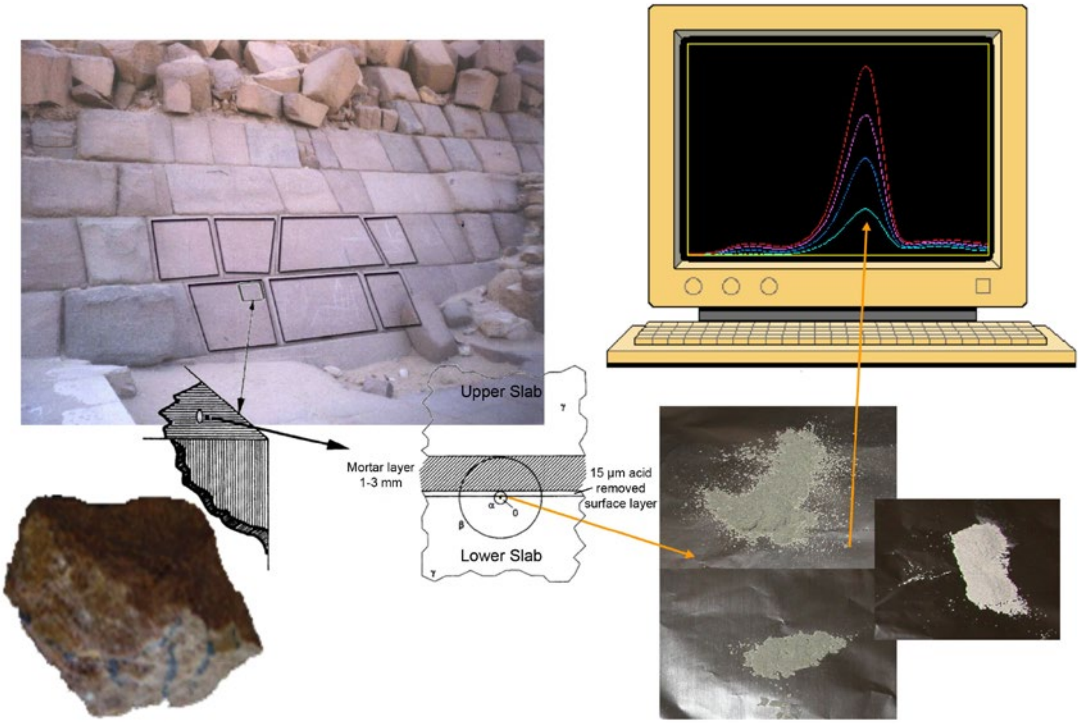

Fig. 1. *An example for sampling a megalithic wall (shown is Mykerinus pyramid Egypt), the actual and schematic piece cut, the upper inner surface and the radiation geometry involved, the removed powder and TL curves.*

erase luminescencent traps. For calcitic stones this period varies between several hours to dozens of hours. In the latter case a dose plateau technique has been devised to estimate the bleaching time in antiquity. Here the residual value, which serves as the base line signal at time t=0 upon which luminescence builds up until today is used. Alternatively, the extracted traces of quartz are measured by standard OSL techniques (Liritzis *et al.*, 2007; Liritzis *et al.*, 2010c).

A historical overview including case studies and tests regarding daylight bleaching and penetration at different rock surfaces is presented below.

2. HISTORICAL OVERVIEW

The development of the surface luminescence dating is based on the property of minerals bleached by sunshine to a level at or near zero. Luminescence of quartz and feldspars grains, exposed directly to daylight are bleached to zero within several seconds (Wintle and Huntley, 1980; Liritzis, 2000). This possibility was applied to examine the TL response of an archaeological stone sculpture by Vaz (1983). However, the possibility of dating buildings made of large stone blocks from their rock surfaces exposed to daylight prior to their setting in the wall was introduced by Liritzis (1994a, b). A critical requirement for successful measurement using surfaces is a well-bleached layer, thick enough to prevent less bleached parts of the rock material from being stimulated in the laboratory. For the latter, the deconvolution / transformation of OSL curves to TL glow peaks, usually provides three components that enable qualitative and quantitative information on the bleaching history of the sample. This bleaching depends on the mineral type and extent of a components contribution to the palaeodose is a useful test to identify more or less bleached components (Kitis *et al.*, 2002; Murari *et al.*, 2007).

A quartzite pebble was dated by Richards (1992), and doubts were expressed for the degree of sufficient bleaching of quartzite artifacts from site Diring Yuriakh (Siberia; Huntley and Richards 1997). Here lithic artifacts made of quartz (quartzites) were found in an excavated profile and TL as well as infrared stimulation (IRSL) was measured on the bottom of the rock that had not been exposed to daylight since its original deposition. The upper layers were exposed to light since they were recovered *in situ*. Experimental tests proved that light transmission through quartzites that bleached deep layers were at depths of several mm. Layers of quartz of thickness 0.25 mm down to 6^{th} layer (1.5 mm) and even lower, were measured by TL and IR (1.4 eV). Light transmission decreased by 1-2% at 1 mm depth, while daylight exposure for 1 year emptied all traps as deep as 2 mm. This suggested that the effect of light penetration with depth occurs as also confirmed the variability in differences in bleaching of different sedimentary quartz types (Richards

1992). Thus for example, 90% reduction in TL at 350ºC for 2 hrs daylight was seen in one case and it was only 50% for another sample. Dates of >75 ka for the 5th stratum by IR (1.4 eV) and ~150 ka the 6th stratum by TL are therefore in doubt. Also the self-gamma dose rate and the soil beta particle dose-rate contribution to the external top layers of quartzite are not accounted.

The first result of surface TL dating of the marble Temple of Apollo Delphi of c.550 BC was dated to 470±200 BC (Liritzis *et al*., 1997b) and two limestone pyramidal buildings at Hellenikon and Ligourio (in Argolid, Greece), thought to be Hellenistic times but TL dated to 2500-2000 BC on overlapping errors, were reported (Theocaris *et al*., 1997). In a recent volume on archaeology and politics, classicist Lefkowitz (2006: 195-202) has discussed latter dates on hypothetical pretentiousness. The loquacity on the pseudo-archaeology issues in general, is a self obvious and scientifically sound attainable consideration. However, her inadequacy in physical methodology, often encountered with academic historians, and/or missing detailed critical reports and finds on the published dates, as well as, misinterpretation of the novel effort, today widely accepted as surface luminescence dating, unjustifiably undermines unsuccessfully the new ages. Though at the end she remains skeptical about these older than thought ages.

The dates were based on powdered samples and TL of limestones. The development of spatially resolved OSL and low light CCD chips based confocal microscopy now can helps the measure of palaeodose, that avoid inhomogeneity and microdosimetry problems (Duller *et al*., 1997). The latter techniques have been refined with high resolution luminescence detection techniques (Greilich *et al*., 2002).

In 1999, feasibility of dating marble monuments and objects was established by studying daylight penetration and bleaching for different exposure durations (Liritzis and Galloway, 1999). This was followed by successful use of IRSL on granitic surfaces and by measuring depth profiling of remaining signal after exposure to a SOL-2 daylight simulator (Habermann *et al*., 2000). Attempts on surface dating by luminescence were also used to date fine-grained feldspars scraped from the surfaces of buried lithic artefacts of volcanic origin (flakes of basaltic trachyandesite recovered from site) of an archaeological site on the wet western slope of the Cascade Mountains of Washington. The excavated section was ^{14}C-sediment-dated due to lack of organic material related directly to cultural phases, and by U-series weathering rinds using ^{226}Ra in excess dating (Morgenstein *et al*., 2003). In this study two sets of powder grains successively scraped from the surface of sample UM630 were processed; the 90-212 μm aliquots gave low signal produced poor precision and inconsistent results and the fine grained 1-8 μm gave an IRSL dose of ~6 Gy and ~9 Gy for two samples using the single aliquot regeneration technique (SAR). Corresponding IRSL dates were 2310±230 yrs and 3347±330 years while a mixed polymineral sample gave 8.7 ka. The dating by U-series as well as indirect dating by calibrated ^{14}C of sediment profile suggests a range of 3500-9000 years B.P. The OSL dates were underestimated perhaps due to anomalous fading and/or complex bleaching characteristics of grains other than quartz and feldspars.

Bailiff and Mikhailik (2003) demonstrated the capability of a scanning technique to perform mapping of OSL from minerals located in the exposed surface of cut specimens and of a stimulation of selected areas to produce OSL decay curves.

In all samples gradients of relevant radiation parameters essential to OSL dating exist in all natural samples. Common techniques to control these gradients are not feasible for stone surfaces. Greilich (2004) presented a new approach for the estimation of D_e, addressing the complexity of dose rate assessment which includes preparation of samples, highly resolved (up to 25 μm) detection of optically stimulated luminescence by imaging using a CCD chip, and a software solution for data analysis. In this way, the area of measurement can be reduced to a size where the gradients become insignificant. A follow up of Greilich's PhD thesis, has been presented with encouraging results for archaeological stone structures. The experimental approach utilizes a high spatial resolution detection technique (HR-OSL) for OSL of minerals that are left in their original petrological context; that is, without any mineral separation. With this approach, steep gradients in microdosimetry at the surface and at grain boundaries become important and are discussed in detail. The new spatially resolved dating technique was successfully applied to the dating of stone surfaces from a stone wall of the medieval castle of Lindenfels (Odenwald, 12th century AD), Germany, and from the pre-Columbian Nasca lines (geoglyphs) around Palpa, in southern Peru (200 BC - 600 AD) (Greilich *et al*., 2005).

A summary of surface dating of various Greek and other monuments e.g. Efpalinion underground water Canal in Samos, 570±300 BC (arch. age 530 BC), a Mycenean wall at Mycenae to 1100±340 BC (arch. age c. 1280 BC), a Classical polygon wall near Delphi 480±350 BC (arch. age 5th cent. BC), was given by Liritzis and Vafiadou (2005).

Current research on surface dating continues with OSL (blue and IR) dating of cobbles (granitic, metamorphic, ultramafic) overlying sediment floors from archaeological sites of Greece. For example for Greece, Mykonos island, Ftelia settlement, dated to 5.8±0.7 and 5.45±0.61 ka BC for the cobble (granite) and sediment respectively (C-14 and ceramic typology gave a range of 4.5-5.1 ka BC). For Sweden 5.6±1.5 ka BC, for the ultramafic rock but underestimates for the sediment due to uncertainties in beta dosimetry. Finally, from a Danish site a modern stone-sample (quartz metamorphic). (Vafiadou *et al*., 2007). In this study, identification of quartz and feldspar was made using linear modulation BLSL after a standard preheat and IR stimulation. To understand the origin of OSL in polymineral rock samples, pulse anneal curves were measured on slice but this did not provide any characteristic decay curves with shapes comparable to granular samples, a cause due rather to the solid slice 1 mm thickness. In this case study the dose recovery and fading tests suggested that IRSL did not give reasonable results, but blue stimulation gives good results.

A new technique of OSL dating of limestones was presented that made use of traces of quartz extracted from limestone being used for the determination of D_e from a Mycenean wall, the Khasekemui Egyptian tomb and a Blockhouse in Argolid (Liritzis *et al*., 2008b Liritzis *et al*., 2010c. These applications followed a synthesis of OSL and TL properties of various rock types (granite, basalt, limestone, sandstone from Greek and Egyptian monuments) for the evaluation of equivalent doses. Single aliquot additive dose (SAAD) procedures (Liritzis *et al*., 2008a) already developed for ceramics (Liritzis *et al*., 1994, 2002; Liritzis *et al.* 1997a; Liritzis, 2001) were used. Recently, Greilich and Wagner (2009) summarized dating of well studied granitic stone geoglyphs of Palpa by High Resolution-OSL where a good agreement with archaeological and geomorphological reasoning was obtained. Dating of the ancient enigmatic 'Dragon Houses' at Styra, Southern Euboea, Greece, of hitherto unknown age, indicated it to be of Classical period i.e. 650±200 and 430±230 BC with apparent re-use at later times dated to late Hellenistic and Roman times (140-420 AD, 160-480 AD), Othoman (1460-1550 AD), Catalanean (1030-1245 AD) and, contemporary ages used by shepherds. These ages are in concordance with historical accounts (Liritzis, *et al*., 2010a). Finally, Strofilas prehistoric fortified settlement, Andros Island, in the Aegean, was dated by TL to 4th millennium BC with a similar age based on obsidian hydration dating using SIMS-SS novel method (Liritzis, 2010)

3. SAMPLING AND SAMPLE PREPARATION OF CARVED ROCK STRUCTURES

In dealing with dating of ancient buildings and buried sun bleached objects/cobbles certain procedures of sampling, sample acquisition and preparation are followed. These are:

1) **slices of drilled cores in granites.** In case of sun exposed rocks *in situ* sub-samples for luminescence measurements are taken by drilling cores from the rock surface using a diamond tipped coring drill, 6 mm internal diameter. Up to one mm thick slices are then cut from the cores using a low speed diamond tipped saw. (Greilich *et al*., 2005 and Vafiadou *et al*., 2007),
2) **surface powder.** In limestone, sandstone or other rock type, the original surface is often covered by thin layer of dust or fine sediment, and/or moss, and organic residues, which are removed by immersing the sample several times in dilute HCl acid bath for short durations and washing these with running water repeatedly to secure a clear surface while preserving the datable layer. A thin layer is then gently removed from the surface in the form of powder and is deposited in acetone bath and from this fine grains are collected, washed in dilute acetic acid, and dried. For other rock types, quartz and feldspar grains are removed by gentle grinding down the surface to a depth less than a few mm and if needed these are etched to remove alpha dose contribution (Vafiadou *et al*., 2007).

4. AGE EQUATION – EQUIVALENT DOSE AND DOSE-RATE

The age of a carved rock in a monument is found from the **Eq. 4.1**:

$$Age = equivalent\ dose\ (D_e) / annual\ dose\ (AD) \quad (4.1)$$

where the D_e 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 D_e is determined by additive-dose procedure or the regeneration procedure. The most used protocols are: a) the Single Aliquot Regeneration (SAR) Protocol (Murray and Wintle, 2000; Duller, 1995), and b) the Single Aliquot Additive Dose (SAAD) protocol, (Liritzis *et al*., 1997a; 2001; 2002). Introduction of the 'single aliquot technique' and 'single grain' approaches in OSL with and relevant sensitivity correction procedures now enable D_e estimation using a single disc or grain and in general several such discs/grains are measured for statistically valid D_e's, (Galbraith *et al*., 1999; Duller *et al*., 2000; Duller, 1995; Galloway, 1993; Liritzis *et al*., 1994; Murray *et al*., 1997; Murray and Roberts, 1997).

Prior to any D_e determination, daylight simulation lamp SOL and natural daylight bleaching of quartz/feldspar samples, either in powder or sliced form, is made for varying duration to estimate optimum time for daylight penetration and complete bleaching. For calcites devoid of quartz a dose-temperature plateau test is applied. This is cumbersome and results in high scatter and large errors (Liritzis *et al*., 1997b; Liritzis, 2010). TL glow curves after exposing to daylight for different durations are subtracted from the growth curve of natural TL. This is done for each temperature between 250-400°C. (**Fig. 2a**). The D_e is then calculated from the corrected glow curves using the **Eq. 4.2**:

$$De= ((N_{TL} - N_l) / (N_{+\beta} - N_{TL}))\times\beta \quad (4.2)$$

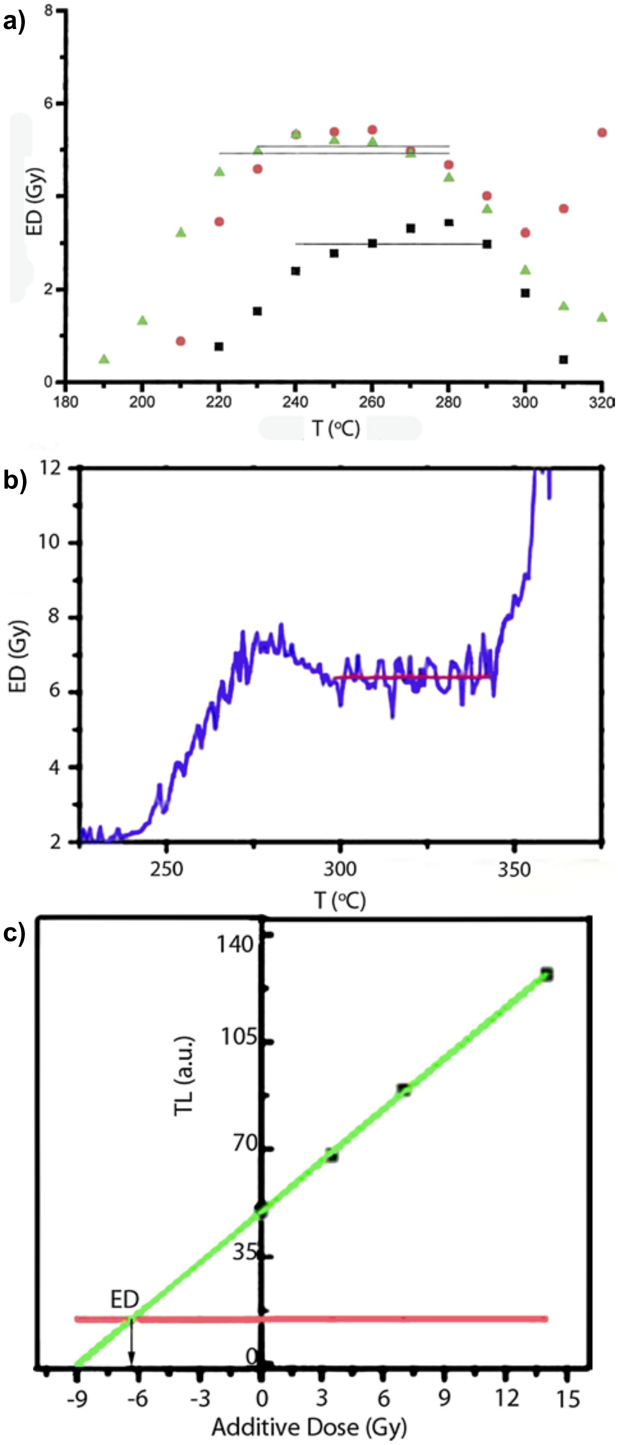

Fig. 2. *Dose-temperature plateau for Strofilas prehistoric settlement fortified wall. A) The symbols refer to natural (squares), 12 h (circles), 24 h (triangles) and 36 h (crosses) of outdoors sun bleaching. The longest plateau was for the 24-36 h exposure giving an average ED of 5±0.2 Gy, b) another example of recovery test with a simulation dose 7 Gy, and c) a representative dose response curve (filled squares) plot for the temperature of 270°C of the glow curve and 12 h bleaching for the earlier (b) sample of Strofilas. Horizontal line indicates the residual TL level after 24 h of bleaching. The arrow shows the equivalent dose of 6.51±0.6 Gy while the equivalent dose plateau yielded 6.61±0.5 Gy. (Liritzis, 2010).*

where N_{TL} the natural TL, $N_{+\beta}$ the natural TL added beta doses curves, β the administered beta dose in Gy, N_{bl} the bleached TL. Application of this formula assumes a linear response and this assumption is valid provided an average dose of 5.0±0.2 Gy, as was the case of our samples. A plot of these doses with temperature i.e. the dose-temperature plateau for various N_{bl} and the longest plateau which corresponds to the correct dose (**Fig. 2a, b**). A representative dose response curve plot for the temperature of 270°C of the glow curve for the sample STR1 is shown in **Fig. 2c**.

The problems encountered with TL of calcites led to the 'quartz technique' i.e. the identification and separation of quartz grains from limestone objects. (Liritzis and Vafiadou, 2005; Liritzis *et al.*, 2008b; Liritzis *et al.*, 2010c). This development make the OSL ages of limestone/ marble buildings/ artifacts more accurate. An example is the application to the limestone with traces of quartz from Valley Temple, Egypt (Liritzis *et al.*, 2008b). The regeneration procedure determined the apparent dose of ~28 Gy, while notable is the saturation starting at about 40 Gy. (**Fig. 3**)

The annual dose-rate (AD) determination requires particular attention. The alpha dose is within about 25 μm range the delivered dose to the grains. Removal of the surface by diluted hydrochloric acid up to tens of microns does not affect the alpha dose of the powdered sample from the sampling depth of half to one mm for less opaque limestones or some mm in marbles or 1-2 mm for granites and less opaque rocks. The beta dose contribution can comprise, a) half of the total 4π dose is from the sampled lower surface side and, b) half of the total 4π dose comes from the upper side is plaster or is a weathered sediment, and c) no dose for thin air layer and gypsum. In case e.g. of heated stone or cobble hit by a meteoric impact and covered by sediments, betas from (a)+(b) are calculated. The gamma dose is derived mainly from

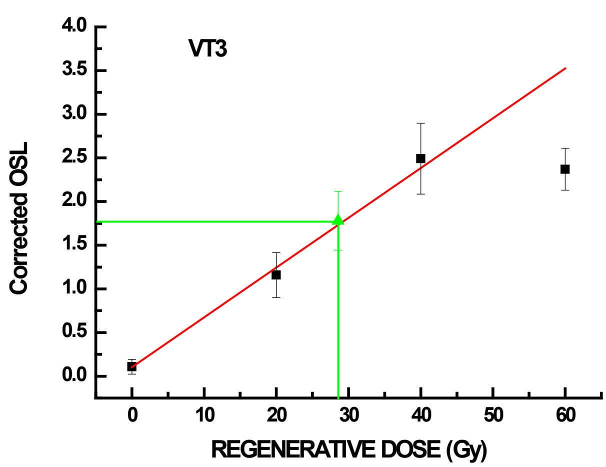

Fig. 3. *Regeneration of geological sample from Valley Temple of extracted quartz and determination of an apparent dose.*

rocks/sediment within a 35 cm radius of sampling point and includes air and soil with in this. The cosmic ray dose varies with geographical latitude, altitude and buried depth and can be calculated reasonably precisely. In limestone environments the gamma is important as being the major component and therefore, its accurate determination is essential. One should bear in mind that the excavated antiquities are often covered by soil/sand during antiquity, often mixed with breccia; thus, the present gamma ray dose-rate reading need not be representative of the gamma dose during the entire past. Collaboration with the excavators helps in optimally realizing the burial conditions in the past, none the less care should be exercised with complex radiation geometry regarding the surrounding material and radioactivity mixture (Liritzis, 1986; 1989; 2010; Aitken, 1985; Liritzis *et al*., 2001), and finally, corrections for possible U-series disequilibrium and updated conversion data are also called for, (Kokkoris and Liritzis, 1997; Liritzis and Kokkoris, 1992; Adamiec and Aitken, 1998).

5. BLEACHING AND PENETRATION EFFECTS

A basic characteristic of the surface dating is securing bleaching of the rock/rock mineral minerals under daylight radiation and the bleaching depth.

An example of bleaching for different hours is given in **Fig. 4** for the marble schists from the Dragon Houses at Styra (Euboea, Greece). The sample acquisition was made by gently removing powder with a file. During a 2 hr daylight transmission, bleaching as a function of depth increased and reached a plateau at around 1.5 mm (**Fig. 5**).

Reduction of luminescence under daylight as a function of depth from surface is shown for marble of Thassos quarry (northern Aegean island) in **Fig. 6**.

It is found that under SOL-2 illumination, the residual luminescence at the top and at 16 mm is 10% and 75±10% respectively for a few to about 40 hours of SOL2 exposure (**Table 1**). The former upper layers imply that the bleaching of marble causes drop of luminescence within dozens of minutes to hours, taking into account however, the residual signal caused by SOL exposure (1 hr SOL = 6 hrs sunlight). The deeper layers indicate that daylight penetration goes deep but bleaching is very slow as a function of long time exposure.

The residual TL as a function of marble thickness is shown in **Fig. 7**, where the longer exposure causes a larger bleaching for the same thickness; while noteworthy is the residual TL at 35 mm for 145 hours of daylight exposure.

Regarding slices, after a SOL2 bleaching of 1 mm thick slice of three rock types (granite, metamorphic and ultramafic), the blue and IR stimulated luminescence shows a rapid drop in dose. For ultramafic rock slide within 100 seconds but large variability in 100 seconds of SOL2 exposure, their granite and metamorphic rock slices reached a maximum bleachable value in 100 and 1000 seconds respectively (**Fig. 8**). Daylight penetration in granitic rocks, for a SOL2 exposure for 14 days, resulted in significant bleaching down to 12-15 mm. All samples were fully bleached up to 5mm depth. Daylight transmission in quartzites, also, has been observed and complete bleaching is made by IRSL (Richards, 1992).

Measurements on luminescence on granite surfaces (**Fig. 9**) have shown that the prerequisite of deep and

Table 1. *SOL bleaching, residual TL and exposure times for Thassos marble quarry.1 hr SOL ~6 hrs sunlight (based on Liritzis and Galloway, 1999).*

Depth (mm)	% Residual TL	Exposure time (hours)
top	10	1.5-40
2	30±10	40±10
4	70±	20±10
8	80±10	20±10
16	75±10	20±10

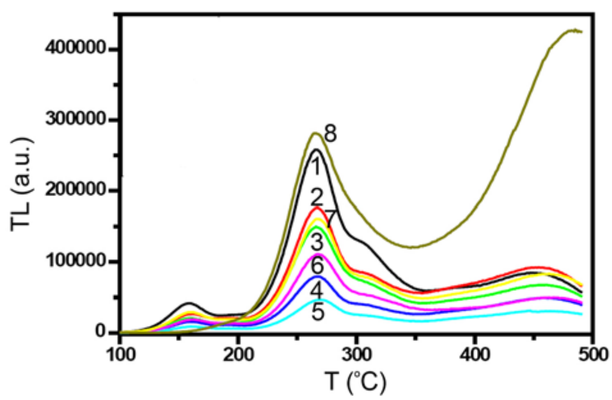

Fig. 4. *Bleached TL curves for different exposure times (1 – 1 h, 2 – 3 h, 3 – 5 h, 4 – 7 h, 5 – 10 h, 6 – 15 h, 7 – 20 h, 8 – ntl) of marble schist from dragon house, at Styra (Euboea Greece).*

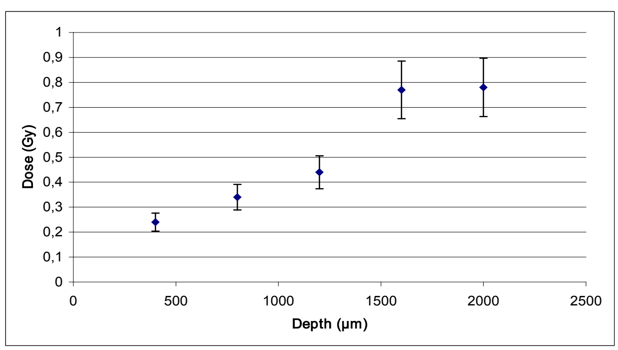

Fig. 5. *Dragon house at Kapsala, Greece. Marble schist surface (containing traces of quartz) exposed to daylight for 2 h and in the acquired 5 layers of 400 µm each, SAR protocol was applied for apparent dose determination (Liritzis, 2010).*

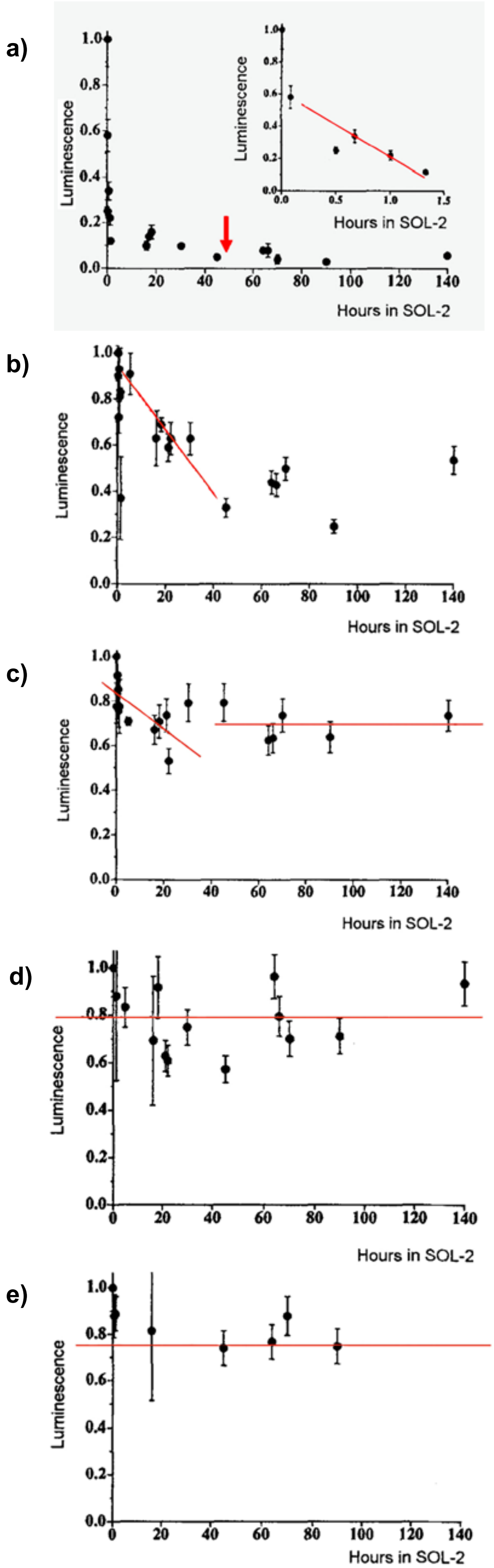

Fig. 6. *TL bleaching versus sun exposure for different marble thicknesses a) surface (0 mm), b) 2 mm, c) 4 mm, d) 8 mm and e) 16 mm (Liritzis and Galloway, 1999).*

efficient bleaching below the rock surface combined with a shallower origin of IRSL emission seems to be fulfilled (Habermann *et al*., 2000). Thus, during a 60 s exposure, the relative contribution of deeper layers did not increase significantly after the first mm. Surely, as shown above, longer exposures lead to daylight penetration to deeper layers (D>2 mm). The results reveal an almost complete bleaching to a depth, D, of at least 2 mm after at least 20 min of exposure to daylight. And for 2 min exposure the residual signal starts at 0.2 for top and reaches plateau at a 0.8 (normalized values) or 80% corresponding to D=1.5 mm depth below surface.

6. RELIABILITY CRITERIA, ERRORS AND REMEDIES

The criteria applied to examine the suitability of a rock material for dating are:

1) *Bleaching of luminescence after exposure to daylight or daylight simulator.* This needs to be tested with the preheat-dose plateau test (Liritzis *et al*., 1997b; Liritzis, 2001; 2010; Berger and Huntley, 1989) Incomplete bleaching prior to the construction can be determined also from radial plots (**Fig. 10**) (Galbraith *et al*., 1999). In the case of quartz, deconvolution of slow and fast components and calculation of D_e based on them may also identify incomplete bleaching (Liritzis *et al*., 2010b; Murari *et al*., 2007)**.**
2) *Dose recovery*, recovering of laboratory dose to test the robustness of the procedures used in the estimation of D_e applying measurement procedure for D_e,
3) Fading, pre-dose and recuperation effects (Aitken, 1998)
4) Sensitivity *correction* of dose recovery by OSL due to thermal heating and pre-heating and/or optical bleaching (Singhvi *et al*., 2010). Alternative ways for

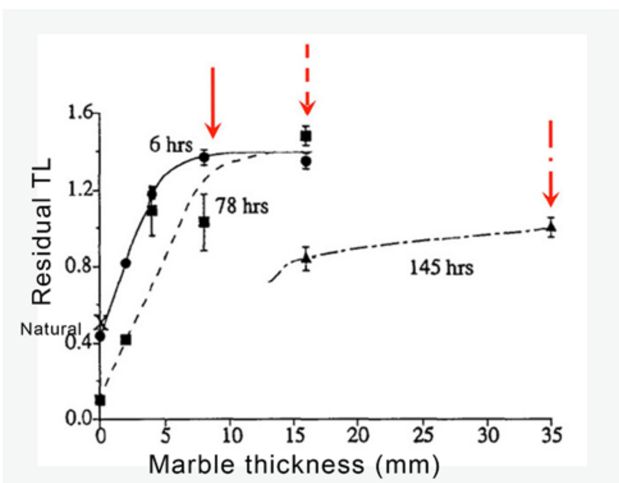

Fig. 7. *Daylight transmission in marble slabs of different thicknesses and three sun exposure times. Arrows indicate onset of saturation (Liritzis and Galloway, 1999).*

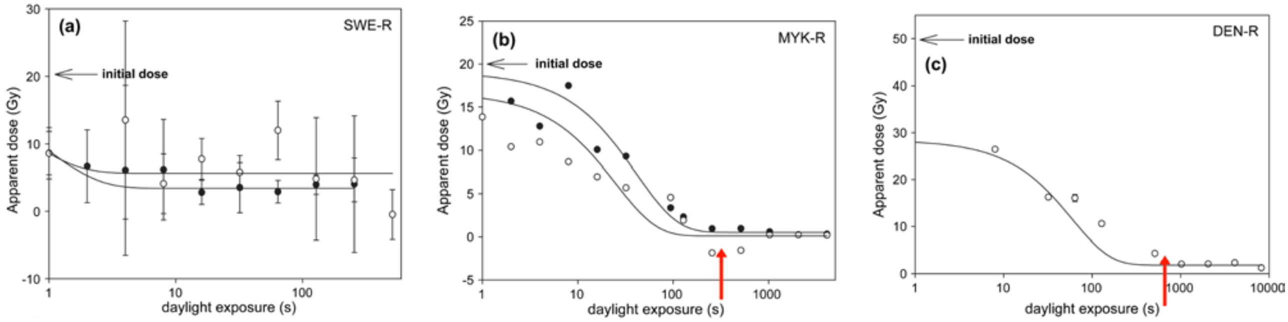

Fig. 8. *Bleaching rates of luminescence by blue (filled circles) and IR (open circles) shining of 1 mm slices of ultramafic (a), granite (b) and metamorphic (c) rocks exposed to winter daylight for different periods of time before measuring the dose using SAR protocol. Fit functions are chosen to present modulation of apparent doses (from Vafiadou* et al.*, 2007).*

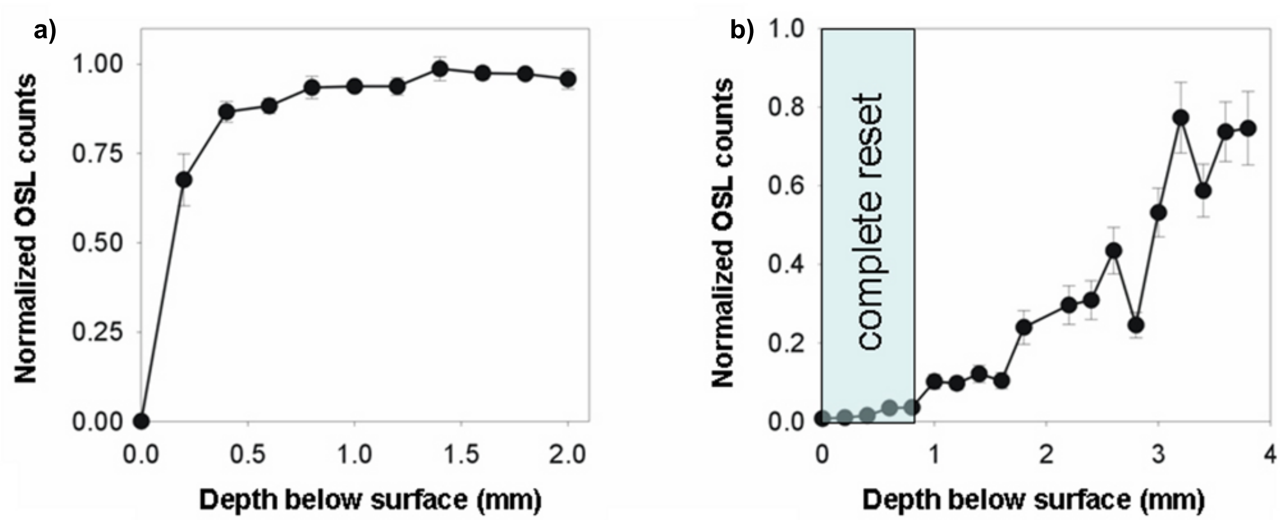

Fig. 9. *a) IRSL signal as a function of layer thickness below surface, where most signal originates in the uppermost layer of about half a mm, and, b) depth profile for a granite rock sliced in steps of 200 µm to a total thickness of 4 mm, of the remaining normalized luminescence signal stimulated by IRSL after exposure to a SOL-2 daylight simulator for 20 min. (based on Habermann* et al.*, 2000).*

D_e determination include the use of the unstable luminescence as described by Liritzis (1995). However, this has not yet been employed routinely.

These criteria are a 'must' in any dating project on a variety of rock types (Liritzis *et al.*, 2008a). The identification of mineral present can be made by XRD, while quartz and feldspar are also identified by probing with IR and blue LED.

Various sources of error and tests and remedies include:

1) Accurate water uptake estimation especially for low α, β dose-rates in samples e.g. quartzite in sediments.
2) Care should be exerted to avoid induction of luminescence by light during sampling of a building or sampling later repairs.
3) Complex radiation geometry from laboratory and natural conditions (Duller *et al.*, 1997; Greilich *et al.*, 2002; Bailiff and Mikhailik, 2003).
4) Dose-rate errors especially due to potassium distribution and U, Th inhomogeneity, pronounced in granites, need the single grain or spot OSL (Bailiff, 2006).
5) Desert varnish effects and other secondary adhesions on rock surface may be avoided by proper selection or removed by weak acid wash.
6) Scattering of additive dose points for calcites, which can be reduced by taking many measurements. However, the new technique of quartz extraction from limestone considerably improves accuracy in D_e.
7) Low radiation fields result in poor accuracies in radiation dose-rates. Gamma-ray dose-rate is most important. Use of multiple methods for dose rate estimation is recommended. With due care for the counting geometry and for possible sand / soil cover during the past.

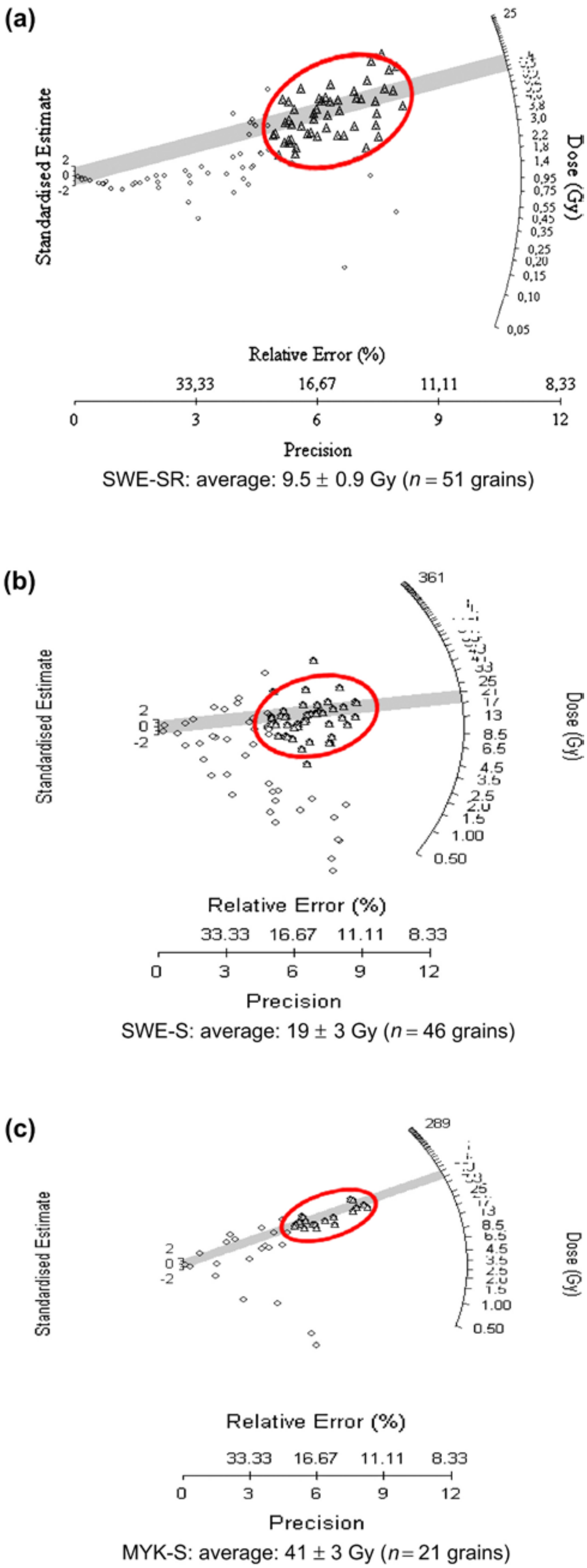

Fig. 10. *Radial plots for equivalent dose measurements from three samples from Sweden and Mykonos, Greece (triangles) and the accepted results encircled on the dark linear belt (Vafiadou* et al.*, 2007).*

8) Destruction of surface and hence the datable layer due either to friction, dilution (due to porosity and low hardness), weathering and erosion, development of salts and secondary minerals and moss/lichens. If a meticulous examination is required and, steps such as e.g. removal of inappropriate surface effects, choice of sampling points with plaster or discards is critical. A safer procedure is to divide the inner block surface into several sub-areas and in this way a geological D_e derived from unrecognizable accidentally rubbed surface parts during last placement are easily pinpointed and excluded (**Fig. 11**).

The TL/OSL dating errors on megalithics may range between ±5-7% (Martini and Sebilia, 2001) while the errors in TL dates of limestones are around ±7 to 20%. (Liritzis, 2001). Use of the quartz technique the errors should reduce the errors to ±5-7% (Aitken, 1985). However use of a limited number of aliquots would imply errors of ~ ±10%.

7. CONCLUSIONS

Archaeological and geoarchaeological materials frequently encountered in archaeological excavation sites, i.e. soil floors and pebbles and monuments made of various rocks and artifacts can be effectively dated by OSL, particularly blue stimulation and provide ages related to the last occupation of prehistoric settlements. Daylight penetration to limestone, quartz and granite, sandstone blocks bleaches the geological luminescence up to variable depths, ranging from 1 mm to several mm depending on the opaqueness and daylight exposure duration. Detailed investigations have shown that the surface luminescence dating of megalithic monuments and artifacts refers to the construction age of the masonry or last use of them followed by burial by soil. As such luminescence directly dates the construction events using its contents. Luminescence is the only direct method of dating buildings as all other dating approaches use materials found within them, which may not be related to the first construction. Architectural and contextual ages from these methods, though precise, may not be reliable due to doubtful attributions. Analysis of several aliquots of surface sub-samples may

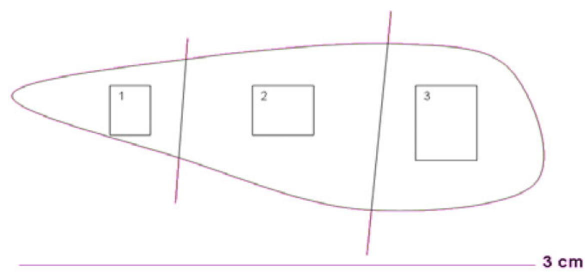

Fig. 11. *Schematic representation of a sample removed from a wall and the divided sub-areas acquiring the powder.*

help distinguish between recent bleaching, geological doses and the archaeological dose clusters in a bi- or multi-modal plot. Application to various parts of the world have so far produced satisfactory results and more will be made in the years to come.

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