Mediterranean Archaeology and Archaeometry, Vol. 9, No. 1, pp. 1-16

OPTICALLY STIMULATED LUMINESCENCE PROPERTIES OF NATURAL SCHIST

E.C. Stefanaki[1,2], D. Afouxenidis[1,2], G.S. Polymeris[1,2,*], A. Sakalis[1], N.C. Tsirliganis[1], G. Kitis[2]

[1] *Archaeometry Laboratory, Cultural and Educational Technology Institute / R.C. 'Athena', Tsimiski 58, 67100 Xanthi, Greece,*
[2] *Physics Department, Aristotle University of Thessaloniki, 54124 Thessaloniki, Greece*

Received: 12/9/2008
Accepted: 19/11/2008
Corresponding author: gpolymer@ceti.gr

ABSTRACT

Schist is a common siliciclastic geological material that has been extensively used in buildings as brick, tile and roofing slates. Its use, especially in the Mediterranean sea is widespread through the centuries. There are various examples from the ancient Greece, such as monuments from Knossos, Karthaia, as well as from modern Greece, such as traditional houses, etc. Schist is a metamorphic crystalline rock composed largely of silicon minerals, such as quartz, muscovite mica and feldspars. The type and composition of schists, as well as, the concentration of each mineral depends strongly on the type and the origin of the schist. Its past and modern use makes it a suitable candidate for archaeological dating, as well as, for retrospective dosimetry purposes.

In the present work a preliminary characterization of schist is performed in order to investigate if some basic properties required for dating applications can be found in this material. The preliminary study concerns the optical stability, the sensitization and linearity of the Infrared Stimulated Luminescence (IRSL) resulting from feldspars, as well as the post IR Blue Optically Stimulated Luminescence (post – IR Blue OSL) resulting mostly from quartz. The results indicate that both signals are rapidly bleached when the sample is exposed to sunlight. The dose response was found to be linear for radiation doses at least up to 75 Gy for the IRSL signal and at least up to 25 Gy in the case of post – IR Blue OSL. The use of a single aliquot measurement protocol, due to the lack of sensitisation, extends the latter dose response linearity region up to 75 Gy for the post – IR Blue OSL signal of schist. Finally, the application of the double single-aliquot regenerative-dose protocol to schist was investigated, in order to recover, successfully, the equivalent dose in 4 – 11 μm grains of the compound.

KEYWORDS: natural schist; OSL; dose response; sensitivity; sensitization; double SAR protocol

1. INTRODUCTION

Optically Stimulated Luminescence (OSL) has been established as a promising dosimetric technique in various fields, such as medical, environmental, personal, space and retrospective dosimetry (McKeever, 2001; Bøtter Jensen et al, 2003; Yukihara et al., 2006). The case of retrospective dosimetry, consists of two major categories, namely the archaeometry luminescence dating (Liritzis, 2000) and accidental dosimetry. Both fields involve the use of any geological material as dosimeter in order to evaluate the accumulated dose over a long time period. Any geological material is a natural dosimeter in action. However, its use as a dosimeter requires a prior systematic characterization in order to find if its properties meet the requirements set by the specific application.

Schist is a common geological siliciclastic material that has been extensively used in buildings as brick, tile and roofing slate. Its use, especially in the Mediterranean Sea is widespread through the centuries. There are various examples from the ancient Greece, such as monuments from Knossos, Karthaia, as well as from modern Greece, such as traditional houses, etc. A preliminary thermoluminescence (TL) characterization of this material, performed by Afouxenidis et al., (2007) indicated that natural schist is a promising TL candidate for archaeological dating and retrospective dosimetry measurements, due to its linear TL response for doses in the range between 1 – 100 Gy (with the 110^{0}C being an exception, yielding saturation at near 50 Gy) and the easy – to – bleach TL signal. Finally, the sensitivity of the TL signal was found to be very stable, with the exception of the 270^{0}C glow peak that was highly sensitized.

In the case of OSL dating using unfired geological materials the basic requirement is the zeroing of latent luminescence signals by bleaching due to exposure to daylight. The surface of stony objects has been exposed to daylight before they have been included in a building. After that parts of their surfaces ultimately become shielded from light allowing the dating of various archaeological and geological events, such as construction and destruction of stone buildings (Greinlich et al., 2005; Vafiadou et al., 2007). A detailed account of OSL and TL on various rock types has been reported by Liritzis et al., (2008a).

There have been several attempts to employ common building materials in dating historic buildings and monuments. Liritzis (1994) proposed the use of limestone to date megalithic structures. Similar attempts were also made to determine the ages of the Temple of Apollo at Delphi and the pyramid-like structures in Greece (Liritzis et al., 1997b; Theocaris et al., 1997). The applied methodology involved thermoluminescence measurements, but was hampered by the fact that a significant residual TL signal in limestone is left, even after a prolonged exposure to sunlight (Liritzis and Galloway, 1999). The same problem in sediment dating was overcome by using OSL techniques (Huntley et al., 1985), while in the case of limestones by the new quartz extracted technique (Liritzis et al., 2008a).

The application of schist in OSL dating according to the principle described above could be very promising. The aim of the present work is to study the basic optical properties of natural and chemically untreated schist, in order to investigate whether it fulfills the necessary requirements as an OSL dating material.

2. MATERIALS AND METHODS

2.1 Sampling

The material used in the present study is a natural schist sample obtained from a roofing slate of a house in the village Koupa, in Nothern Greece. The elemental and mineralogical composition of this specific sample was investigated by the micro-X Ray Fluorescence (μ-XRF) and X Ray Diffraction (XRD) techniques. Spectral results are given in Figs. 1 and 2. These results indicate that the schist sample was rich in silicon and consisted mainly of quartz and albite ($Na(AlSi_3O_8)$), a common type of feldspar. Mineral separation was not attempted in this preliminary study. The reason is that in most cases a very limited amount of material capable for application can be obtained. Therefore, it is very important to find that the mixed natural and chemically untreated material in the state of fine grains possesses all the necessary properties for the OSL dating procedure. So, for the OSL measurements, a piece of the original sample was initially crushed in a vice and grains of dimensions between 4 and 11 μm were selected and deposited on 1 cm^2 aluminum discs using the Zimmerman method (1971). Previously, the outer surface layers were mechanically removed in order to remove weathering products.

2.2 Apparatus and methodology

All luminescence measurements were performed using the RISØ TL/OSL reader (model TL/OSL – DA – 15), equipped with a high-power blue LED light source, an infrared solid state laser and a 0.085 Gy/s $^{90}Sr/^{90}Y$ β-ray source. The reader is fitted with an EMI 9635QA PM Tube (Bøtter- Jensen et al., 2000). All luminescence signals were detected through a 7.5 mm Hoya 340 filter. The latter was chosen, because albite presents its main emission band from ~ 150 to 350 nm, centred at 290 nm (Clarke and Rendell, 1997). This spectral band almost coincides with the transmission characteristics of the Hoya 340 detection filter (Bøtter- Jensen, 1997).

The power level was computer controlled and set at 90% and 70% of the maximum power for the infrared and blue stimulated luminescence respectively, delivering to the sample a total of 36 and ~95 mW cm^{-2} respectively. All measurements were performed using continuous wave OSL (CW-OSL) stimulation mode. In order to monitor independently the OSL contribution from quartz and feldspars, the measurement procedure involved exposure to infrared (IR) light, prior to the measurement of the blue light-stimulated OSL signal (termed post – IR Blue OSL) (Liritzis et al., 1997a; Banerjee et al., 2001; Liritzis et al., 2001; Roberts and Wintle, 2003). We have to stress here that the post – IR Blue OSL signal is likely to be dominated by quartz, but should also contain an unknown contribution from feldspars, since it has been demonstrated that a significant OSL signal can be obtained from feldspar grains even after the infrared stimulated luminescence (IRSL) signal has been reduced to a negligible level (Duller and Bøtter-Jensen, 1993; Roberts and Wintle, 2003).

Stimulation times were chosen to be 150 s for IRSL and 60 s for post – IR Blue OSL. Initial tests verified that these stimulation times were adequate for both luminescence signals to reach the dark current count rate. Both signals were measured; at room temperature without preheat, as well as at 125^{0}C after the sample had been preheated at a temperature of 180^{0}C for 10 seconds. The choice of the specific preheat temperature of 180^{0}C, was based on separate experiments described in the next section 2.3.

2.3 *Preheat test*

The preheat test is performed in order to evaluate the optimal preheat temperature, for both Infrared and Post- IR Blue stimulation measurements, needed to remove the "110^{0}C" glow-peak which is present during the artificial beta irradiation. While investigating the preheat temperature to be used in a retrospective dosimetry study it is necessary to compare the response of both natural IRSL and post-IR blue OSL signals as well as the corresponding regenerated signals, in the latter situation using aliquots in which the trapped charge has been removed by the laboratory bleach and then given a laboratory dose. At the present test, the effect of 10 sec preheats was studied at temperatures ranging between 120^{0}C and 280^{0}C (with a step of 20^{0}C) on the shape of the decay curves obtained subsequently. In the case of regenerated signals, a dose of 4.5 Gy was delivered. It is the ratio of these two signals, namely natural over regenerated luminescence, that is plotted in Fig. 3 as a function of preheat temperature in the case of IRSL (lower figure, B) as well as of Post - IR Blue OSL (upper figure, A).

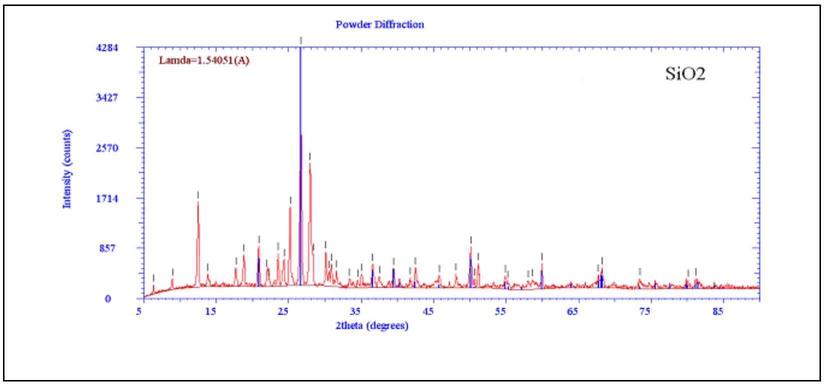

Figure 1. X – Ray diffraction spectrum of schist. One of the main components is quartz (*SiO_2*).

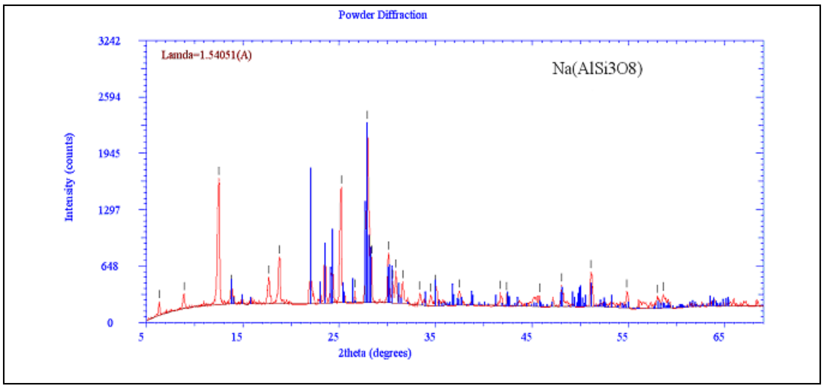

Figure 2. X – Ray diffraction spectrum of schist. One of the main components is also albite (*$Na(AlSi_3O_8)$*), a common type of feldspar.

As can be seen from Fig. 3A there is no systematic effect on the natural over regenerated luminescence ratio for the case of Post - IR Blue OSL curve shapes among the preheat temperatures of 140-220^{0}C. These results indicate a component which is thermally very stable in that specific temperature region, hence extremely useful for dating and retrospective dosimetry. The respective temperature region for the IRSL is between 140 - 180^{0}C, as can be seen from the plateau. Consequently, the preheat temperature should lie among the common plateau area of both IRSL and Post- IR Blue OSL, namely it will be the one of 180^{0}C.

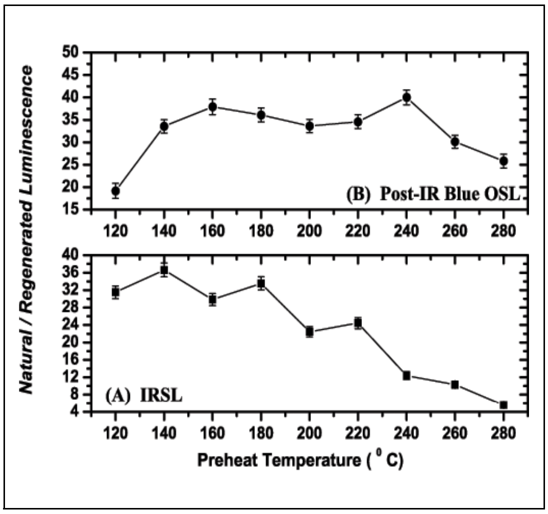

Figure 3. Preheat test for schist for post- IR Blue OSL (upper figure) and IRSL (lower figure). All measured OSL were stimulated at 125^{0}C following a 10 sec preheat at temperatures from 100^{0}C to 280^{0}C. A plateau is observed among the preheat temperatures of 140- 260^{0}C, 180- 240^{0}C in the case of Post- IR Blue OSL, while the respective area for the IRSL is among 100- 180^{0}C.

2.4 Decay curves - Method of analysis

Decay curves for the natural OSL signal of the schist's sample are presented in Fig. 4. The left hand side figure presents the continuous wave IRSL decay curve, whereas the right hand side figure gives the respective post-IR Blue OSL decay curve, both measured at room temperature.

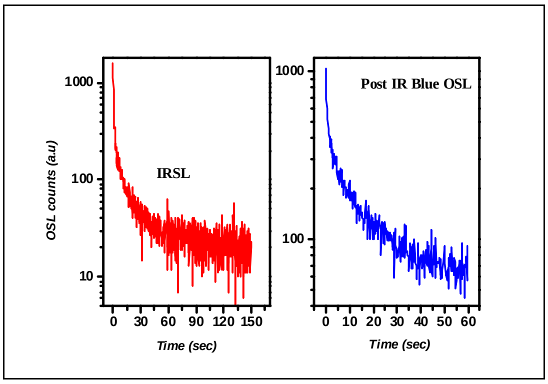

Figure 4. Decay curves of natural OSL signal for the schist sample. The figure on the left shows the continuous wave IRSL decay curve, while the figure on the right shows the respective post-IR Blue OSL decay curve. Both curves were obtained at room temperature. Insets show the respective curves in logarithmic y-scale.

The presence of intense luminescence signals for both stimulation wavelengths is explained by the mineralogical composition of the sample as was indicated by the XRD results, since the schist sample is rich in silicon and consisted mainly of quartz and albite ($Na(AlSi_3O_8)$), a common type of feldspar. The IR stimulated luminescence signal, resulting mostly from feldspars, is more intense than that resulting from post-IR Blue stimulation. However, both signals are very easy to bleach, since they decay very fast in the first seconds of stimulation. Therefore, there is no need for a full component resolved study for the OSL signals, since in both cases the latter consists mostly of one component. Instead, luminescence signals are analyzed using the raw signal integrated over the first second of stimulation. Fig. 4 confirms also that the illumination times used in both cases are adequate to empty traps responsible for both luminescence signals. Therefore, the OSL signal measured at the last 5 seconds of stimulation was subtracted as background from the initial luminescence intensity of the obtained decay curves.

3. RESULTS & DISCUSSION

3.1 Bleaching properties

Adequate optical resetting of previously accumulated luminescence signal is a fundamental requirement for accurate dating. The likelihood of full resetting depends on many factors, but one of the most important is the ability of the mineral used, to bleach rapidly. In order to study the bleaching ability of schist, 22 different aliquots were divided into ten separate groups of two aliquots each. The samples from the first group were measured in order to get the natural IRSL and post – IR Blue OSL signal. The remaining groups were exposed to simulated sunlight in a SOL–2 solar simulator (Dr Hönle) over variable lengths of time. Ten different time exposures were applied, ranging from 5 to 500 seconds. After each exposure, the residual signal of both IR and post – IR Blue stimulated luminescence was measured for both aliquots of each group.

Results are shown in Fig. 5, where the residual IRSL and post – IR Blue OSL signals are plotted as a function of the exposure time. Each experimental point corresponds to the mean value of the measurements carried out for each aliquot. Error bars indicate 1σ deviations. As can be seen from Fig. 5 both luminescence signals are reduced substantially, even after 40 seconds of exposure. The decay curves indicate also that IRSL signal is easier to bleach. Furthermore, after 150 seconds of bleaching, both luminescence signals have reached their minimum values. According to Fig. 5, the OSL signal is reduced to 1% and 6% respectively for Infrared and post – IR Blue stimulation.

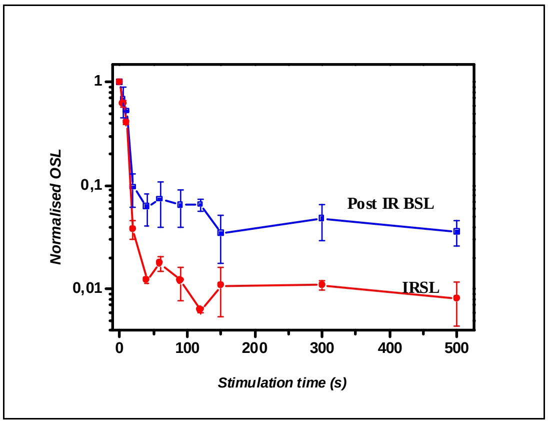

Figure 5. Residual IRSL (circles) and post-IR Blue OSL (squares) signals, as a function of exposure time to simulated sunlight produced by a SOL – 2 solar simulator. The first measurement point represents the natural OSL signal of the sample. Each experimental point corresponds to the mean value of the two measurements carried out in two different aliquots. Error bars indicate deviation of 1σ.

The ability of both luminescence signals of schist to bleach very fast ensures the zeroing of both OSL and IRSL signals at its outer surface when it is exposed to direct sunlight for even very short time. For OSL dating the zero time is set when the material is positioned in a light shielded position of the building.

Since the light penetration is limited to the outer surface layers the luminescence intensities and therefore equivalent dose values will increase as a function of the depth from the surface, reaching a stable value at the depths/layers not influenced, at all, by the sunlight. This stable value will correspond to the natural dose. However, another important aspect of bleaching in nature such as the degree to which daylight penetrates the rock surface (Liritzis et al., 1997b; Liritzis and Galloway, 1999; Vafiadou et al., 2007) was not studied at all. Further work is required in order to define the bleached depth.

3.2 Sensitization

Sensitization of the TL signal is an effect commonly found in quartz. Occasionally, however, it is also reported for feldspars (Bailiff and Barnett, 1994). The sensitization of the TL signal of quartz depends on the type of the quartz and mainly on its thermal and radiation history. The sensitization effect appears also in the case for the OSL signal. Furthermore, the stimulation light may cause sensitivity changes as well. So, monitoring the sensitivity changes as a function of successive OSL measurement cycles (Liritzis et al., 2008b), becomes a crucial test especially when single aliquot protocols are to be applied. The OSL sensitization effect in schist was studied by performing measurements at (i) room temperature and (ii) at 125 ^{0}C after a preheat at 180 ^{0}C has been applied.

3.2.1 Sensitivity test at room temperature

Using the same aliquot 20 successive irradiation–OSL measurement cycles were performed. The dose used for each irradiation was 4.25 Gy. The lower Fig. 6 shows the normalised OSL sensitivity in the case of IRSL, whereas the upper Fig. 6 shows the corresponding post – IR Blue. Although no heating was applied, both luminescence signals were sensitized. The IRSL signal, resulting from feldspar shows an increase of 7.2% per cycle, whereas for the post – IR Blue stimulated signal the increase is 2.25% per cycle. In both cases the sensitization yielded is linearly increased versus repetition cycle. The same experiment was carried out in two other aliquots, using different test doses for each, namely 8.5 and 17 Gy respectively. The results were similar, indicating a strong sensitization of the order of 7-9% per cycle for the IRSL signal and smooth sensitization of the order of 1.5-2.5% per cycle for the post – IR Blue OSL signal. The sensitization of feldspars, prevent the use of single aliquot measurements, unless a sensitivity correction is achieved, while the sensitization of quartz will have a lesser impact in similar measurements.'

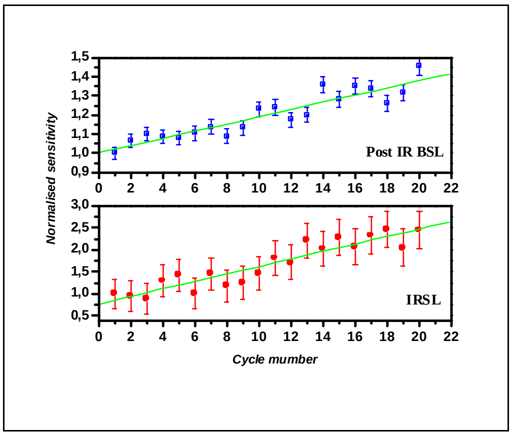

Figure 6. Normalised OSL sensitivity, for succesive irradiation – OSL measurement cycles performed at room temperature. The irradiation dose used for each cycle was 4.25 Gy. The upper figure shows the normalised OSL sensitivity in the case of post-IR Blue OSL, while the lower figure shows the respective IRSL sensitivity. Error bars indicate deviation of 1σ. The solid lines represent the linear regression fitted line.

A basic requirement for any single aliquot protocol, in order to enable reliable palaeodose measurements is that any change in the luminescence sensitivity of samples is monitored, as well as properly corrected for, in the course of the protocol. The most established single aliquot protocol is the single-aliquot regenerative dose (SAR) protocol (Murray and Wintle, 2000), which compensates for any sensitivity changes, occurring as a result of regeneration of the OSL signal, by using a test dose to measure the OSL sensitivity appropriate to the regenerated signals; this is then used for sensitivity changes (Murray and Mejdahl, 1999).

In order to check, whether this particular sensitivity correcting methodology is applicable to schist, the preceding 20 successive measurements were divided into two groups. The first group includes the signal that was obtained from the even measurements (2, 4, 6, etc) and was considered as the test dose signal (T_i), whereas the second group includes the signal from the odd measurements (1, 3, 5, etc) corresponded to the main dose measurements (L_i). Since sensitivity is linearly enhanced with successive cycles, for both luminescence signals (according to Fig. 6), it is expected that the ratio $R_i=L_i/T_i$ provides the corrected signal as far as its sensitivity is concerned. In the present experiment the test dose is equal to the main dose and as a result, the ratio R_i of the corrected signal is expected to be approximately equal to 1. Results indicated ratio values close to unity for both signals, namely $R_{4.25}$ = 1.026 ± 0.052 in the case of IRSL and 0.975 ± 0.065 for the post – IR Blue OSL. This leads to the conclusion that a test dose can be used to correct the sensitization occurring in schist's signal, and the single aliquot protocol can be used accordingly for OSL measurements at room temperature.

However, before the latter conclusion can be undoubtedly claimed, a lack of dose dependence must be shown (Wintle and Murray, 2006). This is experimentally verified by performing measurements on 2 different aliquots as following; each aliquot was subjected to 10 successive irradiation – RT OSL measurement (L_i) cycles, using the main doses of 8.5 and 17 Gy respectively. In both cases, after each cycle, a test dose irradiation – RT OSL measurement (T_i) cycle was subjected, using the fixed test dose of 4.25 Gy. Twice again, for both luminescence signals, the mean ratio $R_i=L_i/T_i$ provides the corrected signal as was expected, namely $R_{8.5}$ = 2.074 ± 0.072 and R_{17} = 4.34 ± 0.25 in the case of IRSL, while for the post – IR Blue OSL signal $R_{8.5}$ = 1.975 ± 0.095 and R_{17} = 3.8 ± 0.175.

3.2.2 Sensitivity test at 125 ^oC

In order to monitor the sensitivity changes of schist's OSL signal, when the latter is measured at the temperature of 125 ^0C, 3 different aliquots were used. Each one was subjected to 9 successive irradiation – preheating – OSL measurement cycles. Three different doses were used for the irradiations of each aliquot, namely 4.25, 8.5 and 17 Gy. Each aliquot was preheated for 10 s at 180^0 C prior to each OSL measurement. Fig. 7 presents the normalised OSL sensitivity in the case of IRSL (lower figure) as well as of Post- IR Blue OSL signal, (upper figure), in the case of an administered dose of 17 Gy. As can be seen from Fig. 7, the normalised sensitivity of both luminescence signals does not seem to vary significantly between the successive irradiation – preheating – OSL measurement cycles. This is also the case for the other two test doses, all yielding similar results.

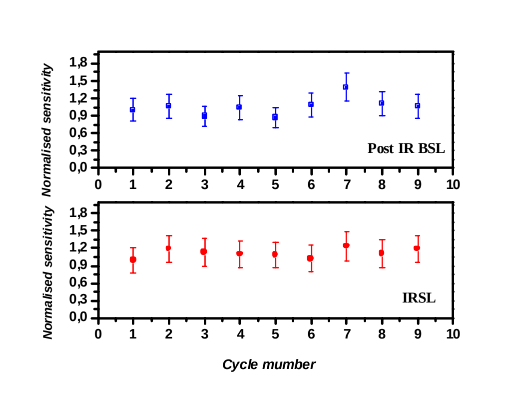

Figure 7. Normalised OSL sensitivity for IRSL signal (lower figure) as well as the post-IR Blue OSL signal (upper figure) and the IRSL signal (lower figure) measured at 125 °C after a 10 s preheat at 180 °C, for succesive irradiation – OSL measurement cycles. The irradiation dose used for each cycle was 17 Gy. Error bars indicate deviation of 1σ.

3.3 Dose response

The dose response is defined as the functional dependence of the intensity of the measured luminescence signal upon the absorbed dose (McKeever et al, 1995). The ideal dosimetric material should have a linear dose response over a wide range of doses. However, dosimeters used in practical dosimetry, such as quartz, exhibit a variety of non linear effects.

For the determination of the dose response curves of schist, only bleached aliquots were used. The OSL dose response curves were obtained using both the multiple aliquot and the single aliquot approach. In the first case one sample is irradiated successively with increasing doses, whereas for the multiple aliquot procedures a different aliquot is used for each cycle of irradiation- measurement. In both cases the irradiation doses that were used covered the range of 0.25 to 75 Gy.

3.3.1. Dose response in multiple aliquots

The multiple aliquot dose response of schist was carried out at room temperature, as well as, at 125°C using a pre-heat at 180°C for 10 seconds. The results are shown in Fig. 8. Solid lines represent the egression lines obtained according to equation ln(OSL) $=ln(A)+B\cdot ln(D)$, with B giving the degree of sublinearity for each case.

Fig. 8A shows the dose response curves of the post – IR Blue OSL measured at room temperature, where one can see that the linearity is restricted to a dose range between 2.5 and 25 Gy. On the other hand when the measurements were performed at 125°C, Fig. 8B, some degree of sublinearity is observed.

Figures 8(C, D) show the dose response curves of IRSL signals obtained at room temperature and at 125 °C respectively. In both cases a very good linear dose response is observed over the entire dose region studied.

3.3.2. Dose response in single aliquot

As it was shown in sections 3.2.1 and 3.2.2, both IR and post – IR Blue OSL signals, are stable under successive irradiation–preheat–measurement cycles of the same aliquot. Based on this stability the OSL dose response study was applied to a single aliquot for the case of OSL measurements at 125°C. The results are shown in Fig. 9, where can be seen that the OSL dose response curves resulting for both stimulation sources are linear over the entire dose region under investigation.

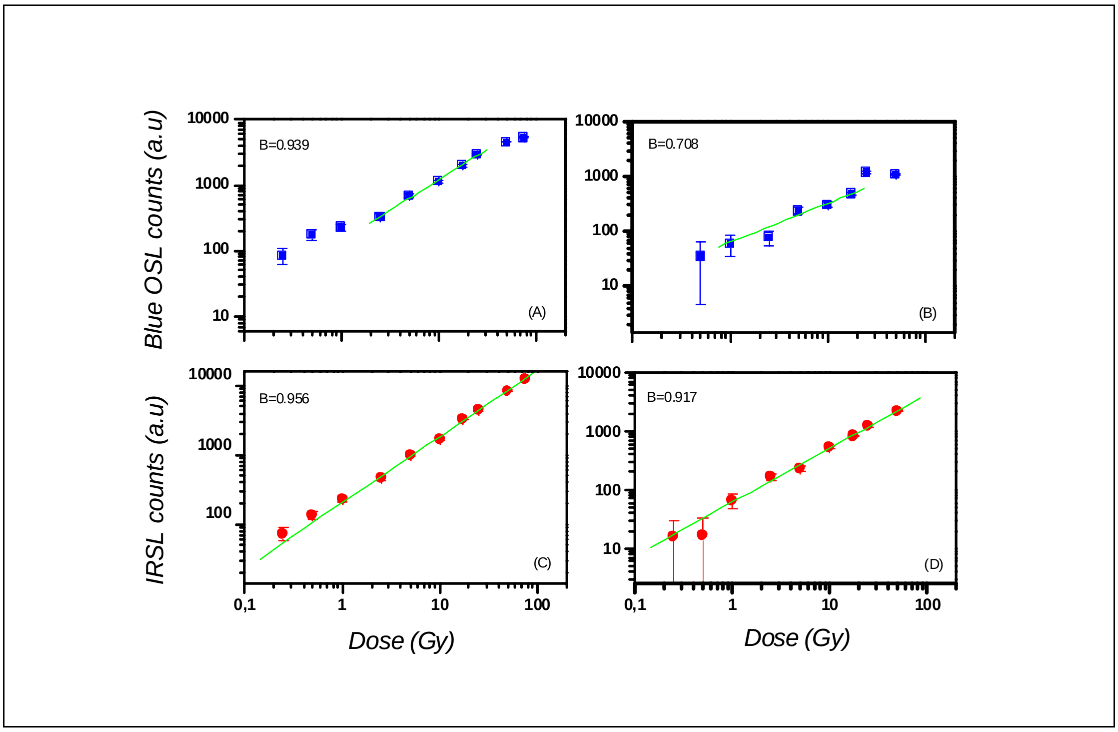

Figure 8. Dose response curves for the post-IR Blue OSL signal measured at room temperature (A), the post- IR BSL signal measured at 125 °C after a 10 s preheat at 180 °C (B), the IRSL signal measured at room temperature (C), and the IRSL signal measured at 125 °C after a 10 s preheat at 180 °C (D) for a dose range of 0.25–75 Gy. Solid lines represent the regression line derived from the equation $Y=A{\cdot}D^B$. B values (linearity coefficients) have been calculated for the linear region of each dose response curve.

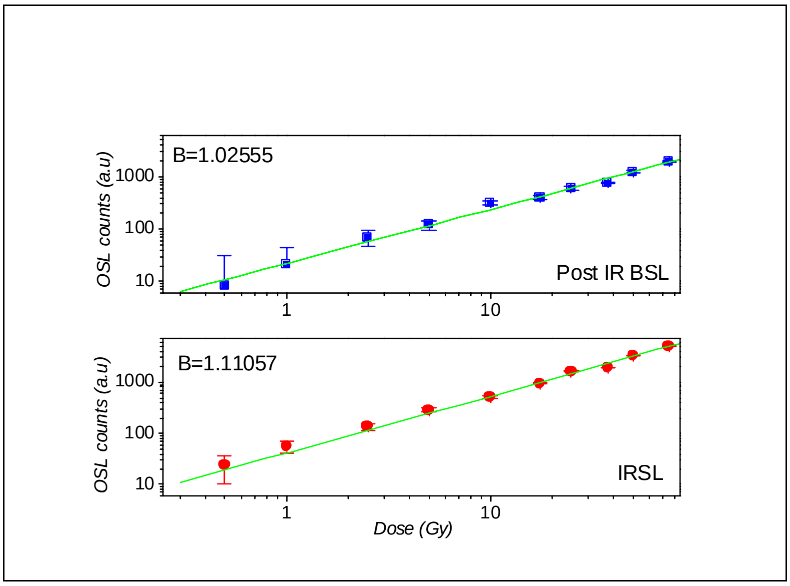

Figure 9. Single aliquot dose response curves for schist for post-IR Blue OSL (upper figure) and IRSL (lower figure). All measurements were performed at 125 °C, after a 10 s preheat at 180 °C. Solid lines represent the regression line derived from the equation $Y=A{\cdot}D^B$. B values have been calculated for the linear region of each dose response curve and are presented for each figure independently.

3.4. Dose recovery with double SAR protocol

A fundamental test of any dating protocol is whether the value of a previously delivered known laboratory dose can be accurately and precisely determined (Roberts & Wintle, 2003). In order to perform a dose recovery test according to Murray and Wintle (2003), 25 aliquots were divided into five separate groups of five aliquots each. The natural signal of the samples was initially bleached by a 4 hour exposure to simulated daylight in a SOL2 solar simulator. The samples were then stored at room temperature for a time period of 10 Ks. An initial OSL measurement for 300 s was used to verify the effective removal of the natural signal and bleach any additional dose accumulated during the storage time. Then, samples of each group were given the same laboratory β dose. 5 different doses were delivered, one for each group, selected to be in the linear region of the dose response, i.e. in the range between 10 and 75 Gy. The double SAR protocol was then applied. The dose delivered was treated as an unknown, and was estimated by the SAR analysis protocol for both luminescence signals (IRSL and post – IR Blue OSL), with a preheat at 180°C for 10 sec and a test dose of 15 Gy (apart from the measurement where the administered dose was 10 Gy, where the test dose was 5.5 Gy)

Figs 10 and 11 present the data obtained from dose recovery experiments for the post – IR Blue OSL and the IRSL signals respectively, after the application of the double SAR protocol. Measured (recovered) dose (figures A), recycle ratio (figures B) and recuperation values (figures C) are plotted as a function of the delivered dose. Recuperation is expressed as a percentage of the initial signal measured. Open circles represent the values of each measurement, while filled squares stand for the mean values of each group. Error bars indicate 1σ deviation. Solid lines in the case of figures A indicate the ideal situation, where the delivered and recovered doses are equal (Y=X line). In the ideal case, all measured doses, namely all experimental points should lie across this line, for both luminescence signals. Solid lines in both B figures indicate the (ideal) value 1 for the recycle ratio.

As can be seen from Fig. 10A the (double) SAR protocol can be used succesfully in order to recover the dose delivered to schist using the post-IR Blue OSL signal. All experimental values for the measured doses lie very close to the actually deliverd doses. The suitability of the protocol is also strongly supported by the values of the recycle ratios, which lie mainly in the range of 0.85 – 1.15, their mean values, which are very close to unity, as well as the recuperation values, which are less than 10%.

However, this is the case only for the post-IR Blue OSL signal, for the entire dose recovery region studied. IRSL measurements overestimated slightly (1.3%) the lowest delivered dose (10 Gy), while the IRSL SAR protocol provides an increasingly underestimation of the delivered dose as the latter increases, reaching an underestimation value of 34% for the highest delivered dose of 75 Gy. Fig. 11A indicates that the application of the (double) SAR protocol in order to recover the dose delivered to schist using the IRSL signal does not provide reliable estimates of the delivered dose. This is also supported by the extremely high values of the recycle ratios, despite the fact that values of recuperation would yield the exact opposite.

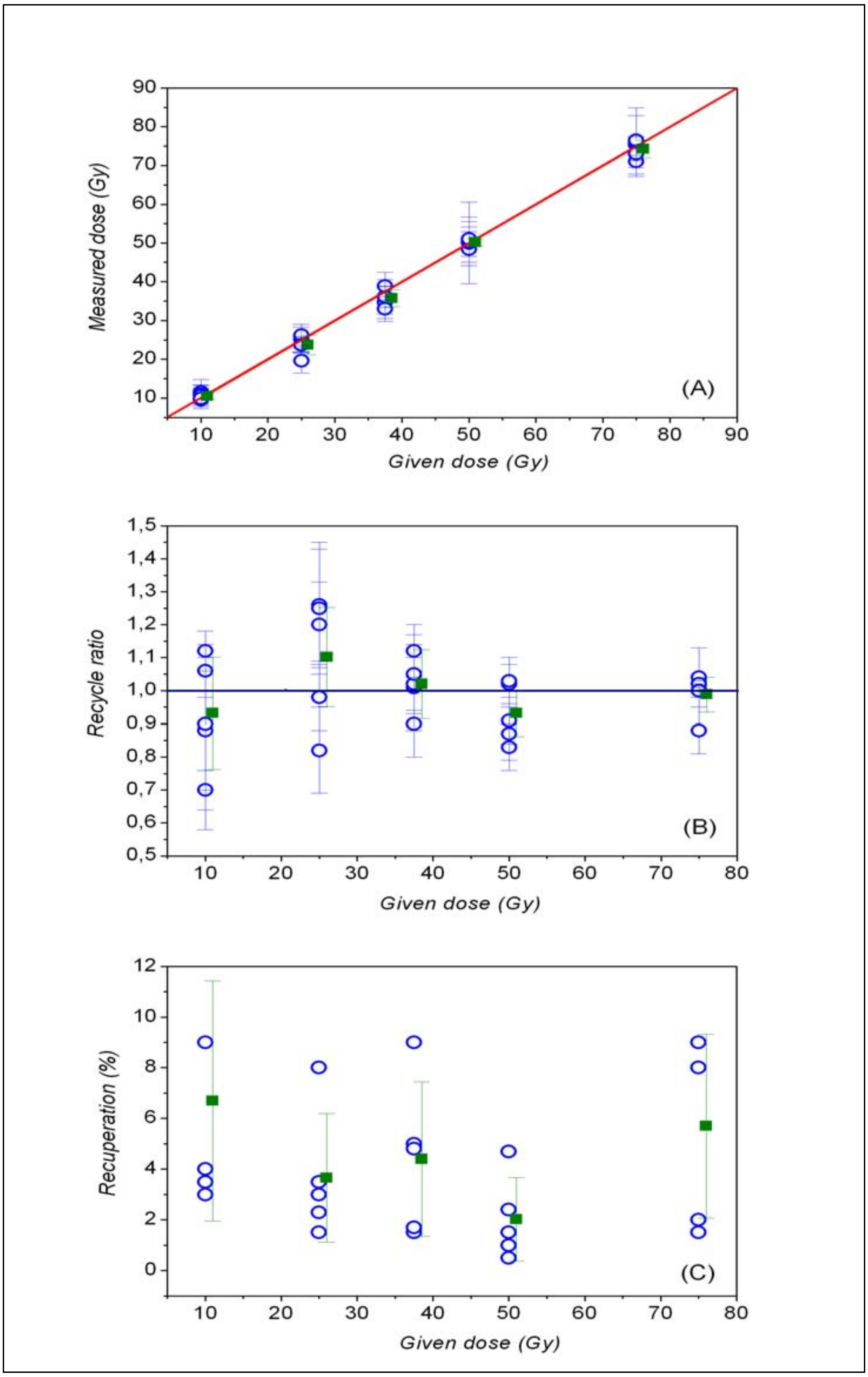

Figure 10. Measured (recovered) dose (A), recycle ratio (B) and recuperation values (C) for post-IR Blue OSL signal as a function of the administered dose. Recuperation is expressed as a percentage of the initial signal measured. Open circles represent individually measured values of each quantity, while filled squares the respective mean value. Error bars indicate 1σ deviation. Solid lines in figures A and B indicate the ideal situation.

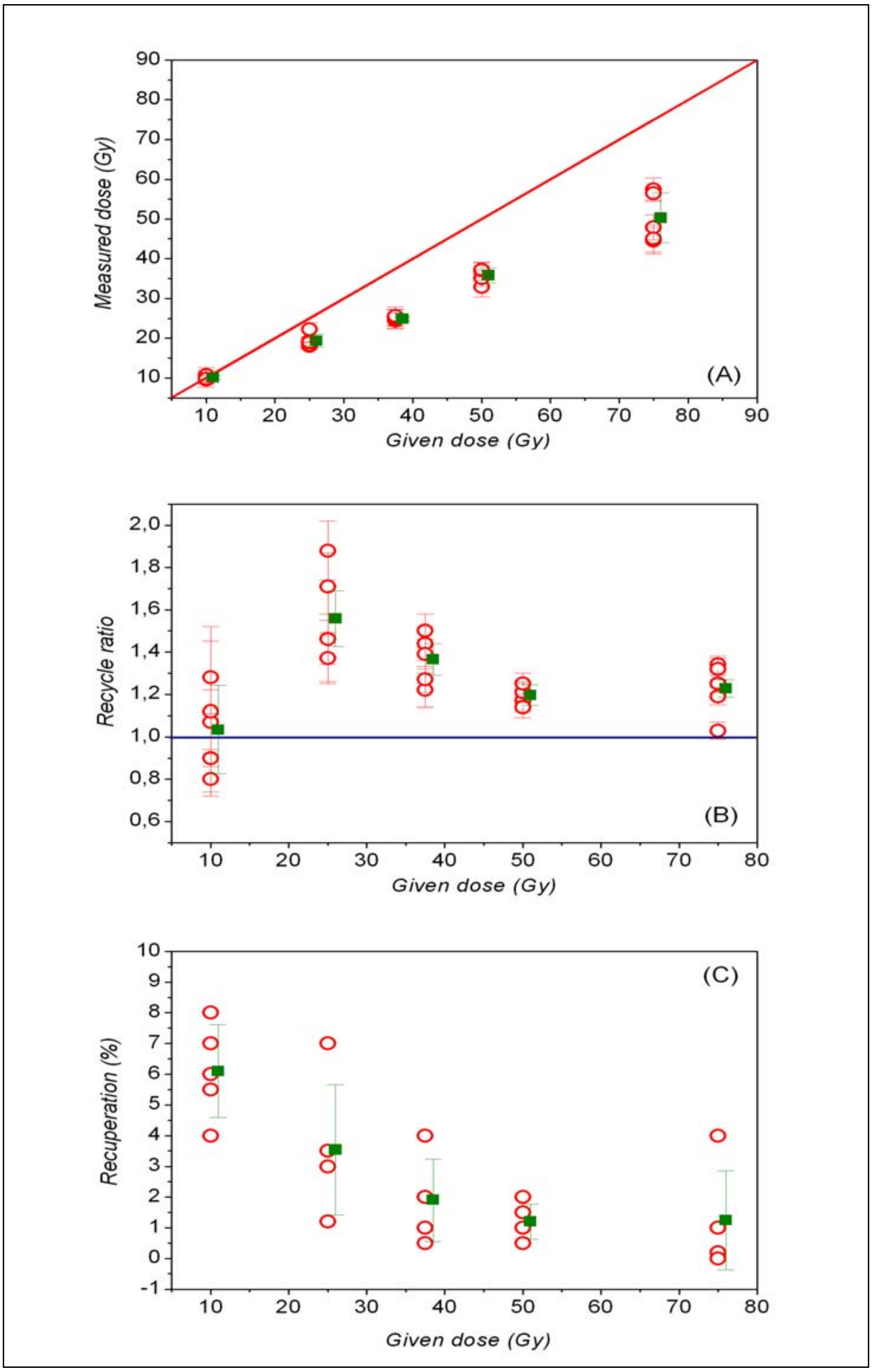

Figure 11. Measured (recovered) dose (A), recycle ratio (B) and recuperation values (C) for IRSL as a function of the administered dose. Recuperation is expressed as a percentage of the initial signal measured. Open circles represent individually measured values of each quantity, while filled squares the respective mean value. Error bars indicate 1σ deviation. Solid lines in figures A and B indicate the ideal situation.

4. CONCLUSIONS

The wide spread use of Schist in many historical monuments in ancient Greece and many other parts of the world, in combination with its chemical composition, constitute two powerful motivations for further investigation of this material for luminescent dosimetry purposes. Schist appears to have a number of interesting OSL properties, such as the ability of both IRSL and post-IR blue OSL signals to bleach rapidly when the sample is exposed to sunlight. Furthermore, both signals are fairly stable. However, certain properties such as dose response and the ability to recover successfully a previously delivered dose depend strongly on the type of the luminescence signal studied, as well as the stimulation/measurement mode. In particular, the dose response was found to be linear for radiation doses at least up to 75 Gy for the IRSL signal and at least up to 25 Gy in the case of post-IR Blue OSL. The use of a single aliquot measurement protocol, due to the lack of sensitisation, extends the dose response linearity region up to 75 Gy for the latter. Dose recovery data suggest that the post-IR Blue OSL signal provides the more reliable value of De, indicating the suitability of the specified post- IR Blue OSL (double) SAR procedure to recover successfully the dose delivered to schist. These properties of the schist suggest that it can be a promising candidate for archaeological dating and retrospective dosimetry applications, at least in the dose ranges between 0.5 and 25 Gy while using post-IR Blue OSL and up to 75 Gy in the case of IRSL. Further work is required in order to study the solar penetration in several schist samples.

ACKNOWLEDGEMENTS

The present work was partially funded by the Greek General Secretariat of Research and Technology and the E.C., under the programme "Excellence in Research Institutes GSRT (2nd round)", sub programme "Support for Research Activities in C.E.T.I.".

REFERENCES

Afouxenidis, D., Stefanaki, E.C., Polymeris, G.S., Sakalis, A., Tsirliganis, N.C., Kitis, G., 2007. TL/OSL properties of natural schist for archaelogical dating and retrospective dosimetry. *Nuclear Instruments and Methods In Physics Research A* 580, 705 – 709.

Bailiff, I.K., Barnett, S.M., 1994. Characteristics of infrared stimulated luminescence from feldspars at low temperatures. *Radiation Measurements* 23, 541–545.

Banerjee, D., Murray, A.S., Bøtter-Jensen, L., Lang, A., 2001. Equivalent dose estimation using a single aliquot of polymineral fine grains. Radiation Measurements 33, 73–93.

Bøtter – Jensen, L., 1997. Luminescence techniques: Instrumentation and Methods. *Radiation Measurements* 27, 749- 768.

Bøtter – Jensen, L., Bulur, E., Duller, G.A.T., Murray, A.S., 2000. Advances in luminescence instrument systems. Radiation Measurements 32, 523 – 528.

Bøtter – Jensen, L., McKeever, S. W. S., Wintle, A. G., 2003. Optically Stimulated Luminescence Dosimetry. Elsevier, Amsterdam.

Clarke, M. L., Rendell H. M., 1997. Infra- red stimulated luminescence spectra of alkali feldspars. *Radiation Measurements* 27 (2), 221- 236.

Duller, G.A.T., Bøtter-Jensen, L., 1993. Luminescence from potassium feldspars stimulated by infrared and green light. *Radiation Protection Dosimetry* 47, 683–688.

Huntley, D. J., Godfrey – Smith, D. I., Thewalt, M. I., 1985. Optical dating of sediments. *Nature*, 313, 105 – 107.

Greilich, S., Glasmacher, U.A. and Wagner, G.A., 2005. Optical dating of granitic stone surfaces. *Archaeometry*, 47, 645-665.

Liritzis, I., 1994. A new dating method by thermoluminescence of carved megalithic stone building. *Comptes Rendus de l' Academie des Sciences* – Serie II, 319, 603 – 610.

Liritzis, I., 2000. Advances in thermo- and opto- luminescence dating of environmental materials (sedimentary deposits) Part I : techniques. *Global Nest* 3 – 27 (Part II: Applications. Global Nest 29 – 49).

Liritzis, I., Galloway, R. B., Hong, D.J., 1997a. Single aliquot dating of ceramics by green light stimulation of luminescence from quartz. Nuclear Instruments and Methods in Physics Research B 132, 457 – 467.

Liritzis, I., Guibert, P., Foti, F., Schvoerer, M., 1997b. The Temple of Apollo (Delphi) strengthens novel thermoluminescence dating method. Geoarchaeology, 12 (5), 479 – 496.

Liritzis, I., Galloway, R. B., 1999. Dating implications from solar bleaching of thermoluminescence of ancient marble. Journal of Radioanalytic and Nuclear Chemistry, 241 (2), 361 – 368.

Liritzis, I., Katsanopoulou, D., Soter, S., Galloway, R.B., 2001. In search of ancient Helike, Gulf of Corinth, Greece. Journal of Coastal Research 17, 118 – 123.

Liritzis, I., Sideris, C., Vafiadou, A., Mitsis, J., 2008a. Mineralogical, petrological and radioactivity aspects of some building material from Egyptian Old Kingdom monuments. Journal of Cultural Heritage 9, 1 – 13.

Liritzis, I., Kitis, G., Galloway, R.B., Vafiadou, A., Tsirliganis, N.C., Polymeris, G.S., 2008b. Probing luminescence dating of archaeologically significant carved rock types. *Mediterranean Archaeology and Archaeometry* 8 (1) 61 – 79.

McKeever, S. W. S., Moscovitch, M., & Townsend, P. D. (1995). Thermoluminescence dosimetry materials: Properties and uses. Ashford, UK: Nuclear Technology Publishing.

McKeever, S. W. S., 2001. Optically stimulated luminescence dosimetry. Nuclear Instruments and Methods in Physics Research B 184, 29 – 54.

Murray, A.S, Mejdahl, V, 1999.Comparison of regenerative-dose single –aliquot and multiple –aliquot (SARA) protocols using heated quartz from archaeological sites. *Quaternary Science Reviews* 18 (2), 223-229.

Murray, A.S., Wintle, A.G., 2000. Luminescence dating of quartz using an improved single-aliquot regenerative-dose protocol. *Radiation Measurements* 32, 57–73

Roberts, H.M., Wintle, A.G., 2003. Luminescence sensitivity changes of polymineral fine grains during IRSL and [post-IR] OSL measurements. Radiation Measurements 37, 661–671.

Theocaris, P.S., Liritzis, I., Galloway, R.B., 1997. Dating of two Hellenic pyramids by a novel application of thermoluminescence. *Journal of Archaeological Science* 24, 399 – 405.

Yukihara, E. G., Sawakuchi, G. O., Guduru, S., McKeever, S. W. S., Gaza, R., Benton, E. R., Yasuda, N., Uchihori, Y., Kitamura, H., 2006. Application of the optically stimulated luminescence (OSL) technique in space dosimetry. Radiation Measurements 41 (9-10), 1126 – 1135.

Vafiadou, A., Murray, A.S., Liritzis, I., 2007. Optically stimulated luminescence (OSL) dating investigations of rock and underlying soil from three case studies. Journal of Archaeological Science 34, 1659 – 1669.

Wintle, A. G., Murray, A. S., 2006. A review of quartz optically stimulated luminescence characteristics and their relevance in single-aliquot regeneration dating protocols. Radiation Measurements 41 (4), 369 – 391.

Zimmerman, D.W., 1971. Thermoluminescence dating using fine grains from pottery. Archaeometry 13, 29–52.