

LUMINESCENCE DATING OF ARCHAEOLOGICAL MATERIALS AND SEDIMENTS IN ROMANIA USING QUARTZ*

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Abstract. Luminescence dating exploits the dosimetric properties of mineral grains found in archaeological and geological materials of interest. The use of luminescence dating in archaeometry stretches back more than half a century, starting with thermoluminescence (TL) dating of heated artifacts. The subsequent development of optically stimulated luminescence (OSL) dating and protocols such as the single aliquot regeneration protocol (SAR) have increased the accuracy and precision of luminescence dating significantly. Quartz-based SAR-OSL dating is now generally considered as a reliable chronological tool, and it is increasingly used in a wide variety of (geo)archaeological studies. Optical dating of quartz has recently been implemented at Babeș-Bolyai University (Cluj-Napoca, Romania). This paper documents the instruments and methods as used in this newly-established state of the art luminescence dating laboratory.

Key words: luminescence dating, OSL, TL, quartz.

1. INTRODUCTION

Thermoluminescence (TL) methods are firmly established for testing the authenticity and dating of art ceramics and other heated materials. Optically stimulated luminescence (OSL) dating of the sediments found at archaeological sites has broken new grounds, greatly aiding to the understanding of different archaeological contexts. Luminescence dating typically covers an age range of a few years to ~100 000 years, with a minimum relative error on the age of ~6%. Under favorable circumstances ages outside this range can be obtained. Comprehensive reviews have been dedicated to applications of luminescence dating in (geo) archaeological contexts [1-3].

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In recent years, there have been a series of instrumental and methodological advances [4-12], through which optical dating of quartz has become the mainstream application in luminescence dating research. Despite this however, the method has not been implemented in Romania up to very recently. Following a few successful dating applications of our group in the newly-established state of the art luminescence dating laboratory in Cluj Napoca [13-17], this paper summarizes the methodology used in state of the art luminescence techniques applied in dating studies in Romania so far.

2. PRINCIPLES OF LUMINESCENCE DATING

Luminescence phenomena encompass a very wide range of processes. However, for the purpose of dating only optically stimulated luminescence (OSL) and thermoluminescence (TL) processes are of interest [4]. OSL and TL is the luminescence emitted from an irradiated insulator or semiconductor during exposure to light or heat, respectively. OSL and TL are stimulated processes, and they are usually accompanied by photoconductivity phenomena. The luminescence signal emitted is dependent on the irradiation history of the sample. Although the processes giving rise to luminescence phenomena in natural wide-gap crystals are complicated and not fully understood this phenomenon can be exploited for dating.

The latent dating information is carried in the form of trapped electrons. These electrons are produced by the interaction of the nuclear radiations with the atoms of the mineral and then may get trapped in certain defects of the crystalline structure. The radiation (α , β , and γ) is emitted by the naturally occurring long-lived radionuclides ^{232}Th , ^{238}U , ^{235}U and their daughters, ^{40}K and ^{87}Rb . There is also a contribution from cosmic radiation. The number of trapped electrons is a measure for the total dose – the paleodose or the archaeodose (the total amount of energy absorbed from the ionizing radiation) the mineral has received over a certain period of time. If also the rate is determined by which the mineral has been absorbing the dose – the annual dose, this period of time (*i.e.* an age), can be determined.

$$\text{Age [Ka]} = \text{Equivalent dose [Gy]} / \text{Dose rate [Gy/Ka]}. \quad (1)$$

The paleodose or the archaeodose is a combined dose, resulting from exposure to α , β and γ radiation and thus it cannot be measured directly. Therefore, it is determined as an equivalent dose, *i.e.* the amount of artificial dose delivered by irradiation in the laboratory that induces a luminescence signal identical to the natural one in the sample. The range of alpha particles and beta and gamma radiation are very different in any medium. The different penetration powers can be exploited to facilitate the dose-rate calculations, namely by selecting only a certain grain size fraction for the luminescence measurements. Fine-grain techniques use minerals with a grain size in the range of 4 to 11 μm . These grains are small enough

to ensure complete penetration by all three types of radiation. Coarse-grain techniques use grains which are large enough (of the order of 100 μm) to enable removal of the outer surface layer that was affected by alpha radiation. This leaves only the contribution from gamma rays and a partially attenuated contribution from beta radiation. The annual dose is derived by calculation based on the estimation of environmental nuclide concentrations.

The moment that is dated with luminescence techniques is a zeroing event during which all the effects on the mineral of its exposure to radiation are removed. In the case of pottery, the zeroing is caused by the firing. For sediments, the zeroing event is the exposure of the sediment grains to sunlight.

The choice of mineral and grain size used for dating usually depends on the availability within the sample. The use of a mineral fraction instead of a mixture of minerals is advantageous because it gives a much better insight of the luminescent phenomenon. In the last years quartz seems to have become the mineral of choice in luminescence dating. This is caused by the development of single-aliquot procedures, more specifically the single-aliquot regenerative-dose protocol [8, 9] that has proven to be very reliable, and to give reproducible and accurate results [18-21]. Furthermore, there has been progress in understanding the mechanism of luminescence from quartz. Kinetic models have been developed [22], while the linear modulation technique allows the identification and characterization of the different components of the OSL signal [23]. The properties of quartz as a natural luminescence dosimeter have been recently reviewed [24].

3. EQUIVALENT DOSE MEASUREMENT

3.1. Sample and aliquot preparation

Conventional procedures are used to extract coarse sized quartz respectively fine quartz grains in our laboratory. All sample preparation is carried out under subdued red and orange light. Samples are gently crushed and treated with diluted HCl (10%) to remove carbonates, and then repeatedly washed with distilled water. To remove organic materials, 10%, respectively 30% H_2O_2 is added and then samples are subsequently washed several times with distilled water. Following this pretreatment, wet sieving is performed (125 μm – 90 μm – 63 μm). After sieving, the left fraction consists of an undifferentiated mixture of minerals, including quartz, feldspars and heavy minerals. These minerals have slightly different densities and for coarse grain dating they can be separated in suspension using heavy liquids. The heavy liquids used in our laboratory are solutions of sodium metatungstate $\text{Na}_6[\text{H}_2\text{W}_{12}\text{O}_{40}] \times \text{H}_2\text{O}$ (a heavy inorganic salt) with distilled water. First the polymineral sample is suspended in a solution with a density of 2.62 g/cm^3 . This enables the separation of quartz and plagioclase grains from the

lighter minerals such as clay, potassium and sodium feldspars. Subsequently, the mineral grains are suspended in a solution with a density of 2.75 g/cm^3 . In this case, quartz and plagioclase feldspars will float while heavy minerals such as zircons and apatite will sink. The floating fraction is kept, washed and dried. Quartz can no longer be separated from plagioclase by means of difference in density. Therefore, a treatment with 40% hydrofluoric acid (40 min) is performed. The feldspars are less chemically resistant and are dissolved. The etching also removes the outer surface of the quartz grains, which reduces the external alpha particle contribution to these grains to a negligible level. A wash with warm (50°C) dilute HCl is applied after etching to remove any precipitated fluorides. This is followed by repeated washing with distilled water and drying. Finally the product is sieved again to obtain the desired grain size.

To extract fine grains, the fraction less than $11\mu\text{m}$ is isolated by settling in Atterberg cylinders according to Stokes' law. For removal of feldspars and obtaining quartz fine grained samples attack with 35 % hydrofluorosilicic acid (H_2SiF_6) (5 days) is performed.

For measurement coarse quartz grains are mounted on stainless steel disks using silicon oil as adhesive. Quartz fine grains are deposited on aluminum disks by acetone suspension.

3.2. Testing the purity of quartz extracts

Complete feldspar removal from samples is desirable and several tests have been devised to check the purity of quartz extracts.

In most studies, a signal above background in response to IR stimulation is attributed to feldspars, as at room temperature the fast component of quartz OSL is not stimulated by IR, whereas a large variety of feldspars respond to IR excitation by emitting in the UV [25]. Fig. 1a shows the OSL response to 100 Gy from an aliquot of 63-90 μm sedimentary quartz extracts (originating from a loess like deposit from Râmnicului Plain in SE Romania) compared to the OSL response of the same aliquot that has been priorly IR stimulated. It can be noticed that the signals are indistinguishable. The insert shows the IR signal recorded after an irradiation to 100 Gy. It can be noticed that the IR signal is negligible compared to the OSL response. The above mentioned observations imply that the signal is quartz dominated. It can be observed as well that the OSL signals rapidly decay, which is characteristic to quartz CW-OSL signal dominated by a fast component.

The "IR depletion ratio" test [26], uses the ratio of the sensitivity corrected [post-IR]-OSL, ($L_{x \text{ post-IR}} / T_{x \text{ OSL}}$) to the sensitivity corrected OSL ($L_x / T_{x \text{ OSL}}$). This test is based on the assumption that the UV recombination centers in feldspars responsible for IRSL and OSL share the same source traps and can be applied to every aliquot analyzed by making an additional measurement at the end of each SAR run. Thus, the sensitivity to infrared stimulation is defined as significant if the

resulting signal amounts to more than 10% of the corresponding OSL signal [21] or if the IR depletion ratio deviated more than 10% from unity [26] and any analyzed aliquot has to fulfill these criteria in order to be accepted. Another confirmation of the purity of quartz extracts comes from observing the 110°C TL peak (which is characteristic to quartz [24]) during thermal treatments.

A general observation that has been made is that during measurement the OSL signal from quartz tends to decrease rapidly, and to a low residual level, while for feldspars the initial decrease is slower. This different behavior potentially provides a mean of differentiation. It is known as well that pulsed optically stimulated luminescence characteristics of quartz and feldspars are very different, and the use of differences in luminescence lifetimes to discriminate between quartz and feldspar signals under pulsed stimulation has been suggested [27]. As luminescence lifetimes associated to the fast component of quartz OSL are around 30 μs , using a pulse timing of this magnitude leads to virtually recording only the quartz signal during the off time as feldspar signals are dominated by a fast lifetime component which rises and falls with the on-period of the stimulation LEDs. Fig. 1b presents a comparison of decay curves obtained on fine (4–11 μm) grains of quartz extracted in our laboratory from Romanian loess (Mircea Vodă section) under continuous wave stimulation, respectively pulsed stimulation on time – 50 μs , off time – 50 μs . It can be observed that the decay rates are indistinguishable proving that any feldspar contamination is negligible.

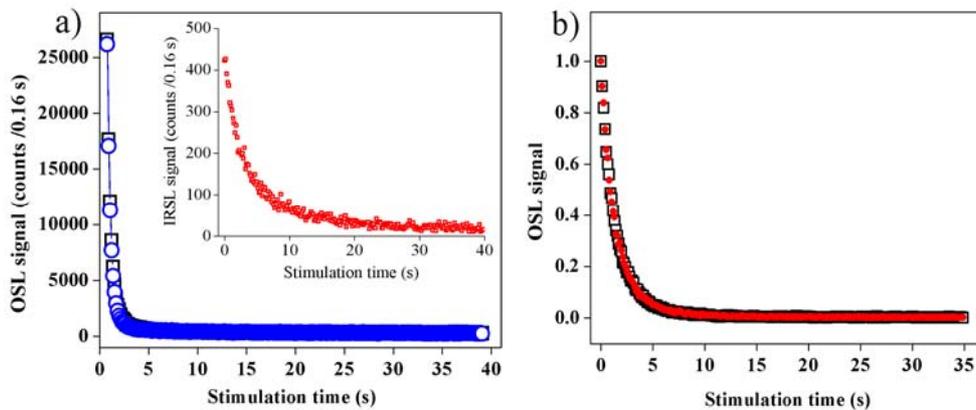


Fig. 1a – OSL response (open squares) to 100 Gy from an aliquot of 63–90 μm sedimentary quartz extracts compared to the OSL response of the same aliquot that has been priority IR stimulated (open circles). The insert presents the IR response of the same aliquot. OSL signals have been measured at 125°C, while the IR signal has been stimulated at 60°C; b – comparison of decay curves of fine (4–11 μm) sedimentary quartz grains under continuous wave stimulation (open squares), respectively pulsed (on time: 50 μs , off time 50 μs) – solid circles. Signals have been recorded after a preheat of 10 s at 220°C. Signals are normalized to initial intensities (first data channel of stimulation, 0.16s).

3.3. Short description of irradiation and measurement facilities

The Risø TL/OSL–DA-20 automatic measurement system enables measurement of both thermoluminescence and optically stimulated luminescence [7]. The system allows up to 48 samples to be individually irradiated (using an incorporated ^{90}Sr – ^{90}Y beta source), heated and optically stimulated using blue and IR LEDs. For optical stimulation of quartz, blue LEDs emitting at 470 nm (FWHM = 20 nm) are being used. The total power from these 28 LEDs is approximately 40 mW/cm² at the sample position. Stimulation can be performed at constant power (CW-OSL – continuous wave OSL) and/or by linearly increasing stimulation power (LM-OSL – linear modulated luminescence). Additionally stimulation can be performed in pulses (POSL) by making use of a pulsing unit that can be used directly with the standard OSL unit [27]. The emitted luminescence is measured by a light detection system comprising a bialkali EMI 9235QA photomultiplier tube and filters. In the case of quartz the luminescence is detected through a Hoya U-340 filter (transmission between 290 and 390 nm).

The dose rate that a sample receives from a beta source is strongly influenced by effects as build-up, backscattering and attenuation [1]. Thus, calibrations must be carried out for different grain sizes and substrates [28]. For calibrating the beta source on our reader, use was made of the calibration quartz produced by Risø National Laboratory. Calibration quartz of different grain sizes is available. The calibration was performed using the SAR protocol. Values of 0.175 Gy/s for the coarse grains on stainless steel disks, respectively 0.135 Gy/s for the fine grains on aluminium disks have been calculated (reference date June 2008).

3.4. The single aliquot regeneration protocol

The SAR protocol truly revolutionized optical dating, and it is nowadays considered as the technique of choice for determining the dose accrued in quartz [8-9, 29]. The main assumption underlying the SAR protocol is that a single aliquot can be analytically used for determining an equivalent dose. The idea is that following the measurement of the luminescence induced in nature (the natural signal) it is possible to measure the response of the same grains to laboratory irradiations, and thus construct a dose response curve, onto which the natural response can be interpolated. Laboratory irradiations however, results in electrons being trapped in unstable traps as well and thus a thermal pretreatment is needed before any optical stimulation. Furthermore, it has been observed that the sensitivity of quartz TL and OSL signals change as a result of preheating, stimulation and irradiation [29]. In the SAR protocol the response to a fixed dose is used for monitoring sensitivity changes. A typical SAR measurement sequence for quartz as proposed by Wintle and Murray [10–11] is presented in Table 1.

Table 1

Generalized SAR measurement sequence for quartz

Step	Treatment	Observe
1.	Give dose D_i	-
2.	Preheat for 10 s at temperature between 160 and 300 °C	-
3.	Optically stimulate for 40 s at 125 °C	Li
4.	Give test dose T_i	-
5.	Preheat for 0 s at temperature \leq than temperature in Step 2	-
6.	Optically stimulate for 40 s at 125 °C	Ti
7.	Optically stimulate at 280 °C for 40 s	-

The measurement procedure starts with the measurement of the natural OSL signal after the aliquot has been preheated ($D_{i=1}=0$ Gy). A test dose is then given, followed by another preheat and the measurement of the OSL response to the test dose. The test-dose preheat (cutheat) is meant to empty the shallow traps. This measurement cycle is then repeated after a regenerative dose is given, and this is repeated as many times as desired. Various regenerative doses can be used throughout the experiment but the test dose is kept constant. By dividing the regenerated OSL signals by the corresponding test dose signals, a growth curve is obtained, which is corrected for sensitivity changes. Fig. 3 shows a typical growth curve for sedimentary quartz constructed in a SAR protocol.

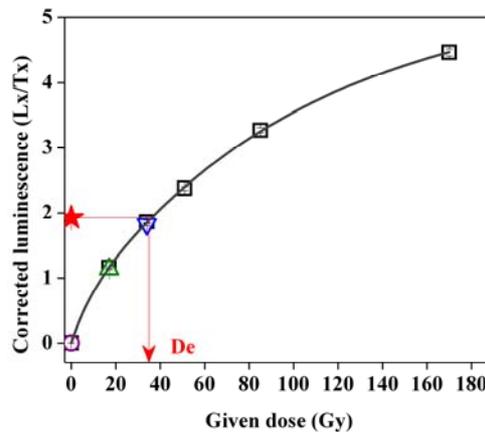


Fig. 3 – OSL dose response curve of coarse 63–90 μm sedimentary quartz extracted from a loess like deposit in SE Romania. Regeneration points are shown as open squares. The up triangle represents the response to the recycling dose, while the down triangle represents the response to the recycling dose prior IR stimulated. The response to zero dose (recuperation) is plotted as an open circle. The natural luminescence signal is depicted as a star. The interpolation of this signal on the corrected dose response curve yields the equivalent dose, 36 Gy in the case of the particular aliquot represented.

In the SAR protocol, it can be checked for if the sensitivity correction is working properly. This is done by repeating a measurement of the response to a

dose that had been previously administrated. The ratio of these two corrected OSL or IRSL signals is called the “recycling ratio” and should ideally be equal to 1. A good recycling value can be also considered as evidence that the growth curve pattern obtained is real and not a sensitivity change artifact [29].

A second important test is to check whether the dose-response curve starts at the origin. This is accomplished by measuring the response to a zero regenerative dose. The “recuperation” of the signal arises from thermal transfer of charge inserted by the test dose into thermally shallow but light insensitive traps which are not emptied by the cutheat. These traps are emptied by the preheat and some of this charge can be trapped into the light sensitive traps. Step 7 at the end of each cycle was later introduced into the SAR protocol to reduce recuperation. For more details, reference is made to works of Murray and Wintle [8–9]. For a sample that behaves well in the SAR protocol, recuperation should be negligible.

The ultimate test for checking the performance of the SAR protocol is the “dose recovery test”. The dose recovery test consists of optically zeroing a sample irradiating it with a known dose and then measuring this given dose using the SAR protocol as if it was an unknown dose. The dose recovery procedure imitates the process in nature, where the sample absorbs the dose before any heating takes place. A successful dose recovery indicates that the first heating does not affect the ability to measure a dose accurately. The dose recovery test gives also indication of the maximum precision available in the measurement protocol.

4. ANNUAL DOSE DETERMINATION

The assessment of the annual radiation dose is an important step in the dating process since it influences directly the final result and its uncertainty is linearly transferred to the age. However, in the determination of the dose rate for luminescence dating it is important to distinguish between the dose absorbed by the soil matrix and by the grain showing luminescence. For example, quartz grains separated from the soil are almost free of internal radioactivity, and the outer layer of large grains can be etched, reducing thus the contribution of alpha radiation to zero. On the contrary, the fine grain technique is concerned only with grains that have received the total matrix dose. In particular, humidity is also an important factor, influencing mostly the alpha dose received by grains [1]. Therefore the annual dose rate of radiation has to be divided at least into three contributions to the varied absorption of alpha, beta and gamma radiation.

Thus, the determination of the dose rate in luminescent dating implies two steps. Firstly, the activities of the radioisotopes in the sample have to be measured and then the relevant activities have to be converted to the energy released per disintegration. This energy has to be related to the alpha, beta and gamma contribution. Conversion is performed using factors calculated from nuclear data

tables, the most recent system being the one proposed by Adamiec and Aitken [30]. The equation for the annual dose in luminescence dating can be written as:

$$\text{Annual dose} = a \cdot \frac{D_{\alpha(U,Th)}}{1+1.5WF} + \frac{D_{\beta(U,Th,K,Rb)}}{1+1.25WF} + \frac{D_{\gamma(U,Th,K)}}{1+1.14W_1F} + D_{\text{cos}}, \quad (2)$$

where: a – is the alpha efficiency factor, W – the water saturation level ($W=W_1$ in the case of sediment dating, W refers to the water saturation level of the ceramic shred in the case of pottery dating, W_1 refers to the water saturation level of the surrounding soil in the case of pottery dating), F – fraction of saturation, D_α , D_β , D_γ – the α , β , γ dose rate contribution derived from radioactive elemental concentration or specific activities using conversion factors, and D_{cos} is the cosmic dose rate. Expressions are available in the literature for calculating cosmic dose rates at any depth, altitude and geomagnetic latitude [31].

Annual dose is determined based on radionuclide concentrations measured in our laboratory by means of high resolution gamma ray spectrometry using an ORTEC hiperpure germanium detector (1.92 keV FWHM and 34.2 % relative efficiency at 1 332.5 keV). A relative efficiency calibration is being used based on IAEA standards. ^{234}Th , ^{226}Ra , ^{214}Pb , ^{214}Bi , ^{214}Bi , ^{210}Pb , ^{228}Ac , ^{208}Tl , and ^{40}K are being routinely measured.

5. AGE CALCULATION AND UNCERTAINTY ASSESMENT

Once the equivalent dose and the annual dose are known, an age can be computed easily by simply calculating the ratio of these two values. However, error assessment is a particularly tedious aspect of luminescence dating. An example is presented in Table 2. Uncertainty in the age comes from both uncertainties in the equivalent dose and in the dose rate. The uncertainty will comprise systematic uncertainties and random uncertainties arising from Poisson statistics associated with counting and sample to sample variations. Error assessment in luminescence dating is summarized in Aitken 1985 Appendix B [1]. Distinction is being made between random and systematic errors. A percentage error in the date corresponding to a given error in each of the quantities and parameters on which the date is based is calculated and then to obtain the overall error the square root of the sum of squares of the individual errors is calculated. This assumes the sources of error are uncorrelated and that the scatter of values for a quantity has a Gaussian distribution. The overall systematic and random errors affecting an age are quoted separately. The random error on a date can be reduced by averaging the results for a number of contemporary samples, but this is not true for systematic errors. As systematic errors are the ultimate barrier for better precision the relative final error on luminescence ages varies on context and is generally not better than 6% on a 68% confidence level.

Table 2

Error budget in SAR-OSL dating of coarse (90-125 μm) quartz extracted from archaeological material -coeval ceramic sherds, previously dated by our group [13]

Sample code		A1	V1	V2	V3
Age + total error (Ka)		6.36±0.53	5.45±0.45	5.13±0.36	7.18±0.56
Relative random error		4.11%	2.94%	1.87%	3.18%
Relative systematic error		7.18%	7.80%	6.86%	7.2%
Error source	Error type				
D _e estimation	Random	4.06%	2.80 %	1.56%	3.07%
Radionuclide specific activity measurement	Random	0.65%	0.90%	1.02%	0.83%
Beta source calibration, efficiency calibration of gamma spectrometer	System.	3.81%	3.81%	3.79%	3.79%
Conversion factors, attenuation and etching factors	System.	4.12%	4.09%	3.97%	3.94%
Cosmic dose rate	System.	0.93%	0.95%	1.02%	1.04%
Internal radioactivity	System.	0.56%	0.57%	0.61%	0.62%
Water content	System.	4.35%	5.33%	3.93%	4.47%
Final age of the 4 pottery fragments 6.21 ± 0.45		Relative final systematic err 1.33%		Relative final random error 7.21%	

6. CONCLUSION

In the past years luminescence dating has undergone significant advances and at the moment is characterized by a degree of quality control seldom seen with other dating techniques. As far as archaeometry is concerned, the method is extremely powerful especially in archaeological and geoarchaeological contexts where no organic material is found, in cases where controversial radiocarbon ages are being obtained or simply for dating of Paleolithic sites, where radiocarbon calibration can be doubtful or even impossible. By their virtue to date the target (*i.e.* archaeological) event directly (manufacturing of ceramics or sediment deposition) luminescence dating methods are can be as well very advantageous in certain contexts. The implementation of TL and OSL dating methods in Romania allow age determination for sediments and archaeological heated materials. As the demand for absolute age determination in our country is high, and a couple of other luminescence dating laboratories in our country are being under development (in Bucharest, respectively Iași) it is hoped that the foundations have been laid for making routine luminescence dating possible.

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