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SAR OSL DATING OF 63-90 µm QUARTZ EXTRACTED FROM AN EEMIAN (PRESUMABLY LACUSTRINE) SEDIMENTARY SECTION AT FLOREŞTI ON THE SOMEŞUL MIC VALLEY

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Abstract: A double-SAR OSL protocol was applied on 63-90 µm quartz extracted from an exposed sedimentary succession from Floreşti on the Someşul Mic Valley, central-northern Romania in order to determine its depositional age. The equivalent doses measured for the three samples analyzed were 291 ± 17 Gy, 331 ± 27 Gy respectively 348 ± 17 Gy. The optical ages obtained were found in agreement within error limits 139 ± 15 ka, 133 ± 16 ka and 134 ± 13 ka, respectively. These new chronological data support the assumption that the analyzed sedimentary record was deposited during the Eemian Interglacial in periglacial settings (probably lacustrine deposits with high sedimentary input).

Key words: OSL, Quartz, SAR protocol, Eemian, lacustrine deposits

1. INTRODUCTION

Optically stimulated luminescence (OSL) of quartz is a powerful dating method, which plays a key role in providing absolute chronologies for late Quaternary sediments (Duller & Wintle, 2011). For decades now, it is highly regarded as an accurate and precise dating method (Murray & Olley, 2002). Luminescence dating plays a major role in palaeoenvironmental reconstruction of the last glacial-interglacial cycle.

Luminescence studies have been carried out in Romania only in recent years (e.g. Timar et al., 2010) the OSL method being mainly applied for establishing absolute chronologies of the key loess sections in the S-E part of our country (Fig. 1) at Mircea-Vodă and Mostiştea (Timar-Gabor et al., 2011, Vasiliniuc et al., 2011, Timar-Gabor et al., 2012, Vasiliniuc et al., 2012 a,b). Other contributions concentrated to the southern part of Romania and include OSL dating of a sedimentary section interbedding the Campanian Ignimbrite/Y5 ash layer (Constantin et al., 2012) at Căciulăteşti site (S-W Romania) and beach ridge sands from the Danube Delta (Vespremeanu-Stroe et al., 2013).

The study area is situated in the western part of Transylvanian Basin at the north eastern border of the Apuseni Mountains (Fig. 1), in a region with scarce data on absolute Quaternary geochronology. The area was previously studied by Posea (1961) who assigned a periglacial origin to the deposits while Pendea et al., (2009) provided the first paleoenvironmental data regarding the Weichselian Pleniglacial based on U-Th and radiocarbon ages along with pedological, palynological and malacofaunal data. Moreover, Timar-Gabor et al., (2010) obtained OSL ages on soils from the western Transylvanian Basin (Feleacu Hills). Fifty km away from our study site, near Turbuţa, Onac et al., (2001) dated a complex Eemian sequence of lacustrine sediments using U-Th TIMS and pollen, in a similar geological setting (Fig. 1).

Given the scarce chronological data, the objective of this study was to apply the optically stimulated luminescence method in order to provide the first absolute depositional ages for the presumed lacustrine sedimentary succession (no marine fossils, frequent plant debris, thick horizontally-laminated sediment) exposed near Floreşti.

Such OSL ages may be combined with future sedimentological and paleontological investigations, as a starting point for stratigraphical and paleoenvironmental reconstructions.

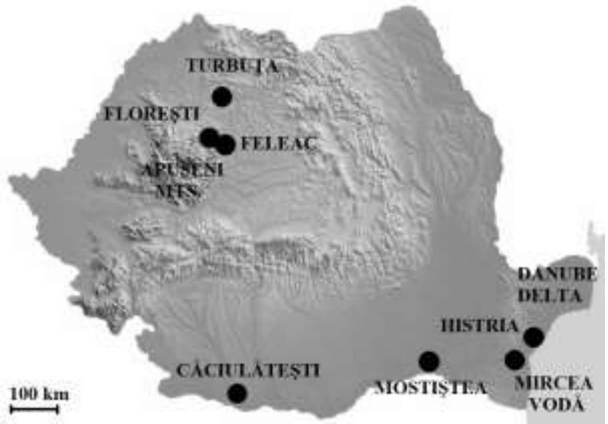

Figure 1. Map of Romania, depicting the location of the studied outcrop (Floreşti). The previously dated sites mentioned in text (OSL and U-Th) are also displayed.

2. GEOLOGICAL SETTING

The analyzed sedimentary outcrop is located on the left bank of the Someşul Mic River, west of Cluj–Napoca, at Floreşti (46°45'3.96" N - 23°28'36.16" E; 380-385 m altitude), in central-northern Romania (Fig. 1). It rests over Eocene sedimentary formations specific in this region (Popescu et al., 1977). The ~15 m thick investigated sequence comprises well-consolidated sandy to clayey sediments overlain by 0.5 metres of Holocene topsoil.

A homogeneous texture and colour with a weakly developed laminated structure can be observed across the entire profile. This reflects a sedimentary environment with low hydrodynamic conditions and a high sedimentary input deposited in a relatively short geological time.

3. MATERIAL AND METHODS

3.1. Principle of OSL dating on quartz

Quartz is one of the most widely used minerals in luminescence dating, due to its abundance in most of the sediments, to its behavioural characteristics, as well as its ability to fulfil the requirements of sensitivity to radiation dose (Preusser et al., 2008).

The method is based on the property of quartz to store the energy of nuclear radiation (Fig. 2a). A low level of nuclear radiation is omnipresent in nature, and the longer the minerals are exposed, the more energy they store. Relevant to dating are the radiation types from the naturally occurring radioelements are alpha particles, beta particles (electrons) and gamma rays, and the cosmic radiation. During mineral exposure to daylight (known as bleaching event) the latent signal is set to zero and, subsequently, when light is blocked by additional sediment, latent signal builds up again through the effect of exposure to ionizing radiation. By stimulation with light, the minerals can release part of this energy by emitting a small amount of light, which is called luminescence (Fig. 2b). The intensity of this luminescence signal is proportional to the total accumulated radiation dose, and hence also to the total acquisition time. The time elapsed since the last daylight exposure of the sediments is represented by the following equation:

$$\mathrm{Age} = \frac{\mathrm{Paleodose}}{\mathrm{Dose\ rate}}$$

The dose rate is defined as the energy that is absorbed from the flux of nuclear radiation. The natural dose rate is expressed as gray per thousand years (Gy ka^{-1}) or as milligray per year ($mGya^{-1}$). The palaeodose (Gy) is the dose absorbed by the minerals grains since deposition. Being a combined dose that results from exposure to α, β and γ radiation, it cannot be measured directly. Thus, in laboratory, it is determined as an equivalent dose (Aitken, 1998).

3.2. Sample preparation

Sampling was performed following a W-E ascending trajectory on a vertical profile of the outcrop. Samples 1T, 2T and 3T, respectively, have been collected at a 12, 10 and 3 m depth. The sediment was extracted by hammering 20 cm-length and 6 cm-diameter stainless steel cylinders into freshly cleaned exposures, perpendicular on the slope face. After withdrawing, the tubes were sealed at both ends by quickly applying light-tight adhesive tape to avoid samples being bleached by sunlight during collection and transportation. The potentially light exposed material from the cylinders extremities was used for dose rate determination while the rest was destined to luminescence analysis.

The quartz extraction and luminescence analysis was carried out in the Dating Laboratory from the Faculty of Environmental Science and Engineering of the Babeş-Bolyai University. The sample preparation for luminescence measurements was performed under low intensity red light conditions. A three-days HCl (concentration of 10%) treatment was employed for carbonated removal, followed by another three-days H_2O_2 (concentration 10% followed by 30%) treatment for organic matter removal. The coarse grain fraction (63-90 µm) was separated through wet sieving. Since this fraction consisted in an undifferentiated mixture of polyminerals including quartz, feldspars and heavy minerals, a two-steps density separation centrifugation using heavy liquids was performed. The heavy liquid solution contained sodium metatungstate $Na_6[H_2W_{12}O_{40}]$ x H_2O (a heavy inorganic salt) and distilled water. First the quartz and plagioclase grains were separated by centrifugation in a 2.62 g/cm^3 heavy liquid solution from the lighter minerals as potassium and sodium feldspars with a density of 2.62 g/cm^3. The second step employed a 2.75 g/cm^3 heavy liquid solution, in which the quartz and plagioclase feldspars were isolated from the heavy minerals such as zircons and apatite.

Quartz grains were isolated from this fraction employing a treatment with hydrofluoric acid HF (40% concentration) for 40 minutes since it no longer could be differentiated from plagioclase by density separation. The etching also removed the outer surface of the quartz grains affected by external ionising alpha radiation, reducing its contribution to the grains to a negligible level (Fig. 2c).

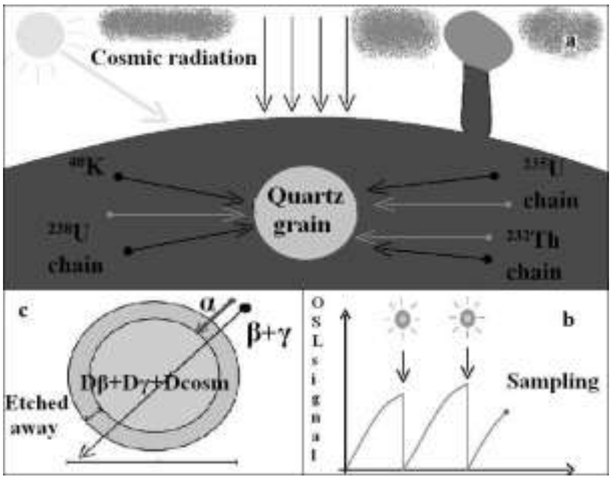

Figure 2. a) Representation of the ionizing radiation from naturally occurring U, Th and K, as well as from cosmic rays. b) Schematic representation of the luminescence dating principles (Preusser et al., 2008). c) Penetrating powers of natural nuclear radiations in 100 μm silicate materials.

Precipitated fluorides resulted after the HF attack was washed away with a 60 minutes HCl (10%). The samples were then resieved using a 63 μm mesh to remove grains that had been reduced in size by the etching. An appreciable amount of mica was observed in our etched quartz after these standard laboratory treatments. Therefore an additional step was introduced in the preparation procedure intended for the mica removal (Kortekass & Murray, 2005). The samples were sprinkled on a dishwashing detergent solution in an ultrasonic bath for ~30 minutes. Subsequently, the solution was decanted and the samples rinsed and dried.

3.3. Analytical facilities and the luminescence measurement protocol

The aliquots were prepared by mounting a monolayer of quartz grains on 9 mm-diameter stainless steel disks using silicone oil. All luminescence investigations were performed on the standard Risø TL/OSL-DA-20 reader equipped with blue LEDs (470 ± 30 nm). IR (875 ± 80 nm) LEDs were used for infrared stimulation. Blue light stimulated OSL signal was detected through a 7.5 mm thick Hoya U-340 UV filter. Irradiations were carried out using the incorporated ^{90}Sr-^{90}Y radioactive source that was calibrated against gamma dosed calibration quartz supplied by Risø National Laboratory. A dose rate of 0.155 Gy/s for the coarse quartz grain mounted on stainless steel disks was obtained. The luminescence analysis of the coarse (63-90 μm) quartz grains were carried out applying a modified single-aliquot regenerative dose (SAR) protocol presented in table 1 (Banerjee et al., 2001; Roberts & Wintle, 2001).

Table 1. The double-SAR OSL protocol

Step	*Treatment*	*Measurement*
1.	Give regenerative dose	-
2.	Preheat to 220 °C for 10 s	-
3.	IRSL at 25 °C for 200 s	-
4.	OSL at 125 °C for 40 s	L_x
5.	Give test dose 15.5 Gy	-
6.	Cutheat to 180 °C	-
7.	IRSL at 25 °C for 200 s	-
8.	OSL at 125 °C for 40 s	T_x
9.	OSL at 280 °C for 40 s	-

It involves an IR stimulation for 200 s at 25^{o}C aimed at depleting the OSL signal from any remaining feldspars prior to stimulation with the blue diodes for 40 s at 125^{o}C, thus ensuring that the measured luminescence is derived only from quartz. The net CW-OSL signal was determined from the initial 0.80 s of the decay curve and an early background subtraction (Ballarini et al., 2007). For correcting for sensitivity change a constant test dose of 16 Gy was used throughout all measurements. High-temperature bleach was performed following each test dose signal measurement, by stimulation with blue LEDs for 40 s at 280^{o}C (Murray & Wintle, 2003).

A thermal treatment consisting of preheating to 220^{o}C for 10 s preceded the IR stimulation prior to the measurement of natural and regenerative signal. A cutheat at 180^{o}C (ramp heating for 0 s at maximum temperature), meant to empty the shallow traps, was applied before the IR stimulations prior to each OSL stimulation of the test dose signals. By dividing each regenerated OSL signal L_x to the corresponding test dose signal T_x a dose-response growth-curve is obtained, which is corrected for sensitivity change.

The performance of the SAR protocol applied was tested in terms of recycling and recuperation. In the recycling test the lowest regenerative dose is measured twice, once at the beginning and once at the end of each individual SAR cycle. If the sensitivity correction is monitored sufficiently, the ratio of this repeated, sensitivity-corrected measurement would be within 10% deviation from unity (Preusser et al., 2009). The recuperation test monitors the thermal transfer from thermally stable, light-insensitive trap to the light-sensitive OSL traps. The L_x/T_x for zero regeneration dose should be < 5% of the natural signal.

3.4. Luminescence characteristics

3.4.1. Equivalent dose estimation

OSL signals of the coarse quartz grains exhibited a rapid decay during optical stimulation. The shape of natural and regenerated signals was found to be indistinguishable from each other (Fig. 3). The aliquots behaved well in the double-SAR protocol. The sensitivity changes occurring throughout a SAR measurement sequence are accurately corrected for (as indicated by the recycling ratios indistinguishable from unity). Recuperation is negligible (<0.3% of the sensitivity corrected natural signal) and the growth curves passed close to origin, as seen in figure 4.

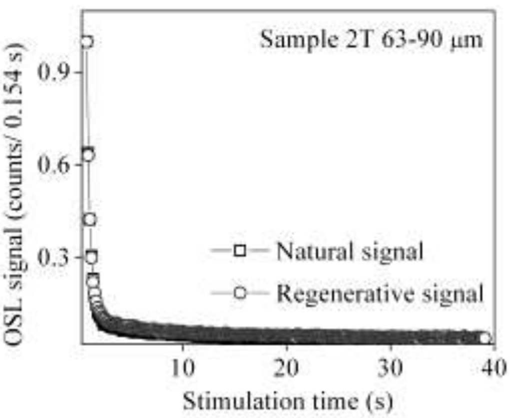

Figure 3. Typical rapid decaying natural CW-OSL signal (open squares) compared to regenerated signals (open circles) from sample 2T.

Equivalent doses have been measured using at least 12 replicate measurements for each quartz sample. The average values (± 1 standard error) are presented in table 2. All equivalent dose values were obtained by interpolating the natural corrected signal on the dose-response growth-curve which was best fitted by a sum of an exponential function plus a linear component.

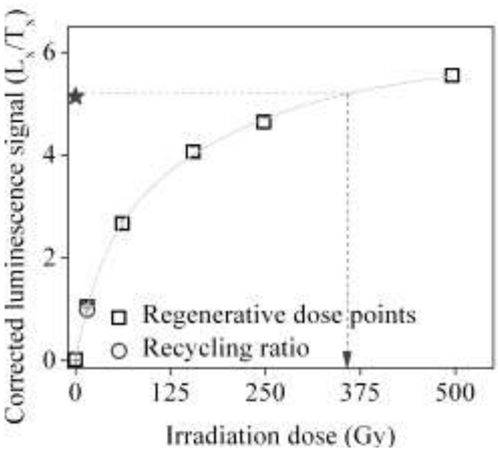

Figure 4. Representative SAR-OSL growth-curve for an aliquot of 63-90 μm quartz from sample 3T. Open squares represent the regenerated doses. The sensitivity corrected natural signal is depicted as a star and the equivalent dose obtained is indicated by arrows. Recycling ratio is depicted as a circle.

3.4.2. Dose recovery test

Further on, we have tested the applicability of the SAR protocol through the dose recovery test. The test consisted in a double bleach for 250 s at room temperature using the blue light emitting diodes, separated by a 10 ks pause between the two stimulations so that any charge transferred into the 110°C TL trap has time to thermally decay (Murray & Wintle, 2003). The aliquots were given known doses chosen to be equal to the estimated equivalent doses, and then measured using the double-SAR protocol. The good recovered per given dose ratio indicate that a known dose given to a bleached and unheated sample can be accurately measured using a preheat of 220°C in combination with a cutheat to 180°C. The dose recovery results are presented in table 3.

3.5. Annual dose rate determination

Radionuclide activity concentrations were measured using high-resolution gamma-ray spectrometry and the dose rates were calculated using the conversion factors tabulated by Adamiec & Aitken (1998). A time averaged water content of 10 ± 2.5% was used to correct the alpha, beta and gamma contributions for the effect of moisture. The beta attenuation and etching factor used in this study for the 63-90 μm fraction is 0.94 ± 0.050 (Mejdahl, 1979) and an internal dose rate of 1.010 ± 0.002 Gy/ka was assumed in the case of the coarser fraction (Vandenberghe et al., 2008). The dose rate contribution from cosmic rays was calculated with the formula found in Prescott & Hutton (1994). The dose rate information is summarized in table 4.

Table 2. Summary for equivalent doses (D_e) of 63–90 μm quartz extract, recycling ratios and recuperation percentage. The uncertainties represent 1 sigma. n is the number of accepted aliquots. All equivalent dose values were measured in a DOUBLE-SAR protocol using a thermal treatment with a preheat of 10 s at 220°C in combination with a cutheat to 180 °C and were determined by interpolating on a dose response curve fitted using the sum of one exponential plus a linear function. Quoted errors represent random errors.

Sample code:	Depth (cm)	D_e (Gy)	Recycling ratio	Recuperation (%)
1T	1500	$291 \pm 17_{n=13}$	1.01±0.02	0.30±0.01
2T	1300	$331 \pm 27_{n=12}$	0.97±0.01	0.31±0.06
3T	200	$348 \pm 17_{n=12}$	0.98±0.01	0.36±0.07

Table 3. The dose recovery test with recycling and recuperations percentage ratios and recovered dose ratio. n represents the number of accepted aliquots. The uncertainties mentioned represent random errors in a 68% confidence level.

Sample code	Given doses (Gy)	Recovered dose ratio	Recycling	Recuperation (%)
1T	1600	$1.12 \pm 0.03_{n=4}$	1.02±0.03	0.32±0.21
2T	1600	$1.09 \pm 0.03_{n=3}$	0.96±0.01	0.67±0.18
3T	1600	$1.13 \pm 0.13_{n=3}$	0.98±0.06	0.23±0.06

Table 4. Luminescence age results for 63-90 μm quartz extracted from Floreşti site. The uncertainties mentioned with luminescence and dosimetry data are random; the uncertainties mentioned with the optical ages are overall uncertainties. All uncertainties represent 1σ.

Sample code	Depth (cm)	Water content (%)	De (Gy)	U-Ra (ppm)	Th (ppm)	K (%)	Total random error (%)	Total systematic error (%)	Total dose rate (Gy/ka)	Age (ka)
1T	1500	10 ± 2.5	291 ± 17 n=13	1.14 ± 0.03	3.97 ± 0.35	1.75± 0.05	6.29	8.41	2.10 ± 0.05	139 ± 15
2T	1300	10 ± 2.5	331 ± 27 n=12	1.72 ± 0.02	6.76 ± 0.12	1.83± 0.05	8.35	8.36	2.49 ± 0.04	133 ± 16
3T	200	10 ± 2.5	348 ± 17 n=12	2.16 ± 0.04	7.79 ± 0.29	1.65± 0.05	5.18	8.16	2.62 ± 0.05	133 ± 13

Note: n represents the number of accepted aliquots. Beta attenuation and etching factor for 63-90 μm assumed to 0.094 ± 0.050. Alpha efficiency factor considered was 0.04±0.02. Radionuclide specific activities have been determined through high resolution gamma ray spectrometry. Cosmic Dose rate – 1T =0.054 ± 0.013 Gy/ka, 2T= 0.063 ± 0.009 Gy/ka, 3T= 0.177 ± 0.027 Gy/ka. Total dose rate includes an external contribution from cosmic rays, the alpha, beta and gamma radiation contribution as well as an internal contribution for the coarse fraction assumed to be 0.013 ± 0.002 Gy/ka.

3.6. Optical ages

Table 4 summarizes the information relevant to age and uncertainty calculation. Uncertainties on the ages were calculated following the error assessment system formalized by Aitken & Alldred (1972). The systematic uncertainty amounts over 8% (1σ) and it originates predominantly from our estimates of the uncertainties associated with the time-averaged water content (25%) and beta attenuation factors. The overall contribution from random sources of uncertainties ranged from around 5 % (sample 3T) to around 8 % in the case of sample 2T. The ages obtained on the 63-90 μm fractions are in good agreement within uncertainties, ranging from 139 ± 15 ka (1T) to 133 ± 16 ka (2T) and 133 ± 13 ka (3T).

4. DISCUSSION AND CONCLUSION

The optical ages place the depositional moment of the sediments into the Eemian Interglacial which, according to Shackleton et al., (2003), extends from 132 to 115 ka.

Based on palinological and $\delta^{18}O$ isotopes data from a long lacustrine sequence in northwest Greece Tzedakis et al., (2003) assigns the 15 ka duration to the last interglacial in southern Europe in agreement with results presented by Shackleton et al., (2003).

The Eemian represents the most recent geological chronostratigraphic interval with conditions mostly resembling the present interglacial. It was characterized by high eustatic sea level, retreat to minimum size of global ice-sheets and establishment of biotic assemblages closely resembling those of the Holocene (Turner, 2002).

Based on isotopic U-Th measurements on snails from a lacustrine deposit located at Turbuţa, 50 km from our site, Onac et al., (2001) obtained ages between 112.2 ± 8.9 ka and 127 ± 6.3 ka. These Eemian absolute ages can be well correlated to the pollen data that suggest a possible early warming phase of the Eemian. Such Eemian warming would have been created the conditions for ice melting from nearby Apuseni Mountains, releasing a high amount of water. Thus, a great sedimentary input in the lower altitude surrounding areas would have been deposited in a relatively short period.

The aforementioned context explains the similar OSL ages obtained on the samples analysed, assigning a simultaneous depositional event at Floreşti. Based on the opinion of Posea (1961) who considered a periglacial origin of the deposit, and its sedimentological features, we can conclude that the analysed sedimentary sequence was deposited in continental (probably lacustrine) settings.

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