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GEOCHRONOMETRIA 41(3) 2014: 256–264
DOI 10.2478/s13386-013-0157-y

Available online at
www.springerlink.com

DIFFERENT APPROACHES TO DATE BRICKS FROM HISTORICAL BUILDINGS

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Received 2 August 2013 Accepted 6 February 2014

Abstract: The application of Thermally (TL) and Optically (OSL) Stimulated Luminescence on bricks used as building material has allowed solving an chronological issue in the field of historical building dating.
The possibility to use one or more methodologies of dating is closely related to the luminescent and granulometric characteristics of the sample. Using some brick samples collected in the church of Sain Seurin in Bordeaux (France), this paper discusses the implications and the possibility to use different approaches and techniques for dating. With this aim luminescence measurements were performed on both polymineral fine grain and quartz inclusion phases extracted from each brick. For Equivalent Dose (ED) and consequently age determination, TL on mixed fine grain fraction (FG), OSL on quartz inclusions (QI) and on mixed fine grain (FG*) fraction, were used. The results obtained suggest the advantage of using OSL technique on fine grain fraction cleaned up by IR stimulation (FG*), but the use of quartz inclusion represents indeed a good alternative.

Keywords: luminescence dating, polymineral fine grain technique, IR stimulation time, [Post-IR] OSL, quartz inclusion technique, preheat plateau.

1. INTRODUCTION

The historical building dating is usually indirectly made by thermally stimulated luminescence techniques (TL) on polymineral fine grained phases extracted from bricks. The first step of the application of this technique was made by Goedicke *et al.* (1981) on Venetian villas (north Italy) dated from the 15th to the 17th centuries. The results appear both precise and accurate using the polymineral fine-grain technique. This was confirmed by other dating works on European historical buildings located in Denmark (Abrahamsen *et al.*, 1998), Czech Republic (Cechák *et al.*, 2000), Finland (Hutt *et al.*, 2001), Germany (Göksu and Schwenk, 2001) and Italy (Martini and Sibilia, 2001).

Some authors (Bailiff and Holland, 2000; Bailiff, 2007) found the reliability of this technique when applied to English ceramic materials to be inconsistent. They, alternatively, used optically stimulated luminescence techniques (OSL) coupled with Single Aliquot Regeneration (SAR) protocol (Murray and Wintle, 2000, 2003) on quartz inclusions extracted from brick, obtaining advantages in terms of signal sensitivity compared with thermoluminescence (TL) ones.

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ISSN 1897-1695 (online), 1733-8387 (print)

Starting from a polymineral fine grained phase, etching procedures were used to remove the feldspar component with good results (Prasad, 2000; Mauz and Lang, 2004) and then was possible to apply OSL measurements and SAR protocol for the determination of the equivalent dose. However the attempts to remove feldspars using various chemical etching procedures have generally not yielded satisfactory samples. Prasad (2000) describes an HF treatment procedure for dissolution of fine grained feldspars from polymineral samples with mixtures of quartz and different percentages of feldspars. The results obtained suggest that only samples with up to 40% of feldspars have a high chance of success using etching procedures. In the case of high feldspars contamination is useful to use, for the equivalent dose determination, post-IRSL measurement techniques applied to not etched polymineral fine grain fractions, reported in the literature as double SAR measurement protocol (dSAR) (Zhang *et al.*, 2007; Zhang and Zhou, 2007).

In recent years, the development of multiple independent measurement techniques used on the same sample has led to process of intercomparison between different research groups (Guibert *et al.*, 2009).

The aim of this study was to apply different independent methodologies on each sample in order to reach greater reliability of the final dating results through a comparison between standard thermoluminescence (TL) and optical stimulated (OSL) measurements on different granulometric fractions (polymineral fine grained and quartz inclusions). This approach assumes an even more important role if only a small number of samples can be collected.

2. EQUATION AGE

The use of different granulometric phases, regardless of the methods used (TL or OSL), involves different age equations.

In the case of fine grain polymineral fraction we have:

$$Age = ED/(k \cdot D_\alpha + D_\beta + D_{\gamma+cosm}) \quad (2.1)$$

where ED is the equivalent dose and k is the alpha efficiency, different for TL and OSL measurements. D_α and D_β are respectively, the annual dose contributions derived from alpha and beta decay of the radioactive contents present in the sample and that together give the annual dose from the sample itself. D_γ is the contribution to the annual dose resulting from gamma emissions of the radioactive material present in the environment. The cosmic dose value is mainly due to the latitude and the depth from the Earth's surface (Prescott and Hutton, 1988).

In the case of quartz inclusion, or more generally of coarse grain, we have:

$$Age = ED/(f \cdot D_\beta + D_{\gamma+cosm}) \quad (2.2)$$

where f is the attenuation factor depending on grain size (Mejdahl, 1979). All dose contributions to the annual dose must be corrected by factors that take into account the porosity of the sample and the average moisture level of the sample during its life.

3. MATERIALS AND METHODS

The samples analysed, identified as STS#TL4, STS#TL5 and STS#TL6, are part of a more comprehensive research program within the European research network (GdRE) "Architectural ceramics and dating methods" which involves many European groups working in the field of historical building's dating (Guibert *et al.*, 2009). They come from the church of Saint Seurin (Bordeaux, France), dated by historians around the sixth century. It is one of the oldest religious buildings of Bordeaux which has been renovated and rebuilt between the twelfth and fourteenth centuries. The site is today used as an underground museum of Christian origins of Bordeaux.

Sampling was done in the area under the crypt of the church, where, during an excavation, a Christian burial ground of about 400 m^2 was found.

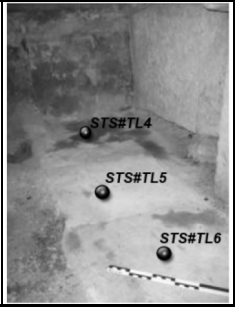
Table 1 shows the details for the samples studied with ID number, sampling area and photo of the sampling points.

Sample preparation

After removal external 2 mm the samples were mechanically crushed using an agate mortar. The crushed material was sieved in order to obtain Ø < 40 μm and 90 < Ø < 150 μm granulometric fractions. The Ø < 40 μm fraction was used to obtain the polymineral fine grained phase and the 90 < Ø < 150 μm fraction was used for the extraction of quartz inclusions. Both the fractions were etched in 10% HCl for 100 minutes, then in 10% H_2O_2 for 48 hours in order to eliminate, respectively, carbonate phase and organic part.

The Ø < 40 μm fraction was further separated into two parts. According with standard procedures the first part (used for TL measurements) was etched in 1% HF

Table 1. *ID sample number, sampling area and photo of the sampling points in Saint Seurin Church (Bordeaux, France).*

Site	ID Sample	Sample	Sampling point	Photo
Saint Seurin (Bordeaux, France)	STS#TL4	Brick	Saint Fort Cenotaph (Floor — East side)	
	STS#TL5	Brick		
	STS#TL6	Brick		

for one hour and then in 10% HCl for 25 minutes to remove eventual fluorosilicates. So with a process of sedimentation in acetone, according to Stokes' law, polymineral fine grained $4 < Ø < 11$ µm fraction (FG) was obtained (Aitken, 1985; Guibert *et al.*, 2009; Gueli *et al.*, 2009; 2010).

The second $Ø < 40$ µm part was not etched in HF and the $4 < Ø < 11$ µm sedimented fraction (FG*) was used for OSL measurements (Aitken, 1998).

Starting from the $90 < Ø < 150$ µm range mineralogically undifferentiated phase, the quartz inclusion fraction (QI) was obtained after different steps. Using different densities of sodium polytungstate solution, quartz was separated from feldspars and other silicate minerals; afterwards it was etched in HF (40%, 45 min), to remove the external layers and the consequent alpha dose contribution, and then washed in HCl (10%, 25 min) to eliminate any fluorides produced (Bailiff and Holland, 2000; Bailiff, 2007).

Application of TL dating technique to polymineral fine grain

FG was used for TL dating applying the added dose technique for ED determination (FG-TL-AD) (Aitken, 1985; Guibert *et al.*, 2009). For each sample, aliquots were prepared; the first 6 were subjected to the natural thermoluminescence reading and the others, divided in groups of 6, were irradiated with increasing β doses, and then their thermoluminescent signals were read. TL glow curves were recorded by heating the aliquots up to 500 °C with a 5°C/s heating rate in a nitrogen environment. In order to eliminate the variation of luminescence intensity, due to the small different mass of grains deposed on the aliquots, normalization sequence was made giving the same dose to all the aliquots (Atken, 1985). Temperature region from 300–350°C (with 280–370°C plateau region) was used for the construction of the growth line TL intensity *vs* dose and the subsequent extrapolation of the Q_β value (**Fig. 1**). This last value represents the artificial beta dose needed to obtain a TL signal equivalent to the natural luminescence emission due total absorbed dose. In order to evaluate the possible non-linearity behaviour of the sample at low artificial beta doses, q_β correction was determined from the intercept of the "second" growth curve behaviour (**Fig. 1**) (Aitken, 1985; Guibert *et al.*, 1996). *ED* of (1) was calculated adding Q_β and q_β values. For each sample a study of fading was carried out by comparing the TL signals of irradiated aliquots and those analyzed with a delay of 15, 30, 40 and 45 days between the end of irradiation and the TL measurements (Aitken, 1985). From artificial luminescence signals induced by calibrated beta and alpha doses the luminescence efficiency coefficient k, necessary to correct the alpha dose contribution to the annual dose, was determined (Guibert *et al.*, 2009).

Application of the OSL dating technique to polymineral fine grain

Due to the different luminescent optical characteristics of quartz and feldspars is necessary to separate their OSL signals to obtain useful quartz dose estimates. FG* was used for OSL dating applying double SAR protocol (FG*-OSL-dSAR) (Roberts and Wintle, 2001; Zhang and Zhou, 2007; Kim *et al.*, 2009).

To check the degree of feldspars contamination in FG* fraction, the coefficient R was calculated on a group of three aliquots for each sample. It is determined by the ratio between the normalized OSL intensity after IR stimulation (post-IR OSL/T_2) and the normalized OSL emission (OSL/T_1) (Mauz and Lang, 2004). OSL/T_1 represents the OSL signal normalized by T_1 test dose and post-IR OSL/T_2 the OSL emission obtained after IR stimulation normalized by T_2 test dose.

So, in order to eliminate feldspars luminescent contribution, for *ED* determination, before each blue-light OSL stimulation (BOSL) measurement, IR stimulation was applied (dSAR procedure) (Roberts and Wintle, 2001; Zhang *et al.*, 2007). Before dSAR procedure, a test to identify the optimal IR stimulation time was undertaken (Wang *et al.*, 2006). The optimal duration of IR exposure for each sample was chosen considering the results obtained using different times in the range 100–500 s at a temperature of 50°C (Roberts, 2007; Kim *et al.*, 2009).

Because thermal treatment prior to measurements may transfer charge from light-insensitive traps to light-sensitive ones, it is important to investigate the influence of preheating required by *ED* determination procedures (Murray and Wintle, 2000). Preheat plateau tests were conducted for both samples in this study using the dSAR measurement protocol (**Table 2**) (Zhang and Zhou, 2007)

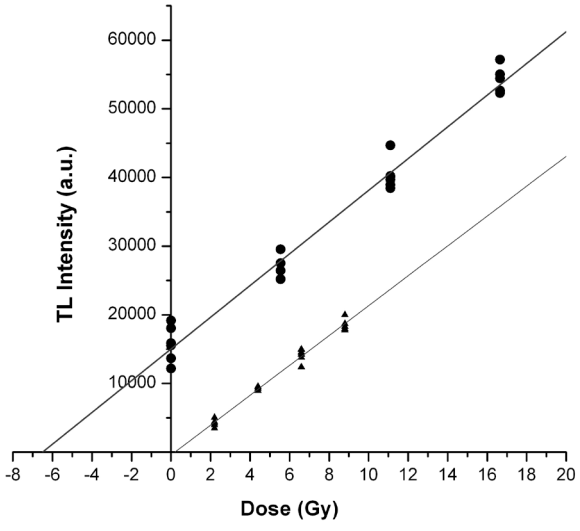


Fig. 1. *Equivalent beta dose determination by added dose technique of the STS_TL#4 sample: Q_β (circles) and q_β (triangles) evaluation from growth curves.*

across a range of temperatures from 140°C to 260°C for 10 s. A cut heat of 160°C was used after the test dose. An IR stimulation duration of 250 s at 50°C was employed as discussed above.

Application of the OSL dating technique to quartz inclusions

The purity of QI fraction was verified by infrared stimulation on some aliquots of each sample (Aitken, 1998). Following the SAR procedure, the *ED* values were then obtained by OSL technique (QI-OSL-SAR) (Murray and Wintle, 2000, 2003). The OSL signal of QI is normally measured following a relatively low preheating treatment, typically from 200 to 220°C for 10 s (Bailiff and Holland, 2000; Murray and Clemmensen, 2001; Ramzaev *et al.*, 2008; Wallinga *et al.*, 2001). These low temperatures are justified in order to limit the heating transfer of charge from deep traps to the OSL traps which influences significantly the dating of this kind of samples (Rhodes, 2000; Murray and Clemmensen, 2001; Wallinga *et al.*, 2001).

For all the samples, the ED variation vs. preheating temperature were evaluated. The aliquots of each sample were grouped in four and each group was subjected to the SAR protocol measurements using different preheating temperatures from 140 to 260°C for 10 s (Kiyak and Canel, 2006). The cycle of SAR protocol was repeated 5 times using increasing regeneration doses D_i from 2 to 10 Gy with a test dose of 1 Gy. A cut heat of 160°C was applied after the test dose.

QI-OSL-SAR subsamples were measured using a SAR procedure as outlined in **Table 2**, but omitting steps 3 and 7, and using a preheat of 180°C in step 2. Each *ED* value is the weighted average of 16 aliquots.

Recovery test

An experimental procedure to detect possible atypical response of each sample at dose values is necessary. This could in fact influence the Single Aliquot Regeneration procedure results. In this work the Recovery test was used.

The dose recovery test consists on the application of SAR on QI grains and dSAR on FG* fractions on aliquots to which a laboratory dose has been given followed by optical bleaching (Murray and Wintle, 2003). In such a test, the ratio of the measured to given dose should be closer to unity.

Three aliquots of each sample were bleached by LED blue light stimulation at room temperature for 10000 s. Later, to the same aliquots, a known dose was administered and then, using the same experimental conditions discussed in the previous sections for both quartz and polymineral fine grain fraction, *ED* values were measured.

Table 3 summarizes the range-size, the etching procedure and the type of analyses performed on each sample.

Table 2. *Sequence used to measure the L_i/T_i signals necessary to obtain ED from FG*-OSL-dSAR subsamples.*

Step	Treatment	Observed
1	Give dose (0 Gy for natural signals), D_i	-
2	Preheat (140–260°C for 10 s)	-
3	Stimulate with IR light for 250 s at 50°C	-
4	Stimulate with blue light for 40 s at 125°C	L_i [post-IR] OSL
5	Give test dose, 1 Gy	-
6	Cut heat to 160°C	-
7	Stimulate with IR for 250 s at 50°C	-
8	Stimulate with blue light for 40 s at 125°C	-
9	Repeat steps 1–8 for 5 times for regeneration doses in the range 0–10 Gy.	T_i

L_i and T_i were derived from the decay curves, taking the first 0.8 s minus a background estimated from the last 3.5 s integral of the OSL signal. L_i/T_i is the sensitivity-corrected [post-IR] OSL intensity.

Annual dose components

The alpha contribution to the annual dose was calculated from the natural U and Th contents measured by ICP-MS using the conversion factors of Guérin *et al.* (2011). This value was compared with the data obtained on thick sample layer using ZnS scintillating discs by an integral alpha counter system (Alpha Counter Unit AL03 model, AEDI, Milano, Italy). This reader allows also coincidence measurements useful to discriminate the alpha contributions coming from both U and Th chains. Indications about possible disequilibrium of U chain were obtained from the comparison between the two evaluated U alpha contributions (Aitken, 1985; Feathers *et al.*, 2008; Stella *et al.*, 2013). K contribution to the beta annual dose was calculated from the content assessed by FUS-ICP (Fusion with lithium borate for ICP) using the conversion factors cited above.

All the dose contributions were corrected on the basis of the porosity factor (*W*) measured experimentally (Aitken, 1985) and water saturation factor (*F*) chosen on the basis of sampling point (height, inside and outside, *etc.*) and water content evaluation of each sample at the excavation. In this particular case an *F* value of 0.5 ± 0.2 was chosen. W was calculated measuring the weight of a fragment under different conditions of damp: totally dry (after having it kept in oven to 40°C for 48 hours) and in saturation (after having left it in water until weight changes of the sample were not found).

The annual environmental dose rate was measured using very sensitive TL dosimeters ($CaSO_4$) enclosed in capsules placed in situ at the sampling points (Guibert *et al.*, 2009; Gueli *et al.*, 2009; 2010). Cosmic radiation was calculated according with Prescott and Hutton (1988). The building is located at 44°50′36″ N latitude and the cover thickness over the sampling area is represented only by the fabric roof. No depth correction was then considered.

Table 3. *Procedures code, range size, etching procedure and analyses performed on each sample.*

Sample	Procedures	Range size	Etching procedure	Analyses performed
STS#TL4	FG-TL-AD	4 < Ø < 11 μm	• 10% HCl for 100 min • 1% HF for 1 hour • 10% HCl for 25 min	• TL with Added Dose technique for ED measurements • Non linearity correction • Fading study
	FG*-OSL-dSAR	4 < Ø < 11 μm	• 10% HCl for 100 min	• Feldspar contamination test • Preheat plateau test • [post-IR] OSL ED as a function of IR stimulation time • dSAR method for ED determination • Recovery test
	QI-OSL-SAR	90 < Ø < 150 μm	• 10% HCl for 100 min • 40% HF for 45 min • 10% HCl for 25 min	• Feldspar contamination test • Preheat plateau test • SAR method for ED determination • Recovery test
STS#TL5	FG-TL-AD	4 < Ø < 11 μm	• 10% HCl for 100 min • 1% HF for 1 hour • 10% HCl for 25 min	• TL with Added Dose technique for ED measurements • Non linearity correction • Fading study
	FG*-OSL-dSAR	4 < Ø < 11 μm	• 10% HCl for 100 min	• Feldspar contamination test • Preheat plateau test • [post-IR] OSL ED as a function of IR stimulation time • dSAR method for ED determination • Recovery test
	QI-OSL-SAR	90 < Ø < 150 μm	• 10% HCl for 100 min • 40% HF for 45 min • 10% HCl for 25 min	• Feldspar contamination test • Preheat plateau test • SAR method for ED determination • Recovery test
STS#TL6	FG-TL-AD	4 < Ø < 11 μm	• 10% HCl for 100 min • 1% HF for 1 hour • 10% HCl for 25 min	• TL with Added Dose technique for ED measurements • Non linearity correction • Fading study
	FG*-OSL-dSAR	4 < Ø < 11 μm	• 10% HCl for 100 min	• Feldspar contamination test • Preheat plateau test • [post-IR] OSL ED as a function of IR stimulation time • dSAR method for ED determination • Recovery test
	QI-OSL-SAR	90 < Ø < 150 μm	• 10% HCl for 100 min • 40% HF for 45 min • 10% HCl for 25 min	• Feldspar contamination test • Preheat plateau test • SAR method for ED determination • Recovery test

Instruments

All measurements were performed using TL-DA-10 and TL-DA-15 automated Risø readers equipped with EMI 9235QA photomultipliers (Bøtter-Jensen, 1997; Bøtter-Jensen *et al.*, 2000). TL glow curves were recorded in the TL-DA-10 detection system using Corning 7-59 and Schott BG-12 optical filters. OSL and IRSL signals were obtained using TL-DA-15 detection system using, respectively, 41 blue LEDs (470 ± 30 nm) and a laser diode (830 ± 10 nm). The stimulation units delivered ~30 mWcm^{-2} for OSL and ~240 mWcm^{-2} for IRSL at 90% power. Both OSL and IRSL emissions were detected in the 260–390 nm region using an Hoya U340 optical filter. Artificial irradiation was performed with an external ^{241}Am calibrated alpha source delivering 2.7 Gy/min and two ^{90}Sr-^{90}Y calibrated beta sources integrated in the TL-DA-10/15 systems delivering, respectively, 1.2 Gy/min and 6 Gy/min.

4. RESULTS

TL measurements

Fig. 1 shows, for sample STS_TL#4, the straight growth lines from which the beta equivalent dose Q_β (a) and the correction q_β (b) were obtained. The parallelism between the two straight growth lines, obtained for each sample, excludes sensitivity changes of the samples due to various heating cycles. No fading was observed within the limits of experimental errors for the bricks tested.

OSL measurements

FG-OSL-dSAR*

The choice to remove the luminescent signal due to feldspars with IR stimulation rather than etching procedures during the sample preparation phase is due to the significant *R* coefficient values obtained for the three samples: 0.38 ± 0.02 for STS_TL#4, 0.39 ± 0.02 for STS_TL#5 and 0.42 ± 0.03 for STS_TL#6.

Fig. 2 shows the change in [post-IR] OSL ED as a function of IR stimulation time for the STS_TL#4 sample. The *ED* increases from a value of ~3.5 Gy when no IR stimulation is used and it reaches a value of ~4.75 Gy after 250 s of IR stimulation. The ED plateau obtained at longer IR stimulation times (from 250 s to 500 s) suggests the choice of the 250 s IR stimulation time in the dSAR protocol. This result was obtained for all three samples.

This is validated by the behaviour of [post-IR] OSL decay curves obtained for the same sample at different IR stimulation time compared to the quartz inclusion ones. In fact the initial OSL signal corresponding to IR stimulation time ≤ 250 s shows differences due to more rapid decay of the quartz respect to feldspars (**Fig. 3**) (Roberts, 2007; Kim *et al.*, 2009).

Plateau tests carried out for the three samples (STS_TL#4, STS_TL#5 and STS_TL#6) show that, considering the experimental errors, no significant changes in the average *ED* value in the whole range of investigation (**Fig. 4**). A preheat temperature of 200°C for 10 s are used for *ED* measurements.

So, the *ED* values considered for dating obtained by dSAR method (range dose from 0 to 10 Gy), based on the results of the tests made, were determined using preheating temperature at 200°C for 10 s and IR stimulation at

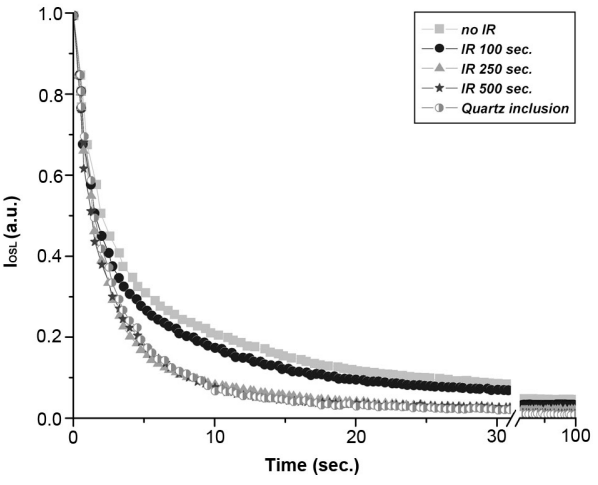


Fig. 3. *The [post-IR] OSL decay curves for the data shown in Fig. 2 related to STS_TL#4 sample. The OSL decay curves from FG* and from QI fraction are compared for IR stimulation times from 0 to 500 s.*

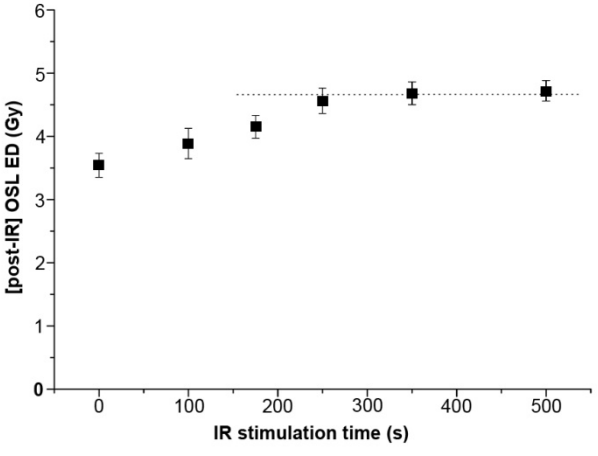


Fig. 2. *Post-IR OSL ED values for FG fraction of STS_TL#4 sample, determined for different IR stimulation times after a preheating of 200°C for 10 s. Each point is the average result from three aliquots (1σ deviation), and the corresponding error is calculated using Analyst version 3.24 (Duller, 2007). The dotted line is the mean of the data points from 250 s to 500 s.*

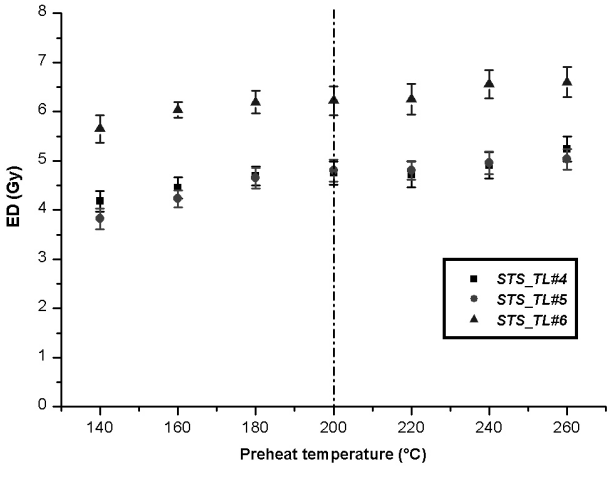


Fig. 4. ED *values (1σ deviation) obtained on FG*-OSL-dSAR subsample for STS_TL#4, STS_TL#5 and STS_TL#6 samples as a function of preheating temperature.*

50°C for 250 s. Each *ED* value is the weighted average of 24 aliquots.

QI-OSL-SAR

An example of plateau test, for the sample STS_TL#4, is reported in **Fig. 5**. It not show significant variations of ED values within the experimental error in the range 160–200°C, while at higher temperatures the *ED* increases. Therefore a preheat of 180°C was adopted.

Recovery test

The dose recovery test confirms the reliability of the measuring parameters used for both SAR and dSAR procedures. For each sample, the value of ratio *R* is closely to 1 (**Fig. 6**).

Annual dose components

The alpha doses obtained from ICPMass procedures are in good agreement with Alpha counting data within the experimental errors confirming the U chain radioactive equilibrium (**Table 4**). So, the alpha dose rate obtained from U and Th contents (ICP-Mass) and the internal beta dose from U, Th, K and Rb were determined.

Table 5 shows the experimentally measured porosity factor (*W*), the saturation factor (*F*) chosen, the k value, the internal dose contributions (D_α and D_β) for each brick together with the external dose component (D_γ).

Age calculation

Table 6 shows, respectively, the *ED* values, the annual dose rates, the dating results for all the subsamples and the corresponding calendar dates.

5. CONCLUSIONS

The results obtained show that in the case where for each technique and method are conducted appropriate preliminary tests to equivalent dose measurements, a good convergence on the final data can be obtained.

In particular, the OSL procedures applied appear to reduce the dispersion on the final age compared to data obtained by TL on the fine grain phase. However the possibility of using a double SAR method for *ED* measurement on fine grain fraction is closely related to the granulometric characteristics of the sample and also to its degree of feldspars content.

In addition, this procedure also needs larger amounts of sample because it is essential to carry out some preliminary studies before the evaluation of the equivalent dose such as the preheating and IR tests to found, respectively, the best preheating temperature and IR stimulation time.

However the samples collected from the Church of Saint Seurin permit to date the structure to the 4th century. The age results obtained are not in contradiction with the stratigraphical and archaeological analysis of the site.

The present study shows that the imposed restraints on historical buildings dating regarding the limited number of

Table 4. *Comparison between internal alpha dose rate values calculated by ICMass and measured by alpha counting system to obtain percentage difference (Δ%).*

ID	Alpha dose rate (mGy/a)		
	From ICPMass content	Alpha counting	Δ%
STS_TL#4	20.96 ± 0.83	20.18 ± 0.78	3.70
STS_TL#5	21.73 ± 0.86	20.99 ± 0.73	3.36
STS_TL#6	15.58 ± 0.60	16.12 ± 0.59	3.49

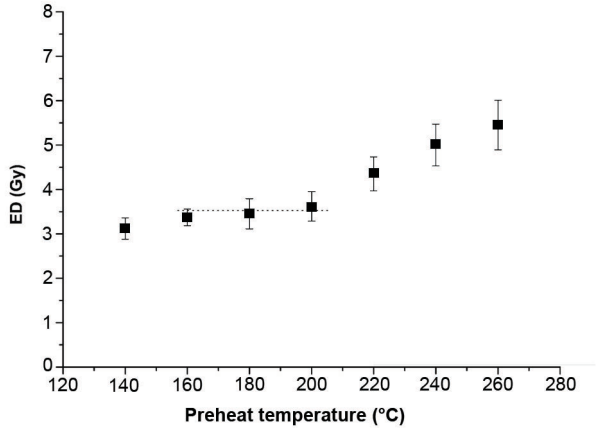


Fig. 5. ED *values (1σ deviation) obtained on QI-OSL-SAR subsample for STS_TL#4 sample as a function of preheating temperature. The dotted line shows the ED plateau region.*

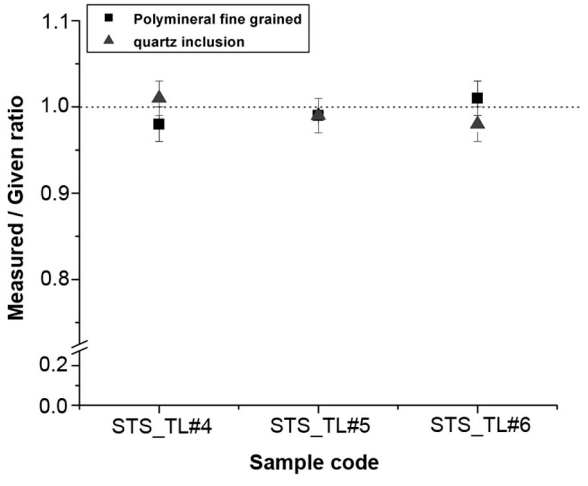


Fig. 6. *Data obtained from dose recovery test (measured to given dose ratio) on the three analyzed samples (1σ deviation). In the dSAR (FG*-OSL-dSAR) and SAR (QI-OSL-SAR) sequences, a preheat at 200°C for 10 s and at 180°C for 10 s, were respectively performed both with a cut heat at 160°C.*

Table 5. *k value determined for fine grain fraction by standard Added Dose (FG-TL-AD), porosity W and saturation factor F, internal D_α, D_β and external $D_{\gamma+cosm}$ contribution to annual dose.*

Sample	Subsample	k	W	F	D_α[(1)] (mGy/a)	D_β[(1), (2)] (mGy/a)	$D_{\gamma+cosm}$ (mGy/a)
STS_TL#4	FG-TL-AD	0.111 ± 0.012	0.161 ± 0.002	0.5±0.2	18.58 ± 1.09	1.14 ± 0.05	1.07 ± 0.03
	FG*-OSL-dSAR	0.041 ± 0.005					
	QI-OSL-dSAR	-			-	1.05 ± 0.05	
STS_TL#5	FG-TL-AD	0.131 ± 0.011	0.157 ± 0.002	0.5±0.2	19.32 ± 1.12	1.17 ± 0.05	0.96 ± 0.03
	FG*-OSL-dSAR	0.042 ± 0.005					
	QI-OSL-dSAR	-			-	1.09 ± 0.05	
STS_TL#6	FG-TL-AD	0.133 ± 0.012	0.218 ± 0.002	0.5±0.2	13.30 ± 0.91	2.18 ± 0.12	1.05 ± 0.04
	FG*-OSL-dSAR	0.052 ± 0.005					
	QI-OSL-dSAR	-			-	2.08 ± 0.11	

[(1)]D_α and D_β are corrected for W and F factors.
[(2)]for D_β by quartz inclusion were used following f correction factors: f_U = 0.92, f_{Th} = 0.91, f_K = 0.96, f_{Rb} = 0.75 (Mejdahl, 1979).

Table 6. *Equivalent dose (ED), Dose rate, Age and Date values determined for each sample on fine grain fraction by standard Added Dose (FG-TL-AD) and by dSAR (FG*-OSL-dSAR), and on quartz inclusion phase (QI-OSL-SAR) by SAR protocol.*

Sample	Subsample	ED (Gy)	Dose rate (mGy/a)	Age (a)	Date (A.D.)
STS_TL#4	FG-TL-AD	6.88 ± 0.51	4.27 ± 0.34	1610 ± 150	400 ± 150
	FG*-OSL-dSAR	4.75 ± 0.22	2.97 ± 0.24	1600 ± 100	410 ± 100
	QI-OSL-dSAR	3.45 ± 0.14	2.13 ± 0.12	1620 ± 110	380 ± 110
STS_TL#5	FG-TL-AD	7.82 ± 0.72	4.66 ± 0.36	1680 ± 180	330 ± 180
	FG*-OSL-dSAR	4.82 ± 0.19	2.94 ± 0.23	1640 ± 100	370 ± 100
	QI-OSL-dSAR	3.30 ± 0.11	2.05 ± 0.11	1610 ± 100	390 ± 100
STS_TL#6	FG-TL-AD	8.21 ± 0.47	5.00 ± 0.45	1640 ± 120	370 ± 120
	FG*-OSL-dSAR	6.32 ± 0.33	3.92 ± 0.35	1610 ± 110	400 ± 110
	QI-OSL-dSAR	4.96 ± 0.16	3.13 ± 0.20	1580 ± 110	430 ± 110

collected samples could be avoided crossing the results obtained on the same sample from different protocols.

ACKNOWLEDGEMENTS

This work was realised within the framework of the European research network (GdRE) "Architectural ceramics and dating methods". Special thanks to Armel Bouvier of the University of Bordeaux 3 and Pierre Guibert, director of CRPAA du CNRS de Bordeaux, for valuable discussions. We are pleased to acknowledge the technical assistance of Nunzio Giudice, Nunzio Guardone, Antonio Rapicavoli and Vito Sparti.

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