DIFFERENT APPROACHES TO DATE BRICKS FROM HISTORICAL BUILDINGS

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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 Saint 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 poly-mineral 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.
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 the 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:
\[ Age = ED/(k \cdot D_{a} + D_{b} + D_{\gamma\cos}) \]  (2.1)
where \( ED \) is the equivalent dose and \( k \) is the alpha efficiency, different for TL and OSL measurements. \( D_{a} \) and \( D_{b} \) 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. \( D_{\gamma} \) 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:
\[ Age = ED/(f \cdot D_{b} + D_{\gamma\cos}) \]  (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 m² 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 \( \varnothing < 40 \mu \text{m} \) and \( 90 < \varnothing < 150 \mu \text{m} \) granulometric fractions. The \( \varnothing < 40 \mu \text{m} \) fraction was used to obtain the polymineral fine grained phase and the \( 90 < \varnothing < 150 \mu \text{m} \) fraction was used for the extraction of quartz inclusions. Both the fractions were etched in 10% HCl for 100 minutes, then in 10% H₂O₂ for 48 hours in order to eliminate, respectively, carbonate phase and organic part.

The \( \varnothing < 40 \mu \text{m} \) fraction was further separated into two parts. According with standard procedures the first part (used for TL measurements) was etched in 1% HF.

<table>
<thead>
<tr>
<th>Site</th>
<th>ID</th>
<th>Sample</th>
<th>Sampling point</th>
<th>Photo</th>
</tr>
</thead>
<tbody>
<tr>
<td>Saint Seurin</td>
<td></td>
<td>Brick</td>
<td>Saint Fort</td>
<td></td>
</tr>
<tr>
<td>(Bordeaux, France)</td>
<td>STS#TL4</td>
<td></td>
<td>Cenotaph (Floor — East side)</td>
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<td></td>
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<td>STS#TL5</td>
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<td>STS#TL6</td>
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<tr>
<td></td>
<td></td>
<td></td>
<td>Brick</td>
<td></td>
</tr>
</tbody>
</table>

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