Luminescence dating applied to medieval architecture

The north east tower of the Avranches keep (Manche, France)

La datation par luminescence appliquée à l'architecture médiévale : la tour nord-est du donjon d'Avranches (Manche, France)

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Abstract: Avranches’ keep remains constitute a witness of Anglo-Norman knowledge on castle building. Their similarity with other buildings such as Ivry-la-Bataille castle or London Tower required determining the place of Avranches keep in this group: pioneer or imitation? Therefore, samples of brick for luminescence dating were taken from the remaining little tower. Results indicate a chronology later than assumed: second part of the 12th century and first part of 13th century. These dates tend to prove that north-east tower remains would correspond to a reconstruction phase and not to the original construction.


Key words: thermoluminescence dating, architecture, early medieval period, Avranches (France).

Mots clé : datation par thermoluminescence, archéologie du bâti, Antiquité tardive, Normandie, Avranches.
The GdRE activities consist of crossing approaches of several fields: on the studied buildings, the data and interpretations collected originate in building archaeology, stylistic study, as well as numerical data provided by physical methods such as archeomagnetism, radiocarbon dating and thermoluminescence. The keep of Avranches is one of the case studies of this group. Archeomagnetism and thermoluminescence were performed in this study in order to date the last firing of the ceramic materials such as the bricks. This crossing of approaches constitutes an asset for this period for which the question of reused material is systematically raised. In this paper, the luminescence dating results are described and integrated in the interpretation of the history of the building.

1. A Sampling Strategy Adapted to the Keep of Avranches, a Controversial Building

The site of Avranches stands 100m above sea level, on a granite massif. It faces the Mont-Saint-Michel bay and its location gives to the town a strategic defensive position. The town is divided in two parts: the old town strengthened on the west and the later town on the east. The keep is located at the junction of these parts, in the most vulnerable area of the city. Two centers are present in the old town: on one side, the religious power represented by the Bishop living in the bishop palace, near the cathedral, on the other side, the seigniorial power whose dwelling seemed to be the keep.

Since 1883, the keep did not exist anymore as such, due to public works which were entailing to its almost complete destruction. The Nicolas-Méry’s researches (2002, 2003) permitted to reconsider the real dimensions of the monument and to place it in the Anglo-Norman castle architecture.

The discovery of a plan dated 18th century and the identification of the early ground coverage of the building allowed us reconstructing its internal spatial divisions. Its reconstructed plan led to indisputable form comparisons with the emblematic Anglo-Norman keeps such as the Ivry-la-Bataille one, that of the Tower of London (fig. 1) or this of Colchester.

The building techniques used in the masonry evidences its age; indeed several masonry sections are built in a herring-bone pattern or "opus spicatum", and stone rubble alternating with rows of bricks are used for the wall facing and in the springs of some arches remaining in the north-eastern part.

The place of the building in the Anglo-Norman castle architecture remains a disputed issue. Whether its edification is one of the first examples of early Anglo-Norman keeps or the result of a continuity of castle building during a war period needs to be ascertained.

Amongst the remains of the original keep, a structure identified by one of us was suitable for a sampling series (fig. 2): it is located at the junction of the surrounding northern wall (north-west/south west oriented) and the load-bearing wall from the northern wall to the south-western wall. A previous passageway through this load-bearing wall, turned into a niche, is built with bricks for the arch and the jambs. Furthermore in the extension of the load-bearing wall, north orientated, i.e. in the thickness of the northern surrounding wall, a second arch presents the same features; this second passageway, which likely opened on the outside, displays internal facings also built up with bricks and

Figure 1: Plans of three Anglo-Norman castles; from left to the right, Avranches’ keep, the London tower, and Ivry-la-Bataille’s keep (Seine-Maritime, France).

Figure 1 : Plans de trois donjons anglo-normands ; de gauche à droite, le donjon d’Avranches, la Tour de Londres, et le donjon d’Ivry-la-Bataille (Seine-Maritime, France).
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associated with rubble disposed in opus spicatum. In order to determine the chronology of the use and the making of these bricks, five brick samples were taken from the two architecturally distinct masonry units mentioned. This was in view of checking the assumption of an anteriority of the Avranches keep on other emblematic buildings which take parts of the large corpus of Anglo-Norman C11th-12th keeps.

Four bricks were sampled from the inside arch, opening on the north in the thickness of the surrounding wall: two samples in the jambs (BDX 8846 and 8847) and two from the arch itself (BDX 8848 and 8849 fig. 3). A fifth sample was taken from the south wall of the previous passageway (turned into a niche) facing the arch where came the four first samples (BDX 8850) from. In spite of the little number of samples available due to the limited area of welle preserved remains, 5 samples are enough to get a sufficiently accurate age of this structure. However, the dates that are to be obtained are only representative of this little part of the whole castle building.

2. EXPERIMENTS AND RESULTS

Methodological aspects

Thermoluminescence dating is described in details in many publications (Aitken, 1985, 1990). We recall that it allows dating the last firing of minerals of items, such as bricks. The age is given by the following age-equation:

\( T = \frac{Q}{I} \)

with \( T \), the time (expressed in years) elapsed since the last firing of the object, \( Q \) (in Gy) is the total dose stored since the last firing (also called “paleodose”) and \( I \), the annual dose rate (in Gy / yr). To measure the time elapsed since the last firing two series of experimentation are performed: the determination of the paleodose and the determination of the annual dose rate. These experimental stages are preceded by a stage of characterization of the material in order to evaluate the most suitable dating protocol.

Brick characterization

Characterization analyses are carried out both on thin and thick sections: the aim is first to evaluate the mineral and elementary composition of the samples, as well as the distribution of the different mineral species. Furthermore possible irradiation heterogeneities are studied, such as those due to the presence of radioactive spots (zircons, monazites, for the most usual species) in the ceramic matrix, even included in quartz crystals used in the luminescence (Blain et al., 2007, Guibert et al., 2009). In addition, checking the homogeneity of a series of samples may reinforce the idea of contemporaneity of the brickmaking.

First observations of the fabric are made with the binocular microscope under natural light. The five bricks show different clay colors which vary from red to dark brown. It can be noticed that the bricks show different grain size characteristics, with white or grey plurimillimetric sized inclusions. The three bricks BDX 8846, BDX 8849 and BDX 8850 are more porous then the two other ones.

In cathodoluminescence, a mauve luminescence is observed which is characteristic of siliceous clays (Bechtel, Schvoerer, 1984). A number of blue and mauve inclusions are visible on the five bricks; they could be K-feldspars and quartz respectively. The two bricks BDX 8848 and BDX 8849 have associations of inclusions of different nature which correspond to K-feldspars and calcosodic feldspars imbricated with quartz crystals, remains of granite rock fragments, in agreement with a possible local origin of the raw material. These polycrystalline or polymineral inclusions are only observed on large size grains, larger than 500 μm, which will restrict the datable grains.

The analysis of the thin sections in polarizing microscopy shows the presence of muscovite and biotite inside some quartz crystals of the brick BDX 8850. These inclusions could also entail a dosimetric obstacle for the study in thermoluminescence (difficulties to calculate the internal dose
Figure 3: (See colour plate) Samples location on the Avranches' keep remains.

Figure 3 : (Voir planche couleur) Localisation des échantillons prélevés pour la thermoluminescence dans le donjon d’Avranches.
rate of the grains), but as they are all plurimillimetric inclusions (more than 5 mm diameter) they will be removed for the dating process.

Finally the elementary composition of the keep bricks is made by EDXS analysis with the Scanning Electron Microscopy (fig. 4).

Therefore the bricks display (table 1):

- a relatively variable elementary composition, especially the ratios Al/Si, Ca and Fe contents, implying a heterogeneity of the raw material or of the origin of the raw material.
- high contents in iron oxides, as it has been noticed for the Mont-Saint-Michel samples (Blain et al., 2007).

A comparison of the variability is established between the data obtained from the X-ray analyses with the SEM and analyses performed with the high resolution gamma spectrometry. The differences noticed in the composition in major contents of the bricks are comparable with the radiochemical composition (fig. 5). Bricks BDX 8847 and BDX 8850 especially show uranium contents much higher than the three other samples.

Finally the data gathered allow using quartz inclusions of “standard” grain size (80-200 μm) for the dating process; dosimetric complexities due to the possible presence of radioactive spots within the quartz grains as it occurred in the case study of St-Philbert-de-Grandlieu, Armorican massif (Guibert et al., 2009) are in this case avoided. Moreover, the variability of composition of the bricks could be interpreted as a possibility of material reuse or a non-uniform chronology of the bricks, what was checked by luminescence dating.

Protocol

After gently crushing the ceramics, quartz are extracted following a three-step process:

- HCl etching (1M) for two hours to dissolve the carbonated material.
- H$_2$O$_2$ treatment for two weeks to dissolve the organic material (50 vol.)
- H$_2$SiF$_6$ etching (31%) during 72-hour cycles, renewed twice or three times.

This last step aims to dissolve selectively the feldspars and aluminosilicates, keeping unaltered the quartz. After each cycle, an observation of the material is performed by cathodoluminescence on small aliquots, in order to monitor the progress of the acid etching and the purity of the quartz powder obtained.

Then grains are heavy liquid separated with a polytungstate sodium solution, to remove the last potential remaining feldspars, as well as the heavy minerals such as zircons or titanium oxides (phases with a density range from 2.63 to 2.67 are kept).

Determination of the paleodose

The process of the added doses and regeneration of the signal is applied for the determination of the paleodose (Guibert et al., 1996). This determination is made with a TL apparatus automated built in the CRP2A (Guibert, 2002). The experimental parameters are the following: heating up to 500°C to a 5°C/s rate in wet nitrogen atmosphere (96% N$_2$, 4% H$_2$O vapour), preheat at 190°C for two minutes. The detection spectral window ranges from 350 to 450 nm, with two Schott BG12 optical filters and one IR fil-

![Figure 4](image-url)

**Figure 4**: Histographic representation of elementary composition of bricks sampled on Avranches’ keep (expressed in weight percentage of oxides).

**Figure 4** : Représentation histographique de la composition élémentaire pour chacun des échantillons de brique prélevés à Avranches, exprimés en pourcentage massique d’oxyde.

<table>
<thead>
<tr>
<th>Elements</th>
<th>BDX 8846</th>
<th>BDX 8847</th>
<th>BDX 8848</th>
<th>BDX 8849</th>
<th>BDX 8850</th>
</tr>
</thead>
<tbody>
<tr>
<td>Na$_2$O</td>
<td>0.65</td>
<td>1.11</td>
<td>0.57</td>
<td>0.78</td>
<td>1.14</td>
</tr>
<tr>
<td>MgO</td>
<td>1.17</td>
<td>1.33</td>
<td>1.18</td>
<td>1.44</td>
<td>1.17</td>
</tr>
<tr>
<td>Al$_2$O$_3$</td>
<td>16.9</td>
<td>17.75</td>
<td>20.2</td>
<td>15.61</td>
<td>15.71</td>
</tr>
<tr>
<td>Si$_2$O$_5$</td>
<td>52.27</td>
<td>58.20</td>
<td>52.3</td>
<td>53.21</td>
<td>54.74</td>
</tr>
<tr>
<td>K$_2$O</td>
<td>4.09</td>
<td>4.38</td>
<td>4.42</td>
<td>4.16</td>
<td>3.99</td>
</tr>
<tr>
<td>CaO</td>
<td>14.51</td>
<td>5.64</td>
<td>10.48</td>
<td>14.98</td>
<td>8.71</td>
</tr>
<tr>
<td>TiO$_2$</td>
<td>0.72</td>
<td>0.73</td>
<td>0.76</td>
<td>0.77</td>
<td>0.62</td>
</tr>
<tr>
<td>Fe$_2$O$_3$</td>
<td>9.69</td>
<td>10.85</td>
<td>10.09</td>
<td>9.04</td>
<td>13.91</td>
</tr>
<tr>
<td>Total</td>
<td>100</td>
<td>100</td>
<td>100</td>
<td>100</td>
<td>100</td>
</tr>
</tbody>
</table>

**Table 1**: Elementary composition in percent of samples from Avranches, obtained by X ray analyses on SEM-EDXS (Scanning Electronic Microscope – Energy Dispersive X-ray Spectrometry).

**Tableau 1** : Composition élémentaire en pourcentage massique mesurée sur les échantillons d’Avranches, au MEB-EDS.
ter, as well as a photomultiplier tube EMI 9813 QA. Irradiations given in the laboratory are made with a $^{90}$Sr-$^{90}$Y β source with a ~0.075 Gy/s (± 1.5%) dose rate.

Several steps are required to determine the paleodose. The first one consists of reading the thermoluminescence of a first set of aliquots, with or without doses added to the natural dose. A second step consists of measuring the thermoluminescence of another set of aliquots previously annealed and irradiated in the laboratory with doses surrounding the irradiations used in the first step. A growth law of thermoluminescence versus added dose is deduced from the experimental data of the second step which is applied to the experimental data of the first step (Guibert et al., 1996).

The annealing conditions for the second step must create an electronic configuration of the material close to its archaeological state, i.e. as close as possible to the electronic state of the quartz after the archaeological firing, which constitutes in Bordeaux a particular physical approach. The TL response of the reirradiated material is studied after annealing during an hour at different temperatures ranging from 400 to 800 °C. By comparing the signal related to the natural dose of the non-annealed material to the signal produced by doses given in the laboratory, the most suitable annealing temperature is determined (best resemblance between the regenerated TL after annealing and natural TL).

Finally since the hexafluorosilicic acid ($\text{H}_2\text{SiF}_6$) etching doesn’t dissolve the external part of the quartz grains, it is necessary to take into consideration the α contribution in the paleodose determination. For the k value, a standard value of 0.08 ± 0.02 is applied instead of measurements since the quantity of material was limited and the importance of alpha particles in the global irradiation of the grains was minor.

Determination of the annual dose

The different contributions of the annual dose are determined by various methods. The γ and cosmic environmental contributions are determined by in situ dosimetry and gammametry. Dosimeters are CaSO$_4$: Tm powder in brass containers which are left nine months on the site. Field gammametry is measured with a portable γ spectrometer (NaI : Tl 2’x2’ Canberra NaI Inspector) for one hour.

The internal contribution is deduced from high resolution γ spectrometry data (Canberra-Eurisys Mesures EGPC 200 P17). K, U and Th contents are converted into α and β doses (Adamiec et Aitken, 1998). Two values of 12 ± 4 % (BDX 8847 and 8848) and 15 ± 5 % (BDX 8846, 8849 and 8850) for moisture content were applied to the samples in view of their sampling location, situated up in the masonry wall or exposed to meteorological conditions. Furthermore these choices were guided by saturation measurements performed on the samples, that exhibited maximal water contents ranging from 16 to 20 %.

3. RESULTS AND DISCUSSION

Luminescence and radioactivity data

The table 2 groups together luminescence data, paleodose values and the annealing temperature used. Radioelement concentrations given in table 3 show that contents in K and Th are similar between brick samples (fig. 5). On the opposite, the U content is significantly more variable, as a possible consequence of the diversity of the raw material. Three values of the equivalent uranium content are determined from the gamma spectra:
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Table 2: Paleodose measured associated to their statistical uncertainty and annealing temperature used before the regeneration of the TL signal.

Tableau 2 : Dose archéologiques mesurées et leurs incertitudes statistiques, ainsi que la température de recuit utilisée avant la régénération du signal.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Paleodose (in Gy)</th>
<th>Statistical uncertainty (in Gy)</th>
<th>Annealing temperature (in °C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>BDX 8846</td>
<td>3.88</td>
<td>0.57</td>
<td>600</td>
</tr>
<tr>
<td>BDX 8847</td>
<td>4.21</td>
<td>0.37</td>
<td>625</td>
</tr>
<tr>
<td>BDX 8848</td>
<td>3.27</td>
<td>0.22</td>
<td>600</td>
</tr>
<tr>
<td>BDX 8849</td>
<td>3.12</td>
<td>0.20</td>
<td>650</td>
</tr>
<tr>
<td>BDX 8850</td>
<td>3.93</td>
<td>0.34</td>
<td>575</td>
</tr>
</tbody>
</table>

Table 3: Components of the annual dose and its global value; uncertainties given at one standard deviation, take into account statistical and systematic errors.

Tableau 3 : Contributions à la dose annuelle et valeur globale de celle-ci. Les incertitudes sont données à un sigma, en tenant compte de l’erreur statistique et systématique.

<table>
<thead>
<tr>
<th>Sample code</th>
<th>K content (ppm)</th>
<th>U(Ca) content (as ppm U)</th>
<th>U(Ra) content (as ppm U)</th>
<th>230Pb content (as ppm U)</th>
<th>Th content (ppm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>BDX 8846</td>
<td>2.63 ± 0.04</td>
<td>2.48 ± 0.18</td>
<td>2.73 ± 0.04</td>
<td>2.59 ± 0.25</td>
<td>9.40 ± 0.13</td>
</tr>
<tr>
<td>BDX 8847</td>
<td>2.90 ± 0.04</td>
<td>3.26 ± 0.24</td>
<td>4.06 ± 0.05</td>
<td>3.99 ± 0.33</td>
<td>9.47 ± 0.15</td>
</tr>
<tr>
<td>BDX 8848</td>
<td>2.89 ± 0.04</td>
<td>2.99 ± 0.16</td>
<td>2.90 ± 0.03</td>
<td>2.23 ± 0.22</td>
<td>10.02 ± 0.12</td>
</tr>
<tr>
<td>BDX 8849</td>
<td>2.31 ± 0.03</td>
<td>2.61 ± 0.4</td>
<td>2.95 ± 0.03</td>
<td>3.18 ± 0.24</td>
<td>8.77 ± 0.11</td>
</tr>
<tr>
<td>BDX 8850</td>
<td>3.09 ± 0.04</td>
<td>5.06 ± 0.23</td>
<td>5.62 ± 0.06</td>
<td>5.18 ± 0.34</td>
<td>9.12 ± 0.13</td>
</tr>
</tbody>
</table>

Concerning the long period isotopes, we can notice that the general trend is a weak disequilibrium in the U-series, the activity of 226Ra being slightly greater than 238U. We consider that kind of disequilibrium is inherited from the raw material, initially in disequilibrium. In addition, with two samples, 210Pb is significantly lower than 226Ra. As the radioactive period of 210Pb is 22 years, we interpreted the discrepancy observed between these last isotopes as a result of a continuous partial radon escape from the bricks that can be extrapolated to the whole age of the material. According to these interpretations, we consider the annual dose as constant since the firing of the bricks, and we will neglect the possible changes of gamma dose-rate due to the modifications of the surrounding masonry.

The annual dose was calculated from the radiochemical data by the use of the Adamiec and Aitken’s conversion factors (1998) taking account of the disequilibrium (Guibert et al., 2009a) and of the attenuation of external alphas and betas within the quartz grains, which were supposed to be non-radioactive. This latter assumption is supported by the examination of sections of bricks that do not exhibit any radioactive inclusion trapped in the quartz grains, unlike what we observed in bricks of other medieval sites of the Armorican Massif area (Guibert et al., 2009b). The transmission factors were determined from Mejdahl’s values for beta particles (1979) and from Brennan et al’s data (1991) for alphas. As the etching of the brick powder keeps more or less intact the outer parts of the quartz grains, we took account of the alpha contribution, supposing a k-factor of 0.08 (±0.02). The effective alpha annual dose is then given by the product of the transmission factor by the alpha k-factor and by the energy released by U and Th series alpha particles coming from the baked earth matrix. To reduce uncertainties in dose transmission factor determination for quartz grains the grain size of which ranges from 80 to 200μm, the quartz grains used for TL measurements were sieved at 100, 125 and 160 μm and the resulting different fractions (80-100, 100-125, 125-160, 160-200μm) were weighted, and the mass proportions obtained were used as weighting factors of the specific factors (assuming that TL intensity is uniform within a grain and is proportional to the energy released in grains and not to the dose). Within these subclasses of grains, the grain-size distributions are supposed to be constant. Table 4 reports the values that were used in this work and gives an example of a dose transmission factor determination (from sample Bdx 8846).

Dates obtained

Table 4 shows the five dates obtained. To these dates are associated uncertainties of two types. Firstly the systematic uncertainties affect equally the dates (calibration of the high resolution gamma-spectrometry, of the radioactive sources used…). Secondly, random uncertainties are taken into account; they are random errors on independent measurements amongst them, the uncertainties due to the counting in gamma spectrometry, the uncertainties due to discrepancy of the TL curves used in the paleodose. The dates are substantially homogeneous, whether for the samples from
the arch or that from the wall. In order to check the contemporaneity of the dates, the statistical \( \chi^2 \) test was applied including BDX 8850. \( \chi^2 \) value for this series is about 1.52, which is slightly outside the theoretical range of 1.61 to 9.24 at the 80 % level of confidence (a slight excess of convergence probably due to an overestimation of uncertainties). Nevertheless, if we only take into account the four measurements made on the arch, \( \chi^2 \) value is about 1.89 which is in the range of 1.06 to 7.78.

Given the high homogeneity of the dates, a weighted mean date was calculated for the samples BDX 8846 to 8849. This choice, taking four samples into account instead of five, can be explained by the fact that these four samples belong to the same architectural unit, i.e. the entrance arch of the tower.

### 4. Archaeological Interpreations

of the Archeometric Data

A first examination of the very good agreement of the dates would have suggested the possibility of a fire, but this hypothesis was quickly rejected. Indeed, besides the absence of written records related to this topic, the examination of the building shows no evidence of fire.

After a second reading, the question of the real event dated was raised. It seems, in light of the historic and archaeological information and the dates obtained, that the studied structure can’t be interpreted as a remain of the original building of the keep of Avranches which is assigned to the first half of the 11th century, chronology based upon the architectural features and historical context (D. Nicolas-Méry, 2003, 77-78). The hypothesis of a reconstruction or redevelopment phase can then be suggested.

The mean date needs to be examined on a historical point of view. By 1204, the king of France Philippe Auguste

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**Table 4**: Alpha and beta transmission factors according to the grain size. The experimental mass distribution of quartz grains of the sample Bdx 8847 is given as an example of the way of determination of the mean dose rate transmission factor and corresponding standard deviation.

**Tableau 4**: Facteurs de transmission alpha et beta en fonction de la granulométrie. Les mesures expérimentales de masse obtenues sur l’échantillon BDX 8847 permettent d’illustrer la détermination du facteur moyen de transmission débit de dose, et de son incertitude.

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**Table 5**: TL dates obtained on samples from the Avranches keep, with their statistical uncertainty and weighted mean date of the intern arch of the tower. Dates are presented after water content correction.

**Tableau 5**: Dates TL obtenues pour les échantillons du donjon d’Avranches, leur incertitude statistique et la moyenne pondérée calculée sur les échantillons appartenant à l’arc intérieur de la tour. Les dates sont présentées corrigées en tenant compte de l’humidité.
regains the crown of England to the duchy of Normandy as well as all the other continental territories. Later, by 1236, saint Louis acquires the town of Avranches and proceeds to the almost complete overhaul of its fortifications (Nicolas-Méry, 2003b; Levalet & Nicolas-Méry, 2006). These circumstances could have been favourable to the reconstruction, partial or total, of the keep of Avranches under the aegis of the royal power.

The end of the 12th century and the beginning of the 13th century constitute a period rich on an architectural point of view (Nicolas-Méry, 2004). That is why some hypotheses can be raised in order to attempt to explain this “hiatus” between the construction of the primitive building and the bricks making period. It could be a “surgical” rebuilding of this part of the masonry where the samples come from, in order to install in the thickness of the north wall an access to the latrines or to create a set back space as often seen in Romanesque keeps. Unfortunately, actual remains don’t allow observing some evidences in the masonry. In this case, we may consider that the builders of this construction chose the use of a material omnipresent in the building in order to preserve the harmony.

Finally, the main fact highlighted by the results obtained is the revelation of a reconstruction phase undetected before. However it would be interesting to search for other bricks which presence is confirmed in 19th century iconographic sources, and preserved in the lowest parts of the north wall of the keep currently hidden by a superimposed retaining wall.

References


