New developments in the scientific dating of brick

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Introduction

Fired clay brick has been widely used in the construction of buildings in many parts of Europe since its introduction by the Romans, and the extremely robust physical properties of fired clay enable bricks to endure within the archaeological record for many centuries, notably as structural elements in standing buildings. Most ancient standing buildings, erected wholly or partially in brick, have undergone alterations since their original construction and consequently usually have a complex history. The current approaches to unravelling building histories have the capability to date the original construction and subsequent alterations to within several years or better where structural analysis combined with searches for documentary evidence and tree-ring dating of timbers is employed. However, for many vernacular buildings, difficulties in dating may arise where documentary evidence has not survived, or may have never existed, where tree-ring dates are not available (such as the replacement of original structural timbers, insufficient number of rings, etc.), and where there is an absence of diagnostic architectural features. In these circumstances the margin of uncertainty in dating may increase by at least several decades, depending on the nature of the available building evidence. This paper discusses the potential of a scientific dating method, luminescence dating, that provides a means of determining the date of manufacture of fired clay brick. Although the luminescence method has become well established in the field of archaeology, it has had limited application to building history. This paper provides a brief introduction to the application of the method and its potential for further development in historic buildings analysis, drawing upon the results of a recent test programme of dating brick from late-medieval and post-medieval English buildings.

Issues of brick use in medieval England

It is generally accepted that was a long interregnum following the departure of the Romans from Britain, during which brick was not manufactured until its relatively late reintroduction towards the end of the 12th century.

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1 I thank the organisers of the Colloquium for inviting me to present this paper. I wish to acknowledge the help of Dr B. Lott, Lincolnshire County Archaeologist, and Dr J. Clark, Field Archaeology Specialists Ltd, University of York, who provided me with much valued guidance and access to reports concerning the study in Lincolnshire.


Evidence of the earliest known local production of medieval brick is found in buildings in Essex, although there are examples elsewhere of early medieval buildings where it is uncertain whether ceramic tiles and bricks incorporated into the structure were re-used or of local contemporary manufacture. It is known that some brick was imported from the Low Countries during the 13th and 14th centuries for the construction of high status buildings, but there is documentary evidence for the emergence of a commerce in brick manufacture in detailed records related to brick production in Hull (Yorkshire) and Wisbech (Cambridgeshire) during the early 14th century. In the county of Lincolnshire, lying between these two known examples of production centres, locally produced brick is found in standing buildings that survive from the late 14th century, although many of these buildings tend to be in isolated rather than in urban settings. During the 15th century the use of brick became fashionable in E. England and the number of brickyards and brick making sites expanded to meet local needs, rising to a peak of nearly 200 production centres in the late 19th century, facilitated by the widespread availability of suitable clays.

Apart from the work performed in our laboratory on buildings in Newcastle upon Tyne and Suffolk, only very limited work on the luminescence dating of brick from buildings in England has been performed (Brixworth Church). Elsewhere in Europe the potential of the application of the method to the study the architectural history of buildings was also recognised, and work on standing buildings in Europe has included applications in the Czech Republic, Denmark, Finland, France, Germany, Italy, Poland and work further afield in Eurasia.

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A brief outline of the method

The luminescence method can be used to date heated archaeological artefacts and deposits such as pottery, brick, burnt flint and burnt clay, and also unheated sediments deposited under suitable conditions. The luminescence chronometer mechanism employs the accumulation and storage of electric charge that has become trapped at ‘defect’ sites in crystals, and luminescence dating is consequently referred to as a trapped charge dating method.

When any material is exposed to ionising radiation, energy is transferred to the material and this is referred to as the absorbed dose. Luminescent crystals, such as quartz and feldspars, located within a ceramic can be used to quantify the amount of absorbed dose either by heating the crystals, giving rise to thermoluminescence (TL), or by stimulating them with light to measure optically stimulated luminescence (OSL). The intensity of the luminescence is proportional to the total absorbed dose received since the crystals were last heated to high temperatures which, in the absence of a fire, occurs during the manufacture of the bricks. During kiln firing all the traps are emptied and the accumulation of trapped charge resumes until the laboratory measurements are performed.

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The luminescent crystals found within a clay fabric experience a radiation ‘field’ arising from the decay of radioactive isotopes of uranium, thorium and potassium that are naturally present in low concentrations in clays and other geological materials in the environment. The intensity of the radiation field in the sample, measured in terms of ‘dose rate’, is related to the quantity of the radioactive isotopes within the brick wall and the immediate environment. Except for certain modulations that are discussed below, it is essentially constant over archaeological timescales because the half-lives of these radioisotopes are extremely long (billions of years).

Between the events of manufacture and sampling, luminescent crystals accumulate an absorbed dose, referred to as the palaeodose, the size of which depends on the length of time between the two events and the size of the dose rate. The palaeodose is determined in the laboratory by measuring the intensity of the luminescence from crystals extracted from the dating sample and comparing it with that measured following the administration of a known absorbed dose using a calibrated laboratory radiation source. This experimentation can be performed using procedures based on the measurement of either TL or OSL. The other experimental task is to determine the dose rate and can be achieved by either measuring the average concentrations of radioactive isotopes in the brick and the surrounding environment or by measuring directly the dose rate using a luminescence technique. The radiation emitted by radioisotopes located within about 1 m from the sample can potentially contribute to the dose rate (typically sources within the brick contribute about 65% of the dose rate and the remainder is from sources beyond it), and hence an assessment of material located beyond a sampled brick is performed. Also, the moisture content of the sample has a moderating effect on the dose rate and an average value since emplacement of the brick is estimated.

The luminescence age is obtained by evaluating the age equation:

\[
\text{Luminescence Age} = \frac{\text{Palaeodose}}{\text{Dose rate}} \pm \sigma_A \pm \sigma_B \text{ (years)}
\]

It corresponds to the time elapsed since firing and it is an absolute age that, in principle, does not need to be referred to a secondary calibration. In keeping with common practice in luminescence dating two uncertainty terms are given at the 68% level of confidence (1σ). The first, \(\sigma_A\), is a ‘type A’ standard uncertainty obtained by an analysis of repeated observations and the second error term, \(\sigma_B\), is a ‘type B’ standard uncertainty based on an assessment of uncertainty associated with all the quantities employed in the calculation of the age, including those of type A, and is equivalent to the overall error described by Aitken. The type A term is smaller than type B (typically ±3% of the age) and is used when comparing dates produced by the same laboratory; the type B term should be used when comparing luminescence dates from different laboratories and dates produced using other methods.

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22 M.J. Aitken, An Introduction to Optical Dating.
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The dating results discussed below were obtained by measurement of optically stimulated luminescence (OSL) with quartz grains in the size range 90-150 μm (100 μm = 1/10 mm), and this offers advantages in terms of signal sensitivity25 compared with thermoluminescence (TL). OSL is the more recently developed (1985 onwards) experimental technique and TL was generally used before 1990 but remains in use, the choice depending on sample characteristics. In testing brick from English buildings we have favoured luminescence measurements with ‘coarse’ grains of quartz extracted from the brick fabric to allow a simpler means of extracting the luminescent quartz grains and avoiding the problem of anomalous fading that is associated with feldspar minerals. If present, anomalous fading can cause an underestimation of the age due to loss of the latent luminescence signal26, the extent of which is not predictable with a sufficiently high degree of confidence. Dating measurements can also be performed with the fine grain (2-11 μm dia.) fraction of samples, where, using the standard laboratory procedure, the luminescence is due to both quartz and feldspar minerals. The fine grain technique has the advantage of reducing the dose rate contribution from sources external to the sampled brick. However we have found the reliability of the fine grain fraction to be inconsistent when applied to English ceramic materials, attributed to the behaviour of the feldspar mineral component, and attempts to remove feldspars using various chemical treatments (e.g., acid etching) have, so far, not produced satisfactory samples. On the other hand other laboratories have reported success in using the conventional fine grain fraction, and the mineralogy of the clays used is likely to play a large part in influencing whether or not the fine grain technique yields reliable dates for ceramics manufactured in a particular geographical region.

Testing the method

To test the potential of the method in an English setting, brick buildings with reliable independent dating evidence were sought for a study that was completed in 200627. The main study area selected was Lincolnshire, and within this county we were fortunate in obtaining access to four buildings suitable as dating controls, three of which had recently completed detailed structural surveys. They comprised Boston Guildhall, Doddington Hall, Tattershall Castle and Ayscoughfee Hall; two further buildings, Fydell House and Clarendon House in Wiltshire, for which some dating evidence was available, were included in the study. The results obtained by testing bricks from these buildings are used to illustrate current application of the method.

26 M.J. Aitken, An Introduction to Optical Dating.
Two issues that need to be considered when planning sampling are:

i) the variation in manufacturing date of bricks used in a wall section selected for dating and

ii) brick-to-brick variation in the luminescence characteristics of the grains extracted for measurement.

In the case of the buildings that provided chronological control (Tattershall, Ayscoughfee, Doddington and Boston Guildhall) there was a high degree of confidence that the contexts sampled represented a single phase of construction and this allowed a relatively small number of samples to be collected. However at Ayscoughfee, for example, of two brick samples obtained from the same section of wall (about 2m apart), the luminescence

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<table>
<thead>
<tr>
<th>Lab ref.</th>
<th>Building</th>
<th>Assigned date range A.D.</th>
<th>Assessment</th>
</tr>
</thead>
<tbody>
<tr>
<td>310</td>
<td>St Mary's Guildhall Boston, Lincs.</td>
<td>1390-1395</td>
<td>Buildings analysis incl. dendrochronology²⁸.</td>
</tr>
<tr>
<td>311</td>
<td>Fydell House Boston, Lincs.</td>
<td>Ph 1. 1705-1710</td>
<td>Documentary sources and date marked ironwork (Ph. 2)²⁹</td>
</tr>
<tr>
<td>315</td>
<td>Clarendon House, Wilts.</td>
<td>Ph 1. 1650-1675</td>
<td>Documentary sources, stylistic dating and datestone,³⁰</td>
</tr>
<tr>
<td>317</td>
<td>Doddington Hall Doddington, Lincs.</td>
<td>1593-1600</td>
<td>Documentary sources³¹ and architectural style³².</td>
</tr>
<tr>
<td>318</td>
<td>Tattershall Castle Tattershall, Lincs.</td>
<td>1445-1450</td>
<td>Documentary sources and architectural style³³.</td>
</tr>
</tbody>
</table>

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³¹ An indenture, dated 1 June Eliz 1593 between John Savyle and Thomas Tailor, who built the Hall, records the purchase of the Estate (Public Record Office, Close Rolls 35 Eliz Part 4; Ref C54/1440); Jarvis, Personal communication.
sensitivity of quartz from one brick was negligible (hence no date was produced), while it was more than adequate in the other. Since the fabric of the sampled bricks appeared similar it was not possible to propose a correlation of fabric and luminescence properties, judged by inspection of the surface. If the sand temper had been drawn from a source of similar geological history, differences in temperature and atmosphere during the firing process are known to affect the luminescence sensitivity, but the physical mechanisms responsible for such differences have yet to be pinpointed. This situation is likely to arise in other buildings and hence replicate sampling is advisable if the brick type has not been subject to testing previously. If judged acceptable in terms of the contained damage to the fabric (i.e., drilling), replicate sampling should also be considered to test for the re-use of brick recovered from an earlier (or later) build on the same site or robbed from a demolished structure elsewhere. Clearly, in dealing with either issue, the assessment of sampling at the level of both location and brick is a matter that requires discussion with a buildings specialist.

**Sampling procedure**

In general, a solid sample of at least 400 g is desirable and the minimum required is currently about 150 g. If locations relevant to the study are available that are not normally not in view (e.g., roof spaces, cellars and foundation walls) the cosmetic effect of sampling is usually not an issue. The approach preferred in our laboratory is to obtain a core that extends from the front face to the rear of the brick, obtained by using a diamond faced core drill. Drills of diameter 30-50 mm diameter provide sufficient material and the drill speed and pressure applied during the cutting are adjusted to prevent excessive heating if the drill bit is not water lubricated. The cores, once cut, are marked to indicate their location and orientation and packed in heavy gauge plastic film. Following drilling the core cavity is filled with lime-based mortar and finally plugged using a thin section of the core that includes the outer surface and a clay colorant in the mortar. This procedure has so far proved acceptable for locations where cosmetic repair is important. An alternative is the extraction of a whole brick by removal of the mortar and, although a lengthier process, it allows a section to be cut from the rear of the brick and subsequent replacement of the brick can be achieved without damaging the front face of the brick. To enable work with coarse quartz grains of the type discussed in this paper, sample extraction by means of drilling powder using a conventional abrasive masonry drill is not possible because of the grinding action of the bit. For very hard bricks it is usually necessary to use a water lubricated diamond core bit, which also allows smaller diameter cores to be cut. One further technical point is that by testing material towards the rear of the sampled brick (~10 cm from the surface for these samples), contributions to the dose rate from radionuclide sources located beyond the immediate vicinity of the sampled core such as adjacent walls and layers in the form of plaster that may have been added at a later stage during the history of the building are reduced due to shielding by the brick material located between the volume sampled for extraction of luminescent minerals and the external surface.

In addition to extracting a core, a dosemeter capsule (a silica tube approx 15 mm dia. x 25 mm long) containing specially prepared crystals is inserted into a hole (~1 cm dia.) drilled into a mortar layer near to the core location to a measured depth sufficient to place it at a depth that corresponds to the
rear half of the brick (usually 10 cm in the case of a wall of at least 25 cm thickness). The period of measurement required is several months or more, after which the capsule is retrieved for measurement and evaluation by the laboratory. The crystals contained in these capsules register the component of the dose rate due to gamma radiation emitted by radionuclides in the wall and in the immediate environment of the sampled location. The direct measurement of the dose rate using a capsule by this means enables the calculated overall error ($\sigma_B$) to be reduced. Depending on the equipment available in the laboratory, in situ measurements may also be made with instruments to measure the intensity of the radiation field in the cavity.

**Age calculation**

Once all the various experimental quantities have been determined, the age equation is evaluated. There are several underlying assumptions to note that are related to the nature of the samples and which have a bearing on the interpretation of the dates obtained. The dose-rate in the age equation represents a value that is the average since construction of the building. Various assumptions and estimations concerning the uncertainty in the dose-rate within the sampled part of the structure are made when calculating the age since the average dose rate is derived from contemporary measurements. When calculating the age it is assumed that the bricks were used shortly after manufacture, that the structure from which they were taken remained intact and that the bricks were not re-used from an earlier building. The moisture content of the bricks affects the dose rate and, in the examples discussed here, a value of 3±1% for the average moisture content was applied when calculating the age in all except one location (Tattershall Castle, 5±1%). This relatively restricted margin of variation makes a small contribution to the overall uncertainty (<3%) in the age and assumes the absence of persistent and significantly higher moisture levels during the history of the building. In general this assumption is supported by studies of the equilibrium moisture content of modern fired clay bricks where, for fabrics with a total porosity of ~35% (70% RH), the absorbed water per unit dry weight was found to be ~3%35. However, it is important to note that significantly larger variations in average moisture content would have a potentially strong effect on the value of the dose-rate, although to obtain a significant change in the average value both magnitude and duration are relevant. The calculated effect of changes in the average moisture content on the age for a given value of palaeodose is illustrated in fig. 1, using data for a sample from Doddington Hall.

It can be seen that as the average moisture content rises, the age increases due to the reduction in dose-rate, and the width of the age distribution (as reflected in $\sigma_B$) is related to the uncertainty specified in the moisture content. In moving from a dry condition to an average of 10% moisture content (close to the saturation level for the Doddington Hall sample), the relationship is linear and the age increases from 418±26 to 452±30 years, corresponding to approximately 3.5 years increase in age per % increment in average moisture content. Although prolonged and significant departures from steady-state conditions in the past would be required to affect the average value this issue must be considered when selecting locations for sampling given the potentially important implications for the interpretation of dates.

**Results of the test programme**

The consistently good agreement between the luminescence and assigned date ranges obtained within the time span investigated of about 350 years (table 2 & fig. 2) is promising. For the dating ‘control’ locations of Boston Guildhall, Doddington Hall, Tattershall and Ayscoughfee the mean difference between the central values of luminescence and assigned ages is 5±10 years, (s.d., n= 6), taking each result to provide an independent test of comparison. This suggests that the assumptions made concerning the dose rate in the past were reasonable for this study. Similar agreement elsewhere, is likely to be influenced by the possibility of the selecting sample locations that are currently dry and where there have not been prolonged episodes of elevated moisture levels in the past. The luminescence dates for the pairs of samples taken from the same phase of the same building (Doddington, 317; Tattershall, 318) overlap within the $\sigma_A$ uncertainty limits (the precision), indicating self-consistency of the dating results for each of these buildings; unfortunately the properties of second samples taken at Boston Guildhall and Ayscoughfee Hall were not suitable due to weak luminescence emission. The results (if not indicated, all dates are A.D.) obtained for the three Fydell
House samples 311-2, -4 and -6 (1727±8; ±17, 1709±12; ±20, 1721±10; ±17 respectively) provide a test of current resolving power. The pooled mean date for these samples is 1719 ±6; ±12 and by application of a statistical test it can be shown that the dates are not significantly different\textsuperscript{36}, forming a single group. Hence the range of the pooled mean date (1707-1731) accommodates both of the suggested date ranges for the two phases of construction and subsequent alterations and does not resolve the two suggested phases for this building. In the case of Clarendon House the luminescence date for the later phase sample (315-5; 1730±11; ±18) is consistent with the assigned date range (1717-1737). The testing of a brick from an interior wall (315-4) reveals that it is associated with an earlier phase of construction (?1650-1675) and the difference of 42 years between the dates for 315-4 and 315-5 can be shown to be statistically significant, pointing to the feasibility of resolving differences of half a century in the same building with single samples.

Table 2. Luminescence and assigned dates\textsuperscript{37}

<table>
<thead>
<tr>
<th>Building</th>
<th>Lab. Ref.</th>
<th>Date (\pm \sigma_A; \pm \sigma_B)</th>
<th>Assigned Date Range</th>
</tr>
</thead>
<tbody>
<tr>
<td>St Mary’s Guildhall</td>
<td>310-1</td>
<td>1388 ±16; ±37</td>
<td>1390-1395</td>
</tr>
<tr>
<td>Fydell House</td>
<td>311-2</td>
<td>1727 ±8; ±17</td>
<td>1700-1726</td>
</tr>
<tr>
<td></td>
<td>311-4</td>
<td>1709 ±12; ±20</td>
<td>1700-1726</td>
</tr>
<tr>
<td></td>
<td>311-6</td>
<td>1721 ±10; ±17</td>
<td>1724-1726</td>
</tr>
<tr>
<td>Clarendon House</td>
<td>315-4</td>
<td>1688 ±8; ±18</td>
<td>1667-1690</td>
</tr>
<tr>
<td></td>
<td>315-5</td>
<td>1730 ±11; ±18</td>
<td>1717-1737</td>
</tr>
<tr>
<td>Doddington Hall</td>
<td>317-1a</td>
<td>1586 ±10; ±24</td>
<td>1593-1600</td>
</tr>
<tr>
<td></td>
<td>317-1b</td>
<td>1576 ±14; ±27</td>
<td>1593-1600</td>
</tr>
<tr>
<td>Tattershall Castle</td>
<td>318-1</td>
<td>1455 ±14; ±33</td>
<td>1445-1450</td>
</tr>
<tr>
<td></td>
<td>318-2</td>
<td>1453 ±15; ±34</td>
<td>1445-1450</td>
</tr>
<tr>
<td>Ayscoughfee Hall</td>
<td>319-1</td>
<td>1447 ±13; ±32</td>
<td>1450-1455</td>
</tr>
</tbody>
</table>

The average of the overall error term (\(\sigma_B\)) for the samples tested in the study corresponds to about ±6% of the luminescence age, equivalent to a range of about ±35 to ±80 years for the period 1700-1300 respectively. There is the opportunity to reduce the size of the range by multiple sampling of a given phase to obtain average dates. Overall this level of performance suggests that application of the method to the dating of late medieval brick buildings is potentially worthwhile where conventional analysis has been unable to provide absolute chronological placement to better than about 50 years. In terms of developing the application of the method to medieval buildings, the level of precision (\(\sigma_A\)) that is routinely achievable with English brick requires further investigation by testing multi-phase buildings. Multiple sampling, while usually limited in fully restored buildings, is likely to be significantly

\textsuperscript{36} I.K. Bailiff, “Methodological developments in the dating of brick…”

\textsuperscript{37} The luminescence ages were calculated from the year of measurement, 2005, as indicated in the sample reference code (i.e., Dur05OSLqi-301-1).
improved in terms of access and the structural detail revealed where buildings are undergoing restoration, which is the most suitable time to undertake work of this type.

Figure 2: Graphical comparison of luminescence date and assigned date for: (1) Boston Guildhall, (2) Tattershall Castle, (3) Ayscoughfee Hall, (4) Doddington Hall, (5) Fyddell House (pooled date) and (6) Clarendon House. Concordance of the two dating systems is indicated by the dotted line. The error bars correspond to the overall uncertainty associated with the luminescence date, $\sigma_B$. (Data from Bailiff, 2007)

Conclusion

On the basis of the outcome of the Lincolnshire study, the OSL technique applied to quartz extracted from brick is capable of producing dates that are in good agreement with independent dating evidence. For six samples taken from a group of four ‘control’ buildings the mean difference between the central values of luminescence and assigned ages was $5\pm10$ years (standard deviation, $n=6$). Application to medieval English buildings is now being extended (late medieval by T. Gurling; Late Saxon and Carolingian by S. Blain, including Normandy and jointly with Univ. Bordeaux III). The methodology used is appropriate for application to other standing buildings in other temporal and geographic regions, and might be used with confidence where conventional dating methods are less certain. Although luminescence cannot provide the degree of chronological resolution comparable to the best provided by conventional buildings analysis combined with tree-ring analysis, there are many instances where the dating of brickwork in vernacular buildings is uncertain due to a lack of diagnostic
features or suitable timber structural elements for tree-ring analysis. By
providing a means of directly dating of bricks as artefacts, luminescence has
a role that is complementary to conventional buildings analysis and
introduces a new direction of investigation in the study of the re-use of brick
in structures and the related issue of lost buildings in the archaeological
record.