The Aim of Laboratory Analyses of Ceramics in Archaeology

April 7–9 1995 in Lund Sweden

In Honour of Birgitta Hulthén ass. prof. emer.

Editors: Anders Lindahl and Ole Stilborg

Konferenser 34

Kungl. Vitterhets
Historie och Antikvitets Akademien

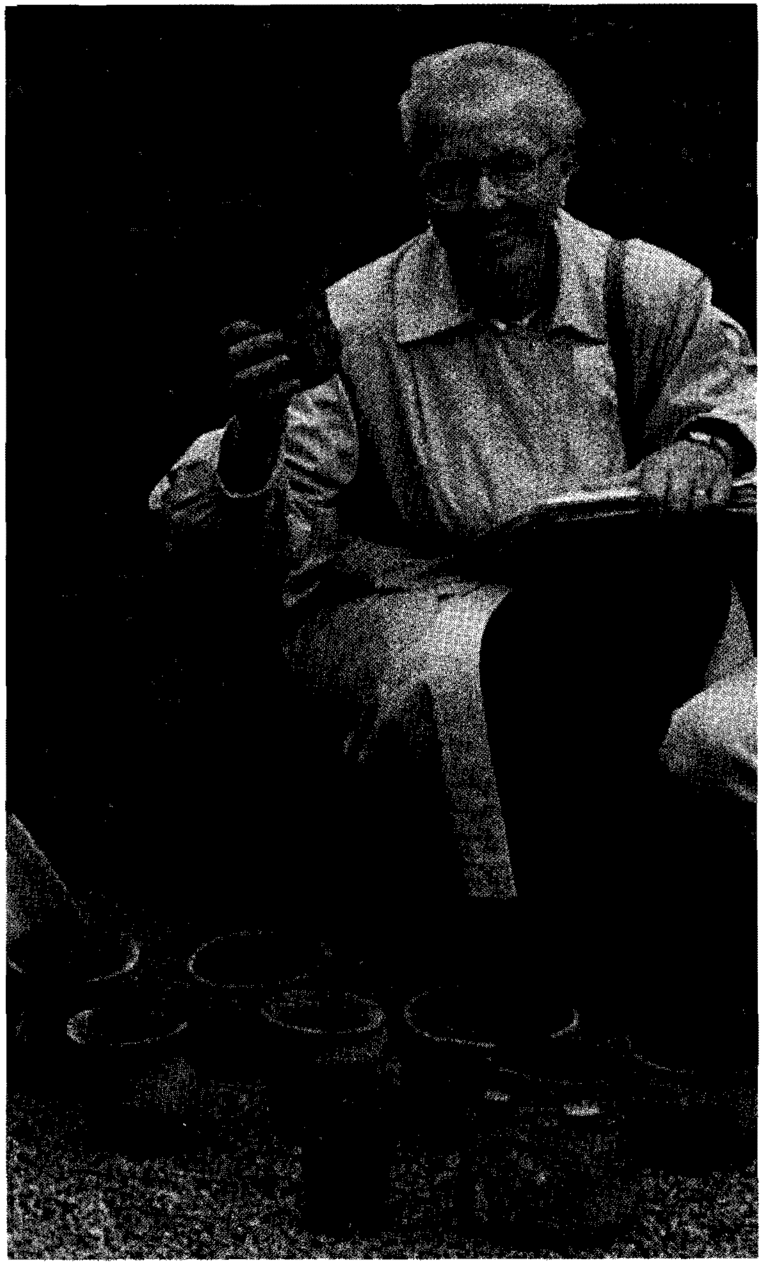
Abstract

The Aim of Laboratory Analyses of Ceramics in Archaeology. Kungl. Vitterhets Historie och Antikvitets Akademien. *Konferenser 34.* Stockholm 1995. 172 pp. ISBN 91-7402-258-X.

This volume contains the papers read and discussed at the workshop "The Aim of Laboratory Analyses of Ceramics in Archaeology". The workshop was sponsored by the Royal Academy of Letters History and Antiquities and the Phillip Sørensen Foundation. It was held at Odengården in Northern Scania in April 1995. The theme of the workshop is of interest to archaeology, archaeometry, ethnoarchaeology and in particular to ceramology. The workshop stressed the need for broad studies of ceramics in both archaeological and ethnografic contexts in order to reconstruct the relations between raw materials, handicraft, use of the products and the social setting. As an answer to the need of closer collaboration between ceramologists SAC "The Society for Archaeological Ceramology" was formed.

Kungl. Vitterhets Historie och Antikvitets Akademien, Box 5622, S-114 86 Stockholm, Sweden

ISBN 91-7402-258-X
ISSN 0348-1433
Distributed by Almqvist & Wiksell International, Stockholm, Sweden
Typesetting: Editorial Office, Uppsala University
Printed in Sweden by Gotab, Stockholm, 1995
English revised by David Gaimster



Birgitta explaining the prehistoric pottery craft to visitors at the Helsingborg Museum 1993.

(Courtesy Helsingborg Museum. Photo: A. Åsgrim-Berlin)

Table of contents

cal maps, field surveys, geological premises to determine ancient clay extraction areas and many others. As a result of such studies the suitability of different clay materials for pottery production could be defined (Fig. 4).

Another question relates to the methods of comparative analysis of clay raw materials and pottery from excavations. The main question is to what extent we are able, through examination of pottery sherds and local raw materials, to identify clays which were used by the ancient potters. Which factors (natural, cultural) are responsible for regional and interregional differentiation of pottery fabrics?

It is a good moment, I think, to propose complex interdisciplinary studies on an international level concerning clays for ancient pottery production in direct relation to local geology, and fabric recipes (influenced by geological but also cultural factors) defined through examination of pottery sherds. Two important results of such studies could be defined: the first one—the creation of a data bank of different raw materials and possible fabric recipes. Clay analyses are also a part of the wide stream of archaeological studies on the natural resources of man, methods of their exploitation, and their significance (cultural, economical, symbolic) in the everyday life of local societies. A good knowledge of clay materials could be regarded as a key to understand many unknown aspects of ancient pottery making.

References

Buko, A. 1981 *Wcześnosredniowieczna ceramika sandomierska*, Wrocław.
Buko, A. 1984 Problems and research prospects in the determination of the provenance of pottery. *World Archaeology* **15,3**. 348–65
Buko, A. 1990 *Ceramika wczesnopolska*. Wprowadzenie do badań, Wrocław.
Czechowski, K. 1918 *Garncarstwo. Wyroby zwyczajne i ozdobne z glin pospolitych*, Warszawa.
Fijałkowska, E. and Fijałkowski, J. 1982 Problem przydatności surowców iilastych rejonu Sandomierza dla ceramiki garncarskiej, *Rocznik Muzeum Narodowego w Kielcach*, **12**. 339–365.
Nicklin K. 1979 The location of pottery manufacture, *Man*, **14**. 436–458.

KVHAA *Konferenser* 34:37–42. Stockholm 1995

Firing Temperature Determinations—How and Why?

By *Mike S. Tite*

Research Laboratory for Archaeology and the History of Art, Oxford University,
6 Keble Road, Oxford OXI 3QJ, England

Introduction

Of all the aspects of pottery technology which have been the subject of scientific investigation, it is almost certainly for firing temperature determinations that the widest range of different scientific methods has been used (Heimann and Franklin 1979, pp. 23; Rice 1987, pp. 426). This perhaps reflects the fact that much of this research has been done by physical scientists who are happiest with quantitative data and who are therefore attracted by firing temperature determinations that are the one aspect of pottery technology to generate actual numbers.

However, the main emphasis of this research has tended to be on the development of new techniques at the expense of the archaeological utilisation of the results. Therefore, as recently highlighted by Gosselain (1992, pp. 243), insufficient thought has been given to the question of the archaeological significance of firing temperature estimates and in particular what they tell us about the actual firing procedures (i.e. open firing or kiln firing) used in the production of pottery.

Methods for determining firing temperatures

The methods used to determine firing temperatures all involve establishing a relationship between the firing temperature and changes in either the mineralogy or the microstructure of the pottery.

The mineralogical changes dependent on firing temperature include the breakdown of the clay minerals, the decomposition of calcite and the formation of high temperature phases such as spinel, gehlenite, wollastonite and mullite. These changes can be followed either by direct measurement using, for example, x-ray diffraction, infrared spectroscopy and differential thermal analysis or by consideration of those properties which are dependent on mineralogy such as colour or magnetic parameters (e.g. saturation magnetisation, coercive force).

The microstructural changes dependent on firing temperature are associated with the progressive sintering and vitrification of the clay matrix of the pottery.

The changes can again be observed directly by examination in section in a scanning electron microscope (SEM) or indirectly through changes in those properties which are dependent on microstructure such as porosity, thermal expansion or shrinkage and hardness.

Since the changes in mineralogy and microstructure with firing temperature depend on the composition of the clay as well as on the firing atmosphere and firing time, a direct estimate of firing temperature from such data is not possible. Instead, one of two comparative approaches are used. The first approach involves refiring samples of the pottery to progressively higher temperatures and determining that temperature at which a change in mineralogy or microstructure occurs. Alternatively, if a sample of the clay from which the pottery was made is available, then a sequence of test pieces fired at a range of temperatures can be prepared and compared with the pottery. With both approaches, the most appropriate refiring/firing atmosphere (i.e. oxidising or reducing) is chosen on the basis of the observed colour of the pottery. Similarly a refiring/firing time ranging from a few minutes to about one hour is chosen depending on what is thought to be most appropriate for the pottery under consideration.

As discussed below, there are very large differences between the maximum temperatures reached in different parts of both open and kiln firings, even in a single firing. Therefore, high precision determinations of firing temperature are of less importance than achieving a rapid throughput of samples so that overall range of firing temperatures can be estimated.

Using a mineralogical temperature scale, x-ray diffraction provides the most comprehensive data on the mineralogy (Maggetti 1982, pp. 121) but the measurements are comparatively time-consuming. Colour measurements in which a large number of sherds can be compared with, for example, a sequence of test pieces is therefore often the preferred method (Hulthén 1976, pp. 1). However, the problem associated with this approach is that colour is critically dependent on whether the firing atmosphere was oxidising or reducing. Shepard therefore regarded the method as unsuitable for grey and black coloured pottery and also questioned its reliability for brown and tan coloured wares (Shepard 1956, p. 223). As a method, it is particularly appropriate in the case of pottery made from iron-rich calcareous clays which pass through a sequence of colours from red to buff to olive in the temperature range 850–1100°C (Matson 1971, pp. 65).

The estimation of the extent of interconnection and vitrification observed within the clay matrix of pottery when examined in section in a SEM provides a effective microstructural temperature scale (Maniatis and Tite 1981, pp. 59). Again such measurements are comparatively time-consuming. With hardness measurements using the simple scratch hardness test (measured in Mohs), a much higher throughput of samples would be possible. However, the results from the scratch test appear to be very dependent on the texture of the pottery and the relationship between firing temperature and scratch hardness therefore does not appear to be very pronounced (Shepard 1956, pp. 113; Matson 1971, p. 70). Heimann and Franklin suggested that a modified Brinell indentation hardness test might provide a more reliable estimate of firing temperature but, as far as I am aware, this approach has never been fully investigated (Heimann and Franklin 1979, pp. 30).

Interpretation of firing temperatures

The two basic procedures that are normally employed for firing pottery are an open firing with no permanent structure (e.g. bonfire or pit firing) and firing in an updraught kiln. Extensive data on the actual temperatures measured during experimental and ethno-archaeological studies of open firings and kiln firings have recently been brought together by Gosselain (Gosselain 1992, pp. 243).

To summarise, open firings are characterised by very fast heating rates and very short times at maximum temperature, typically 20–30 minutes to reach and a minute or less at the maximum temperature. The maximum temperatures reached span the range from 500–900°C with a high proportion in the range 600–800°C. Furthermore, temperature variations of several hundred degrees are observed within a single firing and sometimes even within a single vessel. The firing atmosphere in an open firing can change rapidly from reducing to oxidising. However, the pottery is only very rarely fully oxidised because it is in intimate contact with smoky and sooty fuel and there is insufficient firing time for organic material within the clay to be burnt out. Because of the very fast heating rates only coarse-textured pottery can be open fired, otherwise steam resulting from loss of absorbed water (a proportion survives drying) and chemically combined water cannot escape and the vessel will crack.

Conversely kiln firings, as a result of the much greater thermal mass and the separation of the fuel from the pottery, are characterised by much slower heating rates and much longer times at maximum temperature, typically several hours to reach and 30–60 minutes at maximum temperature. The maximum temperatures reached span the range from 600–1000°C with a high proportion in range 750–950°C. The temperature variations within a single kiln firing tend to be less than in an open firing but are still at least 100°C. The firing atmosphere in a kiln can be controlled to be either oxidising or reducing. This control, together with the longer firing times and separation of fuel and pottery, means that organic material within the clay is burnt out and fully oxidised pottery can be produced. Alternatively, if the kiln is sealed after the maximum temperature has been reached, reduced pottery is produced. Because of the much slower heating rates, both fine-textured and coarse-textured pottery can be readily fired in a kiln.

On the basis of the above data, it is apparent that the maximum temperature reached during a firing does not provide a criterion for distinguishing between an open firing and a kiln firing. However, rather than determining the maximum temperature reached, the mineralogical and microstructural changes used to estimate firing temperatures provide instead a measure of the overall heat input during the firing, that is, some combination of firing temperature-plus-firing time. Kinetic studies of the temperature-time effect on mineralogy and microstructure indicate that, to a first approximation, increasing the time at maximum temperature by a factor of five is more-or-less equivalent to increasing the maximum temperature by approximately 30°C (Norton and Hodgdon 1931, pp. 177; Maniatis 1976, pp. 56). Therefore, instead of trying to determine the actual maximum temperature reached during a firing, it is more appropriate to define an "equivalent firing temperature" which is that temperature maintained for one hour which would produce the observed mineralogy or microstructure.

The equivalent firing temperatures for open firings would then be some 40–80°C lower than the observed maximum temperature which typically would have been reached in less than 30 minutes and maintained for only a few minutes. The equivalent firing temperatures for a high proportion of open firings would therefore be in the range 550–750°C which is less than the 750–950°C equivalent firing temperature range typically observed for kiln firings, for which the maximum temperatures are reached after several hours and maintained for up to an hour.

Consequently, equivalent firing temperatures, when taken together with information on the firing atmosphere (as inferred from the colour of the pottery) and on the texture of the pottery, can be used to differentiate between open firings and kiln firings, which is perhaps the primary aim of firing temperature determinations. Identification of firing procedure helps to establish the level of technological investment and sophistication employed in the firing of the group of pottery under investigation. This information taken together with comparable data for the other stages (procurement and processing of raw materials, forming and decoration of pottery vessels) involved in the production of pottery should enable one to infer the "mode of production". Using the categories defined by Peacock, one might expect open firing to be associated with household production and household industry and kiln firing with individual workshop, nucleated workshop and manufacture(Peacock 1981, pp. 187).

A second area of interest in the context of firing temperature determinations is the consideration of the effectiveness of the technology in producing pottery with the physical properties required for use. The physical properties of the pottery depend on both the composition of the original clay and the firing temperature, time and atmosphere. Thus one is interested in establishing whether, for example, the observed strength or toughness was achieved through the combination of a low refractory clay and a low firing temperature or a high refractory clay and a high firing temperature. In this context, the use of microstructural data to estimate firing temperature has the advantage that these data, at the same time, can also provide information on the physical properties of the pottery under consideration.

Finally, a knowledge of firing temperature becomes particularly important when one considers the production of high fired ceramics such as stoneware and porcelain and also when one compares pottery production with other pyrotechnologies such as metal and glass production. Firing temperatures up to about 1000°C are fairly easily attained, particularly in a kiln. However, to achieve firing temperatures above about 1100–1150°C, better thermal insulation of the kiln and/or higher throughput of hot gases are necessary. These higher temperatures were required for copper smelting and were most probably achieved by application of forced draught. It is therefore interesting to note that, during second half of 5th millennium BC in Mesopotamia when copper smelting begun, a significant proportion of the contemporary Ubaid ware was being fired at temperatures of about 1150°C (Tite and Maniatis 1975, pp. 122). It, of course, remains open to discussion whether the high temperatures achieved in pottery firing provided the technical knowledge for copper smelting or whether the high smelting temperatures were imitated by the potters.

Conclusions

The above discussion has, I hope, established the relevance of firing temperature estimates in the study of the technology of pottery production. However, firing temperature estimates are only occasionally of interest by themselves and should normally be seen as just one aspect in fully integrated studies of the production technology of particular groups of pottery. Such studies should also involve investigating the procurement and processing of the clay and the forming and decoration of the pottery vessels. Furthermore, in a fully holistic approach, the production technology should be considered in the context of the trade or exchange of the pottery and the use to which it is put.

A further question that needs to be considered is to what extent should effort be devoted to developing new techniques for estimating firing temperatures. In my view, the requirement for a reliable method providing a rapid throughput of samples has still not been fully realised and therefore, further investigation of the Brinell indentation hardness test might be worthwhile. Also, the majority of the methods available tend to be more appropriate for estimating firing temperatures above 750–800°C. It therefore might be worthwhile trying to develop methods, perhaps based on infrared spectroscopy, for estimating the firing temperature of low fired pottery.

References

Gosselain, O.P. 1992, The bonfire of enquiries—pottery firing temperature in archaeology: what for?, *Journal of Archaeological Science*, **19**, 243–259.

Heimann, R. and Franklin, U.M. 1979, Archaeo-thermometry: the assessment of firing temperatures of ancient ceramics, *Journal of the International Institute of Conservation-Canadian Group*, **4.2**, 23–45.

Hulthén, B. 1976, On thermal colour test, *Norwegian Archaeological Review*, **9**, 1–6.

Maggetti, M. 1982, Phase analysis and its significance for technology and origin , *Archaeological Ceramics*, Olin, J.S. and Franklin, A.D. (eds.). Smithsonian Institution Press, 121–133. Washington, DC.

Maniatis, Y. 1976, *Examination of Ancient Pottery Using the Scanning Electron Microscope*, Unpublished PhD thesis, University of Essex, Colchester, 56–66.

Maniatis, Y. and Tite, M.S. 1981, Technological examination of Neolithic-Bronze Age pottery from central and southeast Europe and from the Near East, *Journal of Archaeological Science*, **8**, 59–76.

Matson, F.R. 1971, A study of temperatures used in firing ancient Mesopotamian pottery, *Science and Archaeology*, Brill, R.H. (ed.). MIT Press, 65–79. Cambridge, Mass.

Norton, F.H. and Hodgdon, F B, 1931, The influence of time on the maturing temperature of whiteware bodies I, *Journal of the American Ceramics Society*, **14**, 177–191.

Peacock, D.P.S. 1981, Archaeology, ethnology and ceramic production, *Production and Distribution: a Ceramic Viewpoint*, Howard, H. and Morris, E.L. (eds.). British Archaeological Reports International Series **120**, 187–194. Oxford.

Rice, P.M. 1987, *Pottery Analysis—a Sourcebook*, University of Chicago Press, 426–435. Chicago.

Shepard, A.O. 1956, *Ceramics for the Archaeologist*, Carnegie Institution Publication 609, 113–117. Washington. D C.

Tite, M.S. and Maniatis, Y. 1975, Examination of ancient pottery using thescanning electron microscope, *Nature*, **257**, 122–123.

KVHAA *Konferenser* 34:43–48. Stockholm 1995

Assessment of Pottery Function Using Remanent Magnetism Analysis

By *Yastami Nishida*

Nara University, Faculty of Letters, Nara, Japan

Principles and methods

Remanent magnetism analysis is one of the well-known dating methods in archaeology. In Japan this method has mostly been used for the dating of pottery kilns or hearths of pit dwellings. The principle of the method is to measure the geomagnetic orientation of magnetic minerals (mostly titanomagnetite) contained in the specimen. These minerals lose their original orientation when heated over Curie point, and obtain a new orientation while cooling down according to the magnetic field of the location on earth where it is taking place. If detailed chronological data on inclination and declination changes of the geomagnetic field are established, dating of the specimen is possible.

Though almost the same phenomenon occurs at the time of firing pottery or at the time of heating cooking pots over fire, pottery fragments have scarcely been used as specimens of remanent magnetism analysis. The reason is that easily movable objects are not suitable for dating, as this requires the exact record of the location of the specimen at the time of its magnetisation.

Considering these limitations, the author at first thought of using fragments of pottery bases and acquiring data on inclination differences. Differences in inclinations might indicate multiple locations of firing place or time differences. While testing out some experimental analysis, a new possible use for this method arose. The analysis routine is composed of repetitions of demagnetisation and measurement of remanent magnetism. Demagnetisation is usually done to reduce secondary magnetisation which the specimen acquired during the time it lay buried in the ground. Either thermal demagnetisation or alternating field demagnetisation is used. During analysis, we noticed that some specimens showed strong secondary magnetisation while others did not. As to archaeological specimens such as pottery unearthed from one site, different chemical magnetisation during burial is not likely to happen. So, strong secondary magnetisation would mean that the pottery had been heated after the primary magnetisation, that is, pottery firing. In fact, Barbetti had already mentioned the possibility of this method in 1976, and Dr. H. Sakai (Toyama University, Faculty of Science) also discovered the importance of this method