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Seeing the forest for the trees: Assessing technological variability in ancient metallurgical crucible assemblages

Frederik W. Rademakers [a,*], Thilo Rehren [b]

[a] UCL Institute of Archaeology, 31-34 Gordon Square, WC1H 0PY London, United Kingdom
[b] UCL Qatar, PO Box 25256, 2nd Floor, Georgetown Building, a partner of Hamad bin Khalifa University, Doha, Qatar

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ABSTRACT

Metallurgical crucible remains have been found in many archaeological contexts and in varying degrees of preservation. The reconstruction of metallurgical activity through the study of these remains, by means of microscopy and chemical analysis, is undertaken with the aims of understanding technological choices of ancient craftspeople, their use of different raw materials and, by extension, the organisation of production and trade. When large assemblages are available for study, an intra-site comparison of technology and material use within different contexts and throughout time offers interesting perspectives.
Complete crucible examples are rarely found and it is often difficult to reconstruct full crucible profiles based on the fragmented remains. This in turn means that process variability within a single crucible can be hard to assess. Crucible slag is often highly heterogeneous, even within single fragments, enticing analysts to lose themselves in details. Furthermore, the abundance of remains is highly variable, depending on the scale of activity as well as archaeological recovery and preservation, while technological variation within an assemblage can only be detected through study of multiple samples.
Drawing on the analysis of two crucible assemblages, some difficulties and opportunities for technological reconstructions are discussed. Issues related to crucible heterogeneity and inherent process variability are illustrated and a number of interpretative problems arising therefrom are examined. Following a deconstruction of these interpretative issues, some suggestions are made for how, despite methodological difficulties, archaeologically relevant results are obtained where one tries to see the forest for the trees.
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1. Introduction

This paper highlights some issues regarding the study of crucibles used for high-temperature metallurgical operations (Rehren, 2003), illustrated by results from two assemblages studied as part of the first author's PhD research (published in full elsewhere). Here, particular focus is given to open, internally heated crucibles which interact (strongly) with the crucible charge and represent the predominant pre-Roman crucible technology. Crucible slag is defined here as the combination of vitrified ceramic and various contributions from the crucible charge, such as fuel ash and metal oxides, developed at the interface of the crucible and its charge.

This contrasts with – typically later – externally heated examples, often made of more refractory ceramic (Bayley and Rehren, 2007), such as Roman brass-making (Bayley, 1984), medieval European fire assay (Martinón-Torres et al., 2006, 2008) and early Islamic Central Asian steel-making crucibles (Rehren and Papachristou, 2003). In such crucibles, a glaze may form on the exterior and dross is sometimes preserved on the interior, but interior crucible slag is rarely developed. Though this paper focuses in particular on internally heated crucibles, much of the following discussion has bearing on all crucible types.

Crucible studies can be largely subdivided into two categories: investigations of ceramic technology and of metallurgical processes. The former tend to focus on the ceramic fabric, analysing the raw clay and use of temper as well as the crucible design to assess thermal and mechanical properties of the vessels. Thin-section petrography is often used here (*e.g.* Evely et al., 2012). The latter are aimed at understanding the metallurgical crucible process, and technological choices made therein, as well as uncovering variability in the use of raw materials. Here, a stronger focus is given to the analysis of crucible slag, using mounted sections for reflected light microscopy and SEM–EDS analysis (*e.g.* Rehren and Kraus, 1999). This metallurgical perspective is adopted here.

Less studied issues are how crucible heterogeneity affects the sampling strategy for a single crucible or an entire crucible assemblage, how it affects the analytical methodology applied to the study of those samples, and what the interpretative issues arising from this heterogeneity and sampling strategies are.

* Corresponding author.
E-mail addresses: frederik.rademakers@gmail.com (F.W. Rademakers), th.rehren@ucl.ac.uk (Th. Rehren).

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While such problems are particularly in need of investigation for crucible studies, similar considerations must be made for the examination of metallurgical slag more broadly (*e.g.* Bourgarit, 2007 and Humphris et al., 2009). This paper aims to complement these studies and stimulate further research on heterogeneity, sampling bias and representativeness in archaeometallurgy.

2. Materials and methods

2.1. Materials

We present material from two case studies to illustrate the variability that can occur both within single crucibles and throughout entire crucible assemblages. The first assemblage is from Qantir–Pi-Ramesse (Egypt, 13th century BC), where crucibles were used for the casting of bronze objects (Pusch, 1990; Rehren et al., 1998; Rademakers et al., 2015). The second crucible assemblage is from Gordion (Turkey, 6th–4th century BC), where bronze and leaded bronzes were alloyed. The archaeological background for these assemblages is omitted here, as the focus of this paper is strictly methodological. Contextual information for the Pi-Ramesse assemblage is presented by Pusch (1990), while Rademakers et al. (in preparation) will discuss the specific context of the Gordion crucibles (see Kealhofer, 2005 for a broader overview of the Gordion excavations), and their technological interpretation.

2.2. Sampling

Following macroscopic investigation, samples were cut from these crucibles to obtain flat profile sections, mounted in epoxy resin blocks and left to harden. The mounted sections were then ground using increasingly finer abrasives and polished down to 0.25 μm using diamond paste.

2.3. Analysis

The mounted samples were analysed using reflected light microscopy (Leica DM4500 P LED polarisation microscope) and, after carbon coating to ensure surface conductivity, by SEM (JEOL 8600 Superprobe) for structural and textural characterisation of both crucible ceramic and slag. SEM–EDS analysis (Oxford Instruments EDS attachment and INCA software) was performed to obtain quantitative chemical compositions of particular phases (point-microanalysis) and larger areas. Bulk chemical composition was determined by averaging the analysis of five frames (magnification: 100×) for crucible ceramic and crucible slag respectively (similar to Freestone and Tite, 1986). This procedure was performed for 95 samples (49 from Pi-Ramesse, 46 from Gordion) discussed here.

For the presentation of bulk chemistry, ternary diagrams of SiO_2–Al_2O_3–FeO and SiO_2–Al_2O_3–CaO for crucible ceramic and slag composition were constructed (in each case ignoring all other elements). Full compositional data for both assemblages (Rademakers, 2015) will be published elsewhere.

Handheld portable XRF (pXRF) has been used to qualitatively analyse the cleaned surfaces of Pi-Ramesse crucible fragments on site in Egypt (Innov-X Systems, DP 4000). Three 15 second analyses were performed for each fragment using a 40 kV beam, and the data averaged. The raw intensity spectra (in counts/s) have been used to assess the presence of particular elements by hand (checking for characteristic $K\alpha_1$- and $K\beta_1$-intensities), without converting to concentrations (Dungworth, 2000a). pXRF analysis could not be performed for the full Gordion assemblage, due to accessibility constraints.

3. Results

3.1. Within-crucible variability

The first type of variability that can be identified within crucible assemblages is the variation that occurs within crucibles themselves. The analysis of a single crucible sample does not necessarily capture this variability, and some of the differences seen between samples from different crucibles can often be attributed to variability of the same process. This section sets out the main factors influencing this within-crucible variability and its characteristics.

Several process parameters can vary strongly during metallurgical crucible processes. The most important are redox conditions, temperature and the distribution of charge constituents. The first two are strongly related to changing oxygen supply within the crucible, which in turn is controlled by tuyère placement, continuity in bellowing action and charcoal cover. This oxygen supply is a dynamic factor, producing hotter and cooler regions within a crucible, and more oxidising or reducing conditions in different areas. These zones change through time as crucibles often go through several stages in their use, such as prefiring, charging, melting/smelting, casting, cooling and reuse.

The possible reuse of crucibles is not treated in depth in this paper, but obviously introduces important interpretative issues. The likelihood of reuse must be assessed for each crucible assemblage through careful examination of all fragments, and its possible effects on the final interpretation must be discussed.

Most of the metallurgical process information is contained in the crucible slag forming through the melting of the inner surface of the crucible. The degree of this melting is a function of operating temperature and the composition and refractoriness of the ceramic. The ceramic properties are highly homogeneous for the assemblages discussed here (Rademakers et al., 2015, in preparation). More heterogeneous ceramic fabrics, common for early crucibles, may induce more heterogeneous melting behaviour. However, most crucibles are highly heterogeneous with regard to vitrification and slag formation despite compositional homogeneity of their fabric. This changing rate of ceramic disintegration stems mainly from variability in process parameters throughout the crucible. The degree to which the ceramic vitrifies and melts in turn influences the amounts of charge constituents that can be encapsulated by the vitrified ceramic, such as charcoal/fuel ash, ore fragments, metal prills and metal oxides, transforming it into crucible slag. None of these constituents are necessarily present in every particular area of the crucible, even if they occur in one area.

In oxidising areas of the crucible, some of the metal in the charge can oxidise. In the case of copper, contaminants such as iron, cobalt, nickel and arsenic or alloying elements such as tin, lead and zinc are burnt off before the copper itself oxidises (Ellingham, 1944; see Dungworth, 2000a and Kearns et al., 2010). If this happens in an area with a sufficiently developed liquid slag layer, the metal oxides can be incorporated into that slag layer and provide highly distorted information on the nature of the original metal melted in a crucible. Under more reducing conditions, metal prills can be trapped nearly unaltered in the slag, reflecting the original metal composition more closely. The relative proportions of molten ceramic, fuel ash, metal oxides and metal inclusions in the crucible slag can vary highly from one part of the crucible to the next. When no slag is formed, some of these metal oxides typically gather on top of the molten charge as a dross layer.

As a first example, a crucible fragment from Pi-Ramesse is shown in Fig. 1. This fragment is slagged along its entire profile, and differences can already be noted by visual inspection. Closer to the rim (top) the slag layer is fairly regular and thin and its reddish surface is quite flat. Lower down, however, the slag thickness increases and is more variable, and the dark grey slag exhibits a more irregular surface with visibly corroded copper-based prills.

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