

# Bronze Age crucibles from the Kastro-Palaia settlement, Volos, Greece – a contradiction of form and function?

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*ABSTRACT: We present several crucible fragments from Bronze Age contexts in Volos, Greece, linked to a Late Bronze Age (Mycenaean) palace workshop and its predecessor building. Two of the crucibles have side sockets for manipulation, and all are internally fired. Chemical analyses of metallurgical remains from within the crucibles indicate the range of alloy compositions for which they were used, including arsenical copper and tin bronze. Lead isotope ratio analyses point to multiple sources of copper, as would be expected over the extended time period that these fragments represent.*

## Introduction

The Bronze Age site of Iolkos, said to have been on the Pagasitikos Gulf in east-central Greece, is famous as the mythical starting point for Jason's metallurgical reconnaissance to the Black Sea and on to Georgia, searching for the Golden Fleece (Lordkipanidze 2001). It is therefore no surprise that over the last century excavations have revealed evidence for several Bronze Age elite settlements in and around what is today the modern city of Volos in central Greece (Fig 1). Some of these have been linked by their excavators to ancient Iolkos, particularly those including sophisticated architectural remains, tholos tombs and high-status material finds. Less conspicuous are remains of metallurgical activity from that period, even though bronze working seems to have been part of the Bronze Age 'elite package' which included privileged access to exotic materials such as tin metal and the specialist knowledge required to melt and cast metal. This paper focuses on a small assemblage of finds that illustrate the material evidence while also demonstrating the less tangible knowledge that together indicate such elite metallurgical activity.



Figure 1: Aerial view of the Bay of Volos, central Greece, with main Bronze Age sites marked as black dots.

Excavations in 1956–1961 at Kastro-Palaia, a multi-period tell site in the centre of Volos, resulted in it being identified by its excavator with ancient Iolkos (Theocharis, 1961; 1962; 1964; 1965). The excavations

have produced fragments of at least seven different Bronze Age crucibles with remains of copper alloys in them, and at least one stone mould for the casting of circular pendants. Here we present the crucibles and explore what their shape and the nature of the metallurgical remains in them can tell us about their function. The excavator, D Theocharis (1964, 48, 52), interpreted the latest Bronze Age architectural phases of some buildings as a metallurgical workshop; most of the crucible fragments were found in this area. However, one of the crucibles derives from the earliest layer of the buildings dated to the Early Bronze Age II phase (in absolute chronological terms around 2500 BC), a fact which shows the long history of metallurgical activities and knowledge of related procedures at a site that seems to be inhabited continuously from the Early Bronze Age (EBA) onwards. Additionally, the excavator revealed a two-storey Mycenaean building of considerable dimensions in the vicinity of the ‘workshop’, and contemporary with it, which he thought was destroyed by fire around 1200 BC (at the end of the Mycenaean period; in relative chronological terms, at the end of the

Late Bronze Age IIIB period). Theocharis interpreted this important building as a Mycenaean ‘palace’ and tentatively identified the settlement with mythical Iolkos. Whether the site of Kastro-Palaia should be identified with Jason’s Iolkos is a matter for future research and outside the scope of this paper.

Despite the widespread use of metals during the Bronze Age, not many Bronze Age crucibles are published from Greece or the surrounding regions. With this paper we hope to address this lacuna by presenting the finds and their information potential. For the latter, we consider several aspects of the crucibles, including their basic functional and typological design as indicators of chronology or specific technological traditions, and the slag and dross within the crucible fragments as indicators of their use, and as a proxy for the alloys processed in them. Initial analyses showed the presence of copper-arsenic as well as copper-tin in the crucible fragments; this diversity motivated us to investigate their archaeological context and the metallurgical waste within them in more detail.

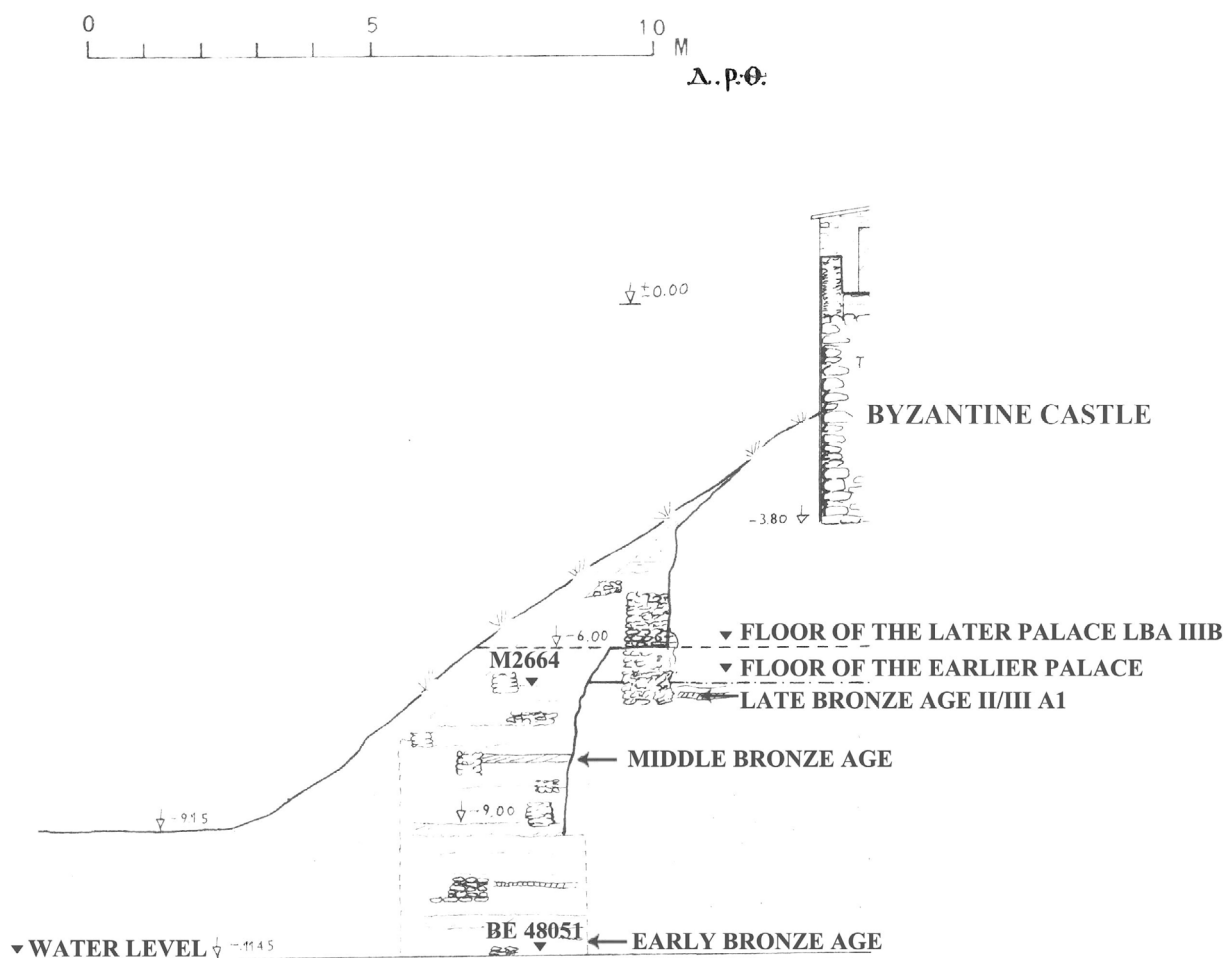


Figure 2: Stratigraphic sketch of the excavation site, adapted from Theocharis’ notebook.

## Archaeological context

The excavations from 1956 to 1961 at Kastro-Palaia unearthed archaeological remains from the EBA more-or-less continuously up to the Byzantine period (Fig 2). For a number of reasons Theocharis was unable to complete his excavations, which led to the establishment, in 2009, of a new five-year project by the then 13th Ephorate of Prehistoric and Classical Antiquities in Volos to complete this work as far as possible (Skafida *et al* 2012). Among other things, this included an initial cataloguing of 1,240 bags of potsherds from the original excavations, a critical assessment of the excavation notes, and an attempt to establish find contexts. Among the potsherds further crucible fragments were found, which are considered here with the two identified by Theocharis, as well as two fragments of Linear B tablets which undoubtedly identify the site as a Mycenaean administrative centre (*ibid*).

The two biggest crucible fragments had already been identified by Theocharis, and can be located in the stratigraphic sketches he produced. Sample BE 48051 (Figs 3 and 8) is from the lowermost section and EBA in date, while M 2664 (Figs 4 and 9–10) is from a Late Bronze Age (LBA) context. Other, less well-preserved fragments (BE 48050 a-b (Fig 12), 48054, 49640, 49638, 49639) were only recognised during the recent project; these can now be linked to the same LBA context as the large fragment M 2664. Associated with these are a stone mould (M 2666; Fig 5), a nearly-complete stone vessel of unknown function (M 2665, possibly a grinder or mortar), and a piece of iron smithing slag (BE 48055). The current assessment of this context suggests that it is a metallurgical workshop floor spanning the LBA to Geometric (9th to 8th centuries BC) periods. Thus, we have a unique EBA crucible fragment, and an assemblage of finds dated to the LBA or beginning of the Early Iron Age.

## The crucibles

In common with other prehistoric crucibles, the finds from Kastro-Palaia are fragments from internally-heated, thick-walled bowls made from ordinary heavily-tempered clay. Apart from the two large fragments, their shape is difficult to reconstruct in detail but is broadly shallow and open-mouthed. This fundamental design is common for prehistoric smelting and melting crucibles, and driven mostly by technical requirements detailed elsewhere (Bayley and Rehren 2007; Thornton *et al* 2010). Significantly, it did not change from the late Neolithic (Ryndina *et al* 1999; Rehren 2009) to the LBA

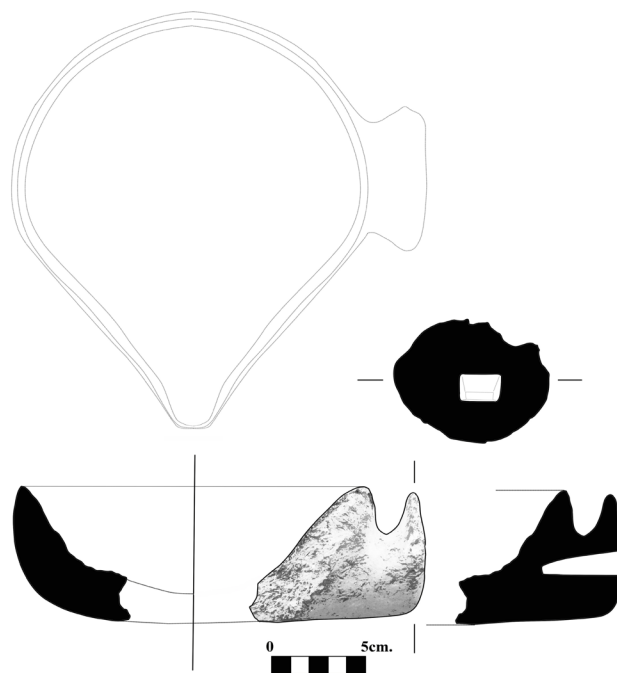


Figure 3: Crucible fragment BE 48051 with tentative reconstruction of its original shape.

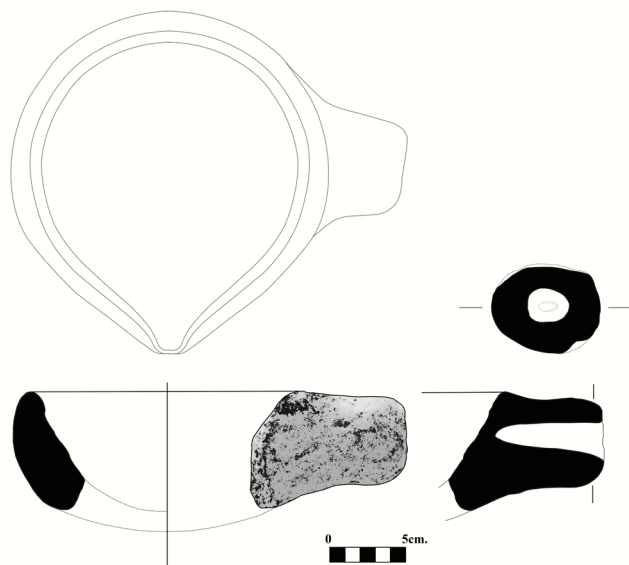


Figure 4: Crucible fragment M 2664 with tentative reconstruction of its original shape.

(Rehren 1997) and into classical Antiquity (Schneider and Zimmer 1986); as such their design does not provide more specific information regarding the date or function of these crucibles. Within this basic technical design, however, there are stylistic or typological variants, for instance how the crucibles were held or manipulated. These seem to reflect geographical and chronological differences driven by cultural traditions and preferences. The two biggest crucible fragments excavated at Kastro-Palaia (M 2664 and BE 48051) are flat-bottomed, shallow round-oval with a protrusion on

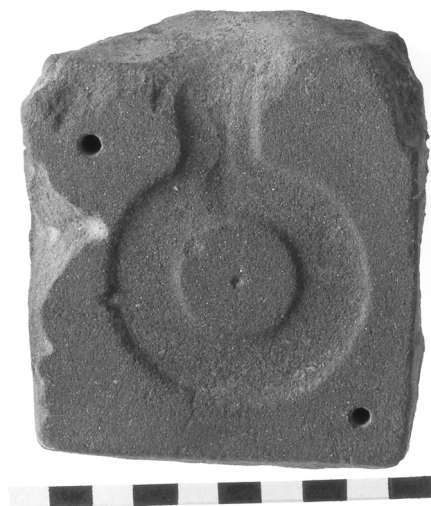


Figure 5: One half of a bi-part stone mould (find number M2666). Scale in cm.

one side containing a deep rectangular socket to receive a (presumably wooden) handle. The inner surfaces of the crucibles have multiple layers of green-stained slag, indicating their repeated use to melt a copper-based alloy. Their inner diameters are estimated to be around 150mm and their depth from bottom to rim is around 60-70mm.

Initial analysis of a thick green stain in the crucible slag in BE 48051 using in situ X-ray fluorescence (XRF) analysis with a spot size of around 1mm diameter revealed copper and arsenic as the main base metal components; this is consistent with the stratigraphic EBA date of this crucible. However, elsewhere in the same crucible XRF showed the presence of copper, tin, lead, nickel and arsenic as well as a little antimony. The other large crucible fragment (M 2664), with four successive layers of crucible slag, has only copper and tin in all layers, with very low levels of lead in some of them, consistent with its LBA stratigraphic date. Similarly, the other, smaller crucible fragments and the mould from the same site mainly show only copper and tin, with only one showing copper and arsenic.

The initial results therefore indicated that there is a certain contradiction between the form and function of two of these crucibles. M 2664, with a socketed handle typical of EBA crucibles is LBA in date and has been used for processing tin bronze, a typical LBA alloy, as have most of the other fragments from that context. In contrast, BE 48051 has an EBA date and form, but contains traces of tin bronze as well as the more typical early arsenical copper. Detailed descriptions of the main crucible fragments and individual results of SEM-EDS analyses on polished samples of crucible fabrics, slag layers and metal prills are given in the Appendix.

## Interpretation

### Fabric

The variability seen in the various crucible fragments is remarkable. The orange fabric of crucible BE 48050 is very rich in mineral temper, both quartz and feldspar, while sample BE 49640 has only organic temper in a pale buff fabric. BE 48051 and M 2664 both have a red-firing fabric with coarse mineral inclusions and organic temper, while BE 48054 has a very fine red-fired fabric. BE 48050a and BE 48050b are similar in their wall thickness and macroscopic appearance of the fabric; however, they have not only different stratigraphic positions, but also very different fabric compositions. All other fragments are visually from distinct individual crucibles, suggesting that there are at least seven crucibles represented in this assemblage.

The composition of the crucibles was determined by SEM-EDS, analysing separately areas of unchanged ceramic (near the outer surface of the crucibles), the bloated part where the ceramic begins to fully disintegrate under the heat during melting the metal, and the innermost slag layer, identified by its elevated base metal content. The ceramic is made from ferruginous clay with variable but generally low lime content (Table 1), and not particularly refractory. We have at present no comparative data for domestic pottery from the site, but visual inspection suggests that the same clay was used for both domestic and technical ceramics. The bloated area has essentially the same composition as the original ceramic, with occasionally increased base metal concentrations; its much higher vitrification, compared to the ceramic, is clearly due to the much higher temperature in this part of the crucible body, and not to a difference in composition or refractoriness of the ceramic. In contrast, the slagged parts do have a considerably different composition. They are rich in base metals (mostly copper and arsenic in BE 48051, and copper and tin in BE 48050b and BE 48054), present partly as metal oxides dissolved in or precipitated from the slag, and partly as metallic prills mechanically trapped in the slag. In addition to this contamination from the metal charge of the crucibles, the slag is also much enriched in lime, magnesia and phosphate. This increase is most marked in the two LBA crucibles and even more apparent when the data is re-calculated to base-metal-free composition (final line for each of the crucibles in Table 1). The concentrations of the main oxides, silica and alumina, are proportionately lower, being effectively diluted by the added lime and magnesia. We interpret this to be due to the incorporation of fuel ash from the charcoal into the slag. Remarkably, there is also a slight increase

in iron oxide and manganese oxide in the slagged layer, indicating that the added material was rich in these oxides, too. Manganese oxide could feasibly come from the fuel ash; many tree ashes are known to contain several percent by weight manganese oxide (Jackson and Smedley 2004). The origin of the iron, particularly at the level of enrichment seen in BE 48054, is less clear, but could possibly be from iron contained in the metal charge and burnt out during the melting stage. Both raw tin and copper can often have significant amounts of metallic iron in them, which would oxidise preferentially during re-melting or fire refining.

### Shape

The two best preserved crucibles both have handles with sockets, even though they were found in different layers. A close parallel to the LBA fragment M 2664 was found a century ago in near-by Sesklo, but is dated to the EBA (Tsountas 1908, fig 288). Other fragments simply indicate a hemispherical bowl-shaped form, although they are too small to exclude the possibility that they may have had handles as well. The best parallel to the socketed crucibles described here are from the EBA, from Tepe Ghabristan in Iran (Thornton 2009, fig 5) and from Fidan and Tell Magass in Jordan (Adams 1999), both with socketed handles extending sideways from the oval crucible form.

Nearer to Volos, but in remarkable design contrast, EBA crucibles from Crete have their bowl set on a stem through which a hole runs to hold the handle (Oberweiler 2005; Poursat and Oberweiler 2011; Evely *et al* 2012), while later crucibles there are hemispherical without handles, but have features described as a 'rocker groove' at the bottom, bridged spouts, or ledge handles opposite the spout (Evely *et al* 2012, 1823–4, fig 3). Evely *et al* (2012, 1824) also mention unpublished crucibles with a socket at the side of Late Minoan IIIB date from Malia in central Crete. Unfortunately, no drawings are provided of these, but they seem to be similar to crucible M 2664 from Volos. Thus, the EBA crucible BE 48051 conforms to the early shapes known elsewhere, while the LBA crucible M 2664, which has a close EBA parallel in Sesklo and a possible further parallel in LBA Crete, is unusual for its date in having a socket.

Thus, crucibles with socketed handles are known primarily from Chalcolithic to EBA contexts where they occur together with simple hemispherical bowls (*eg* Tylecote 1976; Ryndina *et al* 1999). However, they are not well documented from LBA contexts, where simple bowls dominate in Egypt (Pusch 1990; Eccleston 2008), the Levant (*eg* Rothenberg 1990) and Crete (Evely *et*

*al* 2012). This would suggest that both the socketed Kastro-Palaia finds are more likely of an EBA date than LBA, even though one is firmly dated to the LBA based on its stratigraphic context. The other fragments are too small to discuss typologically.

### Alloy used

The metallurgical remains consist predominantly of vitrified ceramic, fluxed by charcoal ash and contaminated by burnt metal from the charge, and a few tiny metal prills trapped in the viscous slag. Thus, the discussion of the alloys processed in these crucibles can only be qualitative, since the relative proportions of the various base metal oxides in the slag differ widely from the original alloy composition (Dungworth 2000, Kearns *et al* 2010).

All crucibles were used for copper alloys, and most analysed fragments yielded evidence for the presence of tin as the main alloying element, except for the small LBA fragment BE 49638, which has a copper arsenic alloy identified by XRF but no tin, and the large EBA fragment BE 48051 (Fig 3 and 8). Near its rim it has a sizeable speck of green corroded metal which only gave copper and arsenic readings. A further analysis in the large area of green corrosion further down in the crucible yielded a tin bronze signature, but with additional arsenic and antimony as well as lead. The SEM-EDS analysis of this slag confirmed this complex signature, and also showed the presence of some nickel (Table 1). This combination of arsenic, tin, antimony and various transition metals is an unusual copper alloy signature; tin bronzes are normally relatively clean, apart from low levels of arsenic which are often found in them. Arsenical copper, on the other hand, does not normally contain much tin or lead. Significantly though, data for EBA copper artefacts from Sesklo show a similarly complex signature (McGeehan-Liritzis and Gale 1988), and artefacts of similar composition have been identified by us among the EBA metal finds from Volos and are awaiting more detailed analysis. It is tempting in this context to mention a group of recently-identified Chalcolithic tin bronzes from the Balkans based on the smelting of copper ore seemingly containing stannite and fahlore (Radivojević *et al* 2013), resulting in a similar complex alloy with tin, arsenic and other base metals.

In contrast, the XRF spectra for all layers in the LBA handled fragment (M 2664) yielded very clean tin bronze spectra, with only a little arsenic in some spectra. The SEM-EDS analyses confirmed this for all of the LBA fragments except BE 49638, with the additional identification of low concentrations of lead oxide in

Table 1: SEM-EDS analyses of Bronze Age crucible fragments from Kastro-Palaia, Volos.

		Na <sub>2</sub> O	MgO	Al <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	P <sub>2</sub> O <sub>5</sub>	K <sub>2</sub> O	CaO	TiO <sub>2</sub>	MnO	FeO	NiO	CuO	ZnO	As <sub>2</sub> O <sub>3</sub>	SnO <sub>2</sub>
BE 48051	ceramic area	1.6	2.6	16.8	58.6	bdl	3.1	8.3	0.8	bdl	6.3	bdl	1.4	bdl	bdl	0.3
	bloated ceramic/slag	1.7	2.5	16.3	56.7	bdl	3.9	8.4	0.7	bdl	5.7	bdl	3.0	bdl	0.6	0.5
	slag area	1.3	2.9	14.3	47.0	0.3	2.5	12.0	0.6	bdl	6.7	0.4	8.1	bdl	3.0	0.5
	slag (metal-free)	1.5	3.3	16.3	53.7	0.3	2.9	13.7	0.7	bdl	7.6					
BE 48050b	ceramic area	1.6	2.4	20.2	59.7	bdl	4.0	1.4	1.0	bdl	8.9	bdl	bdl	bdl	bdl	0.4
	bloated ceramic area	1.7	3.3	22.9	53.3	bdl	4.2	1.2	1.0	bdl	11.7	bdl	bdl	bdl	bdl	0.5
	slag small area	2.0	4.5	14.5	42.1	2.3	2.0	14.8	0.9	1.0	9.5	bdl	1.4	bdl	bdl	4.5
	slag (metal-free)	2.1	4.8	15.5	45.0	2.5	2.1	15.8	1.0	1.1	10.1					
BE 48054	ceramic area	1.1	1.8	18.2	64.0	0.3	3.3	2.7	0.9	bdl	7.0	bdl	bdl	bdl	bdl	bdl
	bloated ceramic area	1.2	2.0	18.3	62.2	bdl	3.5	2.7	1.2	bdl	7.6	bdl	0.8	bdl	bdl	bdl
	dark area of slag	1.6	3.5	8.2	28.0	1.6	1.4	18.0	1.1	1.2	13.9	bdl	8.8	0.3	0.3	12.0
	yellow area of slag	1.6	4.0	8.7	28.8	1.9	1.2	21.2	0.7	1.3	7.7	bdl	6.3	0.4	bdl	16.1
	slag (metal-free)	2.1	4.8	10.9	36.5	2.2	1.7	25.2	1.2	1.6	13.9					
BE 49640	bloated ceramic area	5.0	4.9	14.7	47.3	bdl	4.1	14.5	0.8	bdl	7.2	bdl	0.4	bdl	bdl	0.8
	slag area	4.7	4.4	13.7	42.9	bdl	2.4	22.2	0.5	bdl	5.2	bdl	2.8	bdl	bdl	0.6
	small slag area	5.3	4.1	16.8	49.3	0.2	5.1	11.9	0.9	0.3	5.7	bdl	bdl	bdl	bdl	0.5
	small slag area	5.0	4.7	14.5	48.0	bdl	3.9	14.6	0.9	bdl	6.9	bdl	0.4	bdl	bdl	0.6
	slag (metal-free)	5.1	4.5	15.3	47.6	0.1	3.9	16.6	0.8	0.1	6.0					

Notes: Each line represents the average of several small area analyses. Normalised data in wt%. bdl = below detection limit. Lead was not found in any area above the detection limit of the instrument, thought to be around 0.3wt% for this element. slag (metal-free) = the average composition of the slag area, recast to 100wt% after subtracting Ni, Cu, Zn, As and Sn.

some samples. Of particular interest among the LBA fragments is the presence of uncorroded prills with more than 30wt% tin in two samples (BE 48050b and BE 49640), suggesting that metallic tin and fresh copper were alloyed to produce bronze, as opposed to the mere re-melting of existing bronze which would not give such tin-rich prills. Thus, while the alloy type – tin bronze – is in line with the LBA date of the workshop it does additionally show that the workshop had access to metallic tin, supporting the idea that this was part of an elite or palatial establishment.

Both large crucible fragments show a layered sequence of crucible slag, indicating that they were used repeatedly. Similar multi-use layers have been documented for instance in prehistoric crucibles used for casting in southern Germany (Mecking and Walter 2004), some

with up to eight consecutive layers each separated from the next by a thin layer of ceramic. Re-use of crucibles and application of a thin slip or lining of ceramic is also apparent in some Cretan crucibles (Evely *et al* 2012, fig 16), and indicates the value that these tools held for their owners.

### Metal origin

The find spot of these crucibles in a significant urban settlement with potential connections spanning the entire Mycenaean world raises the question of the origin of the metal. Three slag samples were sent for lead isotope (LI) analysis, with the view of finding possible geological origins for the metal. Two samples were from the large crucible fragments M 2664 (LBA) and BE 48051 (EBA) and a further sample was from BE 49640 of LBA date.

Table 2: Lead isotope abundance ratios in crucible slag from three crucibles from Kastro-Palaia, Volos. Data from Pernicka 2012.

Lab no	Org-ID	$^{208}\text{Pb}/^{206}\text{Pb}$	$^{207}\text{Pb}/^{206}\text{Pb}$	$^{208}\text{Pb}/^{204}\text{Pb}$	$^{207}\text{Pb}/^{204}\text{Pb}$	$^{206}\text{Pb}/^{204}\text{Pb}$
MA-113372	BE 48051	2.0772	0.84180	38.680	15.675	18.621
MA-113373	M 2664	2.0844	0.84688	38.582	15.676	18.510
MA-113374	BE 49640	2.0631	0.83307	38.858	15.691	18.835

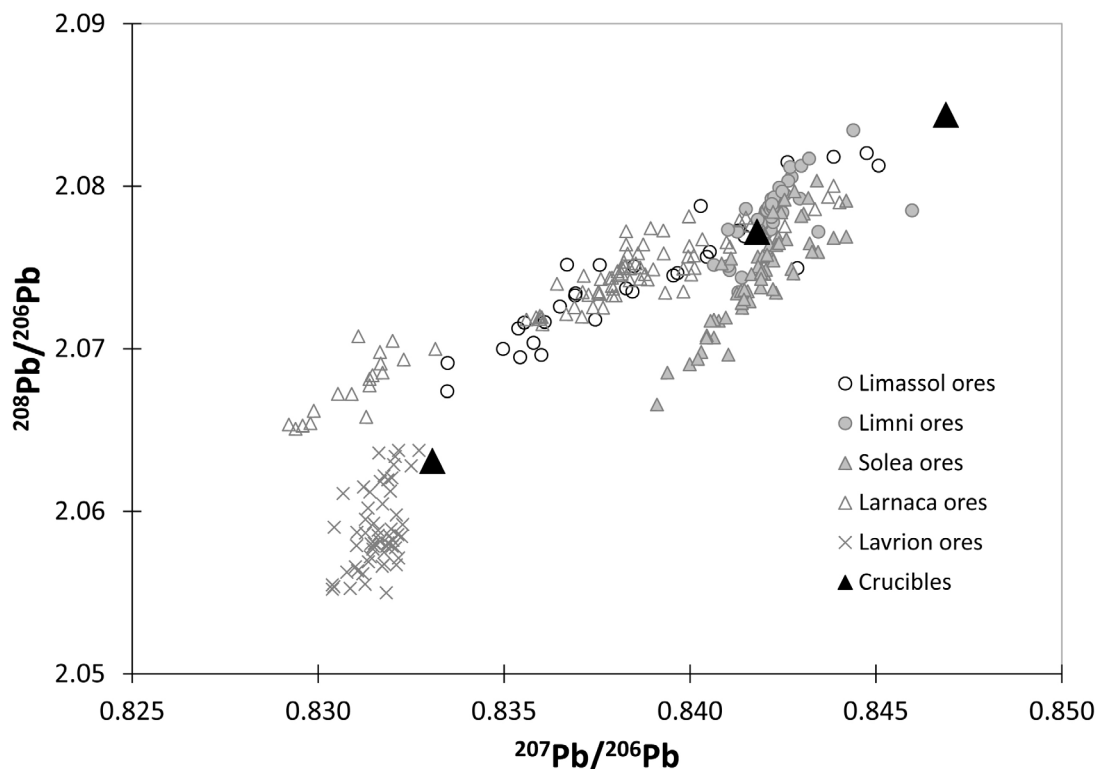


Figure 6: LIA plot of the Volos metal samples (black triangles) compared to LIA data for Cypriot and Laurion metal.

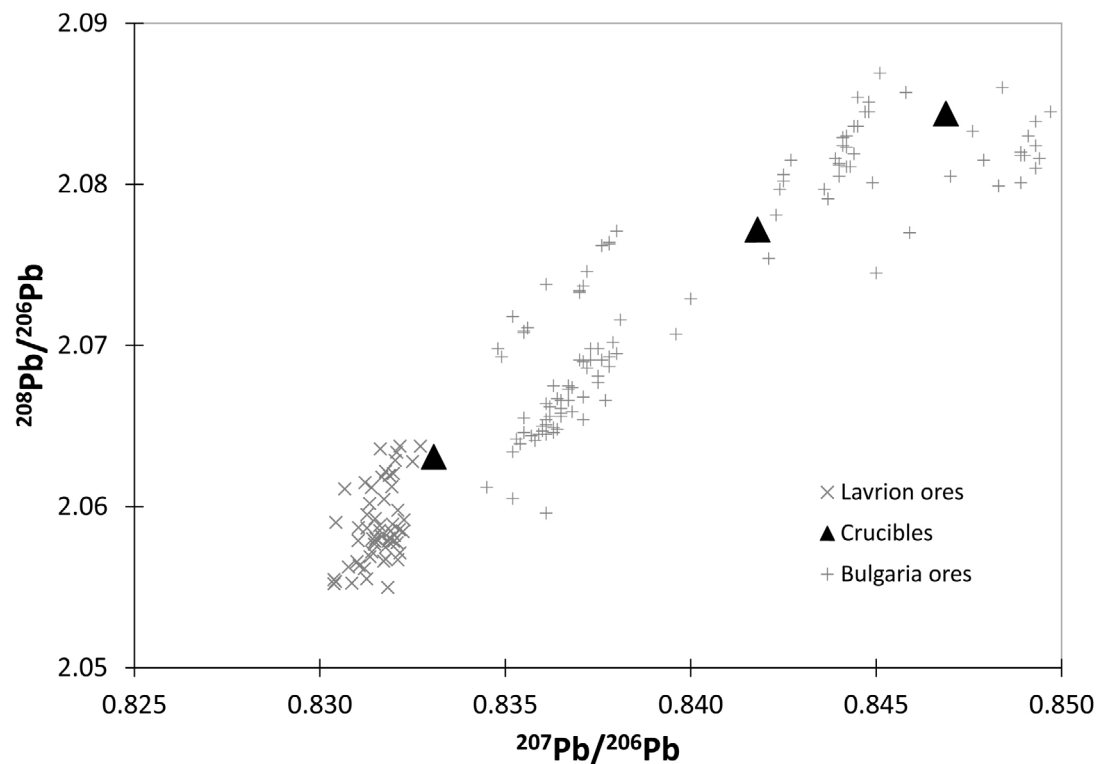


Figure 7: LIA plot of the Volos metal samples (black triangles) compared to LIA data from Laurion and Bulgaria.

The three samples have three very different LI signatures (Pernicka 2012; Table 2), indicating that the metal in them probably comes from different ore deposits. However, when interpreting the data, one also has to bear in mind that the sample from which the lead isotope ratios were determined was not sound metal, but crucible slag. The EDS analyses had shown that the majority of this slag consists of fused ceramic, with a significant further component being fuel ash. Metal oxides from the crucible charge make up only between one third and one half of the total weight of the sample. We therefore have to consider a small but potentially noticeable impact on the overall signature from the lead oxide contained in the ceramic and in the fuel ash. Typical ceramic analyses indicate that it is likely to have 10 to 100ppm lead; values for lead in wood ash are not well published, but probably not much higher. We assume that the bronze itself contained between 0.1 and 1wt% lead, that is about 100 times more than the ceramic or fuel ash. If the burnt metal from the charge contributes between one third and one half of the total weight in the analysed sample, with the balance being fused ceramic and fuel ash, then this would indicate that only around 1 or 2 percent of the total analysed lead is from lead contained in the ceramic plus fuel ash. However, if the ceramic or fuel ash are relatively rich in lead oxide and the metal relatively low in lead, then this contamination can potentially reach several tens of percent of the total lead content. In both cases the potential impact is significantly higher than the analytical error from the analysis itself, estimated to be better than 0.03% (Pernicka pers com), and needs to be considered when interpreting the data. Research is underway to determine the lead isotope signatures of metal prills, crucible ceramic and crucible slag separately in order to quantify this potential effect; initial results indicate that the effect is noticeable, potentially major, but not systematic in its direction (Rademakers pers com). For now, therefore, we have to take the analysed values at face value while bearing in mind that they may be contaminated by lead from the ceramic or fuel ash.

The EBA sample BE 48051 has a very good match with the Cypriot ore deposits of the Limni and the Solea axis, comprising the largest copper deposits there and with substantial evidence for ancient mining (Fig 6). In particular the Solea axis has been linked to the majority of LBA oxhide copper ingots. However, the early date of the crucible makes this attribution uncertain, since no primary copper production is known from Cyprus earlier than the LBA. On the other hand it is intriguing, since evidence for copper working in Cyprus dates back to the Philia phase, broadly contemporary to the EBA, and continues through the MBA, including indications

for smelting sulphidic ores and – as indicated by textual evidence from Mari – the export of copper at this early date (Kassianidou 2008).

Thus, while an attribution of the LI signature in the EBA crucible to the main Cypriot copper source is not entirely unreasonable, it is not necessarily convincing either. Further doubt on a Cypriot origin comes from the complex chemical signature of the alloy worked in this crucible (see above and Appendix). Cypriot copper is typically very low in antimony, tin, lead and nickel (eg Hauptmann *et al* 2002), while such natural alloys are known from near Volos (McGeehan-Liritzis and Gale, 1988) and elsewhere in the Balkans (Radivojević *et al* 2013).

The LBA crucible M 2664 plots at the extreme upper right (geologically old) end of the Cypriot ore fields, with the closest match to ore from the Limni axis; however, the match is not perfect, and other geological origins have to be considered as well. Given the mythical link of Iolkos to Jason's Black Sea travels it may be of interest that all three samples fall near or even within a cluster of Bulgarian ore analyses (Fig 7) even though the match is here again not perfect either. On present evidence, it is therefore difficult to suggest a geological origin for the copper from this crucible based on the lead isotope abundance ratios, in particular since the archaeologically-attested local prehistoric copper smelting remains from near Volos are not yet characterised for their LI ratios.

The closest match for the small fragment BE 49640 is to the ore deposit of Laurion in southern Attika (Fig 6); although this is mostly known as a lead-silver ore deposit with exploitation predominantly in the Classical period, there is circumstantial evidence that it was also a significant copper deposit (Gale *et al* 2009). It has long been noted that numerous Bronze Age copper or bronze artefacts from the Aegean have lead isotope signatures that are consistent with the Laurion deposit, and cannot be matched to any other ore deposit (Pernicka pers com). A Laurion origin for the copper in this crucible, although not proven, is therefore not an unreasonable assumption. It is possible that lead from Laurion was introduced into the alloy, either intentionally or by contamination during recycling, but the very low lead content observed in the crucible makes this unlikely. As with the other samples it is likely that the contamination from ceramic and fuel ash lead has shifted the ratio away from the pure metal signature, possibly explaining the off-set position in respect to the main field of published ore analyses from Laurion.

Some of the ambiguity between different possible geological matches could probably be resolved through trace element analyses of copper metal; however, since our samples are from crucible slag rather than sound metal artefacts this option is not open to us. On balance, the lead isotope evidence is suggesting an Eastern Mediterranean origin (Laurion and Cyprus) for the two LBA samples, while for the EBA sample the origin must remain open at this stage; the unusual combination of copper, tin, arsenic, antimony, zinc, nickel and lead could point to a possible Balkan origin (Radivojević *et al* 2013). A further possibility to be considered is a local/regional origin, not least in view of the local evidence for objects of similar composition from Sesklo (McGeehan-Liritzis and Gale, 1988). In addition, there is field evidence for local prehistoric copper mining (M Vaxevanopoulos pers com) in Pherai, some 20km from Kastro-Palaia, and at Mt Orthrys, some 60km from the site, and for prehistoric copper smelting in the immediate vicinity of Volos (Papadimitriou 1991; Papastamataki *et al* 1994; Tizzoni *et al* 2008). Work is ongoing to characterise this more fully, as the archaeological date of exploitation, lead isotope and trace element signatures of these sources are so far unknown. Only future research will be able to tell whether any of these sources are compatible with the signatures seen in the Kastro-Palaia crucibles.

## Conclusions

The analysis of crucibles combines the study of their design and fabric as an indicator of cultural tradition and ceramic material selection, and of slag and metal content as a proxy of the alloy used in them (Bayley and Rehren 2007). The latter allows only qualitative interpretations due to the shift in element ratios among the alloying components as a result of their different chemical reactivity (Dungworth 2000; Kearns *et al* 2010). Also the LI data has to be seen as a combination of signatures from the metal charge, the crucible ceramic, and the fuel ash. This picture is further complicated through the evident multiple use of crucibles involving different alloys, and the spatial heterogeneity of chemical conditions within a single crucible operation. Thus, the study of these crucible fragments requires a strong element of interpretation and discussion, and still only provides results that are not more than indicative, and highly ambiguous, even when highly-calibrated analytical methods are used.

Despite these acknowledged limitations, a lot can be learned from this study. Here we see two different designs for the side handle of the crucibles, with parallels as far east as Iran. The slag analyses point to an unex-

pected early appearance of a tin-rich complex alloy in the EBA crucible, and the active alloying of fresh tin and copper metal in two other crucibles. The identification of the complex alloy has led to an ongoing follow-on study of the EBA copper-base artefacts from Volos, revealing the presence there of tin-rich complex alloys (Asderaki, unpublished data). It is expected that this will contribute further to the extremely interesting topic of the origin of the metal worked at the workshops excavated at Kastro-Palaia. The three fragments analysed for their lead isotope signature point to three different origins, contributing to the picture of a well-connected and privileged workshop, even if we cannot so far identify the exact origins of the metal. This diversity in raw material access contrasts to the apparent consistency of location of the workshop, which over more than a millennium and a half (from 2800–1200 BC) only moved 40m.

The study here is based on just a few fragments excavated more than half a century ago. Future research in the vicinity of the old excavation, among the archived finds, and in the metal-rich hinterland will hopefully contribute further to our understanding of the cultural connections of the site at Kastro-Palaia, its role as a node within the Bronze Age world, and the development and diversity of metallurgy during prehistory. It is clear already though that even such small finds can provide a wealth of information.

## Appendix: Analytical methods and detailed results

The date of the crucibles was assessed based on unpublished excavation notes from the excavator and a re-examination of associated finds. Non-invasive in-situ analysis by XRF was done using a portable instrument developed at NCSR Demokritos in Athens. The instrument has a measurement spot of *c*1mm diameter, with the positioning of the samples guided by the overlap of two laser beams in front of the tube/detector. Operating conditions were 40kV and 30μA.

Samples for optical and scanning electron microscopy and lead isotope analysis were removed by lifting small pieces of material along existing cracks, or by cutting thin slices to reveal cross sections through the ceramic and slag using a diamond-coated rotary tool. Polished sections were prepared following standard procedures using cold-setting resin and diamond paste. The sections were investigated first by optical microscopy using reflected light, then carbon-coated and analysed using a Hitachi SEM with attached Oxford Instruments INCA EDS system. Lead isotope ratios were determined

following established procedures at the Curt-Engelhorn-Zentrum Archäometrie in Mannheim, Germany, and the results compared to an in-house data base of archaeological and ore samples from the eastern Mediterranean.

### Notes on individual crucible fragments

**BE 48051A** large fragment with nearly complete handle, showing evidence for repeated use with multiple layers of slag of different composition (Fig 8). The handle is formed to provide an oval protective shield 80mm wide and 70mm high, narrowing to a neck on the upper side and partly to right and left. It is continuous having a flat lower profile and a steeply valleyed side view. Minimum size of neck is 40mm high and 60mm wide. It has a rectangular hole, tapering inwards with maximum inner dimensions of 24 by 14mm, and 45mm deep (Fig 3). The vessel wall thickness is relatively constant around 25mm, with a 5mm thick grey and slagged inner zone and a smooth outer surface with numerous burnt-out organic impressions. In fracture there are further numerous burnt-out organic fragments visible, and only few relatively small mineral inclusions around 1mm, max 2mm. The inner surface is heavily bloated and vitrified with a large patch of green-corroded slag. Bloating and slag formation seem to go over the rim near the handle. In situ XRF analyses of the green spot near the rim (Fig 8) revealed only copper and arsenic, while the green encrustations nearer to the centre of the crucible showed copper, nickel, lead, arsenic, tin and antimony. A sample was taken from a partial cross section near the rim through the crucible slag layer, exposing metal prills in a matrix rich in copper oxide.

Three areas of the cross section were analysed by SEM-EDS: un-altered ceramic fabric, fully vitrified ceramic near the inner surface, and areas of crucible slag. Because of the small size of the samples it was not possible to follow a standard protocol of SEM-EDS area analysis; instead, the size of the analysed areas was adjusted to fit the available sample material. Because of this, and the obvious heterogeneity of the material at the scale of investigation, we consider the data only semi-quantitative; where possible the reported values are the average of several measurements.

The un-altered ceramic is not refractory, lightly calcareous and iron-rich. Small amounts of copper and tin were present in both areas that were analysed, arsenic in only one, while nickel, zinc and lead, all identified by XRF, were always below the detection limits of the SEM-EDS system (Table 1). The compositions of the regions of vitrified and bloated ceramic are very similar to that of the non-vitrified ceramic, but with higher copper and tin

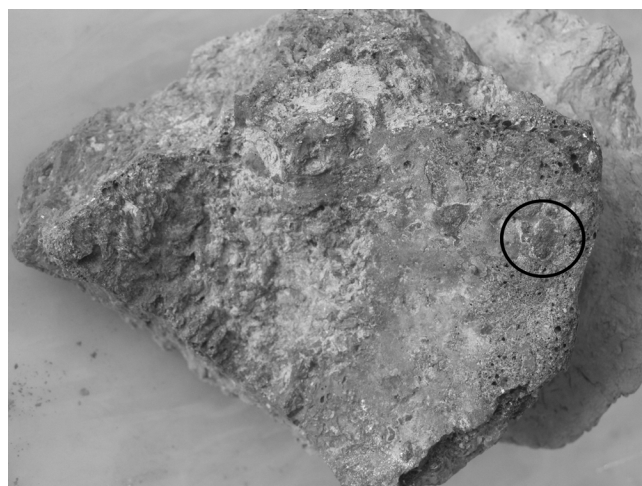


Figure 8: Corroded metal prills in the slagged inner surface of crucible BE 48051. The analysed spot is within the circle on the far right, near the rim of the vessel that runs almost vertically. Image width ~10cm.

contents as well as more regular arsenic contents, and occasional traces of lead and zinc. The areas of crucible slag were significantly higher in copper, arsenic, zinc, tin and lead oxides. Alumina and silica levels were lower than in the ceramic areas, but lime contents were consistently higher. The data indicates that the crucible slag has formed from the underlying ceramic, probably fluxed by calcium oxide from the fuel ash, and has incorporated some metal oxide and metal prills.

**M 2664** A large fragment with complete handle and near-complete profile of the vessel (Figs 9 and 10). Maximum preserved height is 70mm; the wall thickness is variable, c20mm. The handle is c50mm long, oval to round rectangular in section with external dimensions of 70 by 55mm with an oval tapering hole 30 by 23mm max internal dimensions and 70mm deep. The fabric is coarse and mostly red throughout, but grey to black on the entire internal surface to a depth of about 2-5mm into the fabric. Several rock and other temper/inclusions are visible, but not very frequent. Slag covers a bloated layer of ceramic, mostly towards the lower part of the vessel where it is several mm thick in parts, seemingly multi-layered; it is shot through with red and green stains with a smooth inside surface. A series of XRF spot analyses on different slag layers showed copper and tin in all of them, with some lead in the lower layers and possibly a little arsenic in the lowermost layer. A sample was taken from this slag layer where it was separating from the underlying fabric at the bloated zone; the material is very fragile and brittle, showing not much coherence. The largest layered fragment was mounted for OM analysis; several small fragments, thought to be from the same layer, were used for LIA.

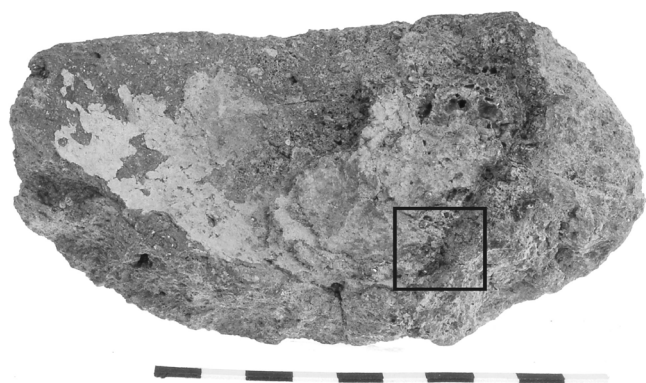


Figure 9: Crucible sherd M 2664 showing the vitrified inner surface with the multi-layered slag sample taken from the area in the box. Scale in cm.



Figure 10: Crucible sherd M 2664 showing the socketed handle set just below the rim. Scale in cm.

BE 48050a A ‘dry’, that is slag-free, rim fragment of a crucible, made of thick red-fired ceramic with a grey zone towards the inside surface and a smooth outer surface (Fig 11). The maximum preserved thickness is 35mm, with 10mm being the grey inner part. The inner surface is rich in coarse mineral inclusions (probably quartz and feldspar) up to 5mm long. The estimated



Fig 11: Crucible sherd BE 48050a (left) shows very little vitrification and no slag cover on its inside; crucible sherd 48050b (right) has some slag layer. Scale in cm.

diameter is 43mm, the maximum rim length 55mm. No sample was taken since there are no visible metallurgical remains on the surface.

BE 48050b A partly vitrified or slagged crucible fragment with a simple rounded rim, made of thick red-fired ceramic with a grey zone towards the inside surface and a smooth outer surface (Fig 11). It was used for alloying tin bronze. The maximum surviving wall thickness is 30mm, with 10mm grey inner part. The inner surface is rich in coarse mineral inclusions (probably quartz and feldspar) up to 4mm long. The maximum preserved dimensions are 40 by 40mm. A crucible slag layer is preserved on about half the surface; it has little visible metal content. A sample was taken as a partial cross section through the upper part, including grey and red ceramic; an additional sample was a fragment of the slag layer that was dislodged during sampling.

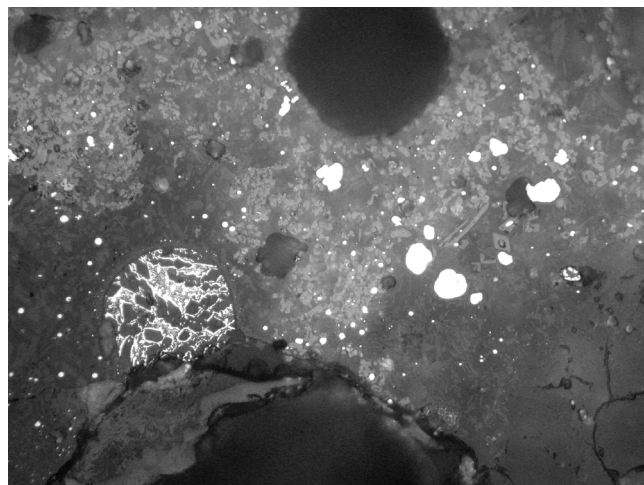


Figure 12: Backscattered SEM image of the crucible slag showing copper-rich droplets (white), a larger two-phase corroded metal droplet (bottom left) and tin oxide crystals (light grey) in the surrounding glassy matrix. Image width c0.4mm.

The sample was large enough to facilitate a more systematic SEM-EDS analysis of the different parts of the cross section. Three area analyses were made of the non-vitrified un-altered ceramic, three area analyses were made in areas where the ceramic had become more bloated and four small area analyses were made in regions where there was slag. Analyses were also made of a few metal prills that were enclosed within the slag-rich area of the sample (Fig 12). The compositions of the two ceramic regions (as fired, and bloated) were fairly similar to each other, with only slight variations in the major components. Compared to the crucible BE 48051, this one has much lower calcium oxide content in both ceramic regions, while the iron oxide content is significantly higher.

Small pockets of slag were found to contain significant levels of lime. Copper oxide contents were variable, and significantly lower than tin oxide. Zinc, arsenic and lead oxides were also variable, but were often not detected and are rather low overall. The analyses undertaken on two of the metallic inclusions within the slag-rich area showed that they were of a very high-tin alloy. Copper particles (Fig 12, centre and top right) contained approximately 75wt% copper and 22wt% tin (as well as 0.4wt% zinc, 1.6wt% arsenic and 0.2wt% lead). The partly corroded, round inclusion (Fig 12, lower left) contained on average 33wt% copper and 37wt% tin, with a high value for oxygen (*c*26wt%). Spot analyses of the darker areas of the inclusion showed them to be much richer in oxygen (*c*33wt%), lower in copper (*c*14wt%) and higher in tin (*c*40wt%), with magnesia, silica, phosphate, lime and iron oxide also present. The lighter areas of the inclusion, in contrast, contained much purer metal, with copper (*c*63wt%) and tin (*c*32wt%) dominating. A second inclusion, larger and more angular was also analysed in the same way, with similar results, although this inclusion had a higher copper content overall (*c*40wt%) and slightly lower tin content (*c*34wt%), the balance being oxygen. These inclusions are thought to consist of delta-epsilon phases of the copper-tin system, with the epsilon phase corroded and hence darker.

The very high tin content relative to copper both in the slag and in the metal prills strongly suggest that the crucible from which this fragments originates was used to alloy tin and copper metal in order to produce bronze. Similar heterogeneous tin-rich copper prills have been found in other LBA crucibles (eg in Qantir, Egypt (Rademakers *et al* in press) or Cham, Switzerland (Rehren 2001)), and it has been argued that such tin-rich prills cannot form during simple melting of existing bronze (Rademakers *et al* in press; Rehren 2001). Due to the preferential oxidation of tin over copper one would expect that any prills from re-melting bronze would have a tin content equal to or lower than the melted alloy; only when combining metallic tin and copper in order to produce bronze can we expect to find prills of a wide range of compositions between pure tin and normal bronze.

*BE 48054* The object is a small body fragment of crucible of thick, red-fired ceramic with a grey zone towards the inside surface; the outer surface is smooth. It was used for bronze re-melting under oxidizing conditions. The maximum preserved thickness is 20mm with a 5mm grey zone. The vessel is made from a relatively fine ceramic with some organic temper and not many mineral inclusions. The maximum preserved dimensions

are 25 by 25mm. The sample taken was a complete cross section plus a loose fragment of crucible slag.

The ceramic parts of the sample ranged from un-vitrified to significantly bloated. Both the vitrified and non-vitrified samples had similar levels of most compounds, but varied slightly in their trace elements. Two areas of slag were analysed that were in close proximity to a large pure copper prill. One area was in a region of slag that appeared dark in reflected light (one small area analysis was taken from this region). The other area was a region that appeared yellow in reflected light (two small area analyses were taken from this region). The slag consists of fused ceramic from the vessel, with significantly higher calcium oxide content (most likely from the fuel ash) and variable amounts of metal oxides. The relatively high content of iron oxide and manganese oxide, the latter not detected in the ceramic itself, are intriguing. Manganese oxide could originate from the fuel ash; hardwood in particular is known to sometimes be rich in manganese oxide (eg Jackson and Smedley 2004). However, the high iron oxide content is surprising, and could indicate that the alloy was relatively rich in iron which then burnt out during re-melting. This would be consistent with the relatively oxidising conditions indicated by the higher tin content compared to copper in the slag, and the tin-free copper prill embedded in this slag (Table 1).

*BE 49640* These four crucible body sherds, two with a grey vitrified bloated zone, and some loose fragments including crucible slag most probably come from the workshop area and are dated to the LBA period. The fragments consist of heavily organic-tempered pale buff ceramic, now very eroded. Some small loose fragments of crucible slag and of body material were selected for analysis. The small sample mounted for SEM-EDS analysis was dominated by heavily bloated slagged ceramic, with a relatively large proportion of copper metal. Four small areas were analysed: three in the slag, and one in an area of bloated ceramic (Table 1). A two-phase metal prill was found to be very rich in tin. It contained approximately 78wt% copper, and 18wt% tin, with traces of zinc (0.1wt%), arsenic (0.3wt%) and lead (0.6wt%).

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