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Front cover. Block 1000.0049 from Naga (photograph courtesy Karla Kroper).
Above. Pottery jar with decoration of sorghum heads from BMC 60, Berber (photograph courtesy Mahmoud Suliman Bashir).

Alloying copper, arsenic and tin – the first crucible evidence from Kerma

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Introduction

During the 2018-2019 season, the Swiss-French-Sudanese mission to Kerma-Doukki Gel undertook a reexamination of the 'bronze casting furnace' located in the western part of the Kerma religious complex (Bonnet 2004), illustrated in Figure 1. After its discovery in the early 1980s (Bonnet 1982, 34-39; 1986), this structure was left partially intact for future work. This strategic decision now enables a complete re-evaluation of a unique metallurgical technology in Nubia. From this perspective, a multidisciplinary team of specialists (archaeologists, archaeometallurgists and paleobotanists) is conducting a research programme focused on copper alloy technologies.

The study of the metallurgical structure and the technological processes carried out during its use in the Middle Kerma period[1] is in progress (Verly *et al.* 2021), and a comprehensive discussion will be published elsewhere (in preparation). Rather than a furnace, it is in fact a huge casting mould (with a substructure of four double heating channels, four lateral casting funnels and a vaulted superstructure), dedicated to the production of large (almost 2m^2) copper alloy plates. This type of structure is hitherto unknown in the ancient world with the exception of the much later 'cross-furnaces' from Pi-Ramesse (Pusch 1990), probably representing an adaptation of the same principle and reflecting a long-term casting tradition in the Nile Valley. Against this background, this paper focuses on a particularly crucial aspect of the casting technology: the crucibles.[2]

Crucible analysis sheds light on the technological choices associated with metallurgical activity in the city of Kerma. It identifies the copper alloys used to cast the large plates, whether these alloys were freshly made or recycled from existing metal stocks, and how this was done. Furthermore, it can provide some insight into the metal sources used to this end, providing complementary perspectives to those obtained from metal object analysis (Rademakers *et al.* 2022). Finally, crucibles can reveal traditions in casting technology through their design in terms of ceramic fabric, typology and operation. This enables an intimate comparison between crucible assemblages found throughout the Nile Valley to investigate technological exchange and cultural interaction from a crafts perspective. This paper focuses on the fabric selection and metallurgical processes evidenced by the crucibles. Another paper, which assesses the crucible typology, manufacture and mode of operation in more detail and compares it to contemporaneous crucibles from the Nile Valley (and particularly Middle Kingdom Ayn Soukhna), is being prepared by the authors. The complete dataset regarding the crucible analysis presented in this paper can be found in an Appendix (see footnote for link).[3]

In addition to the almost complete crucible preserved at the Musée d'Art et d'Histoire (MAH) in Geneva (MAH 27796), the assemblage under consideration includes six fragments uncovered in the abandonment layers of the casting workshop (inventory numbers KV 1011–KV 1016). The other two artefacts analysed were found in contexts outside the religious precinct of the Deffufa. One of them (inventory number KV

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[1] The Middle Kerma period largely overlaps with the Middle Kingdom in Egypt, and dates *c.* 2050 to 1750BC.

[2] Although crucible fragments (MAH 27796) were found deposited in one of the heating channels of the 'mould-furnace' (Bonnet 1986), the actual crucible heating took place in separate heating structures to the east (towards the Deffufa), allowing easy access to the casting funnels (more details to follow in the publication of the full structure).

[3] https://www.sudarchrs.org.uk/wp-content/uploads/2023/11/SARS_FR_SN2023_Kerma_crucibles_appendix.pdf

Rademakers, F. W. *et al.* 2023 [http://doi.org/10.32028/SN27pp2-23].

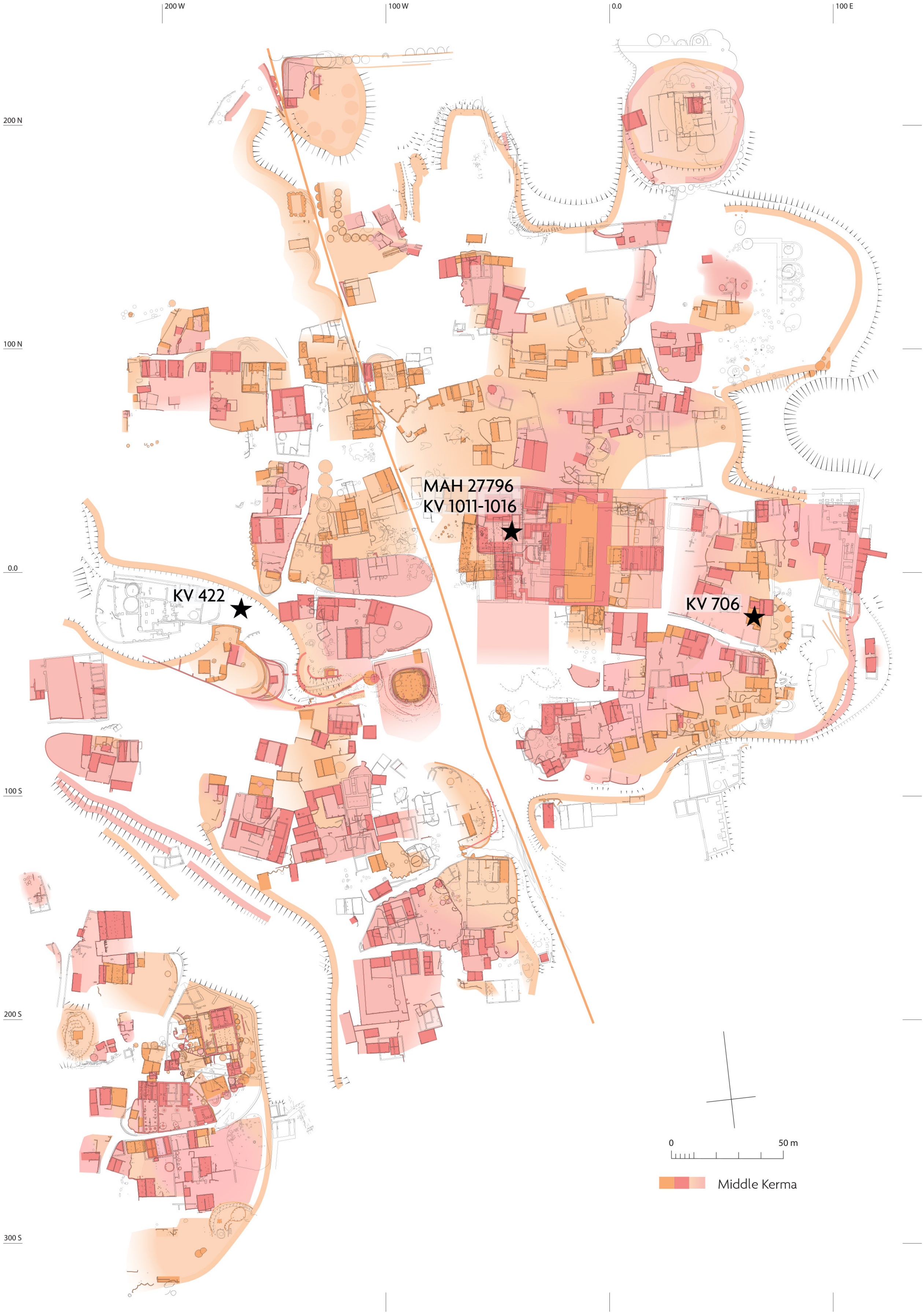

Figure 1. Map showing crucible find locations at Kerma © Mission Kerma-Doukki Gel.

Find number	Context	Date	HH-XRF	Polished block	Thin section
MAH 27796	Casting workshop – 1980 excavation (2018: US051)	Middle Kerma period	x		
KV 1011	Casting workshop – 1980 excavation	Middle Kerma period	x	x	x
KV 1012	Casting workshop – 1980 excavation	Middle Kerma period	x	x	
KV 1013	Casting workshop – 1980 excavation	Middle Kerma period	x	x	
KV 1014	Casting workshop – 1980 excavation	Middle Kerma period	x	x	x
KV 1015	Casting workshop – 1980 excavation	Middle Kerma period	x	x	x
KV 1016	Casting workshop – 2018: US002, P6	Middle Kerma period	x		x
KV422	Circulation levels, north of the western city gate	Kerma period	x	x	x
KV706	House M162	Middle Kerma period	x	x	x

Figure 2. Overview of crucibles analysed in this study.

706) came from the levels of House M162 (Bonnet 2014, 150-151), built to the north of the eastern entrance to the city. Although this fragment was found outside a clearly identified craft context, in a heavily eroded area, it should be noted that the house is contemporary with the large-scale casting activities. The other fragment (inventory number KV 422) was discovered in the circulation levels, north of the western city gate (Bonnet 2014, 168-169). This was a major access point used throughout the occupation of the site and in this context it is particularly difficult to assign the artefact to a specific period with certainty.

Methodology

The crucibles (listed in Figure 2) first underwent visual examination, whereby macroscopic descriptions are made of the crucible shape, fabric and traces of use. This involves photographic documentation, using both 2D (focus stacking) and 3D methods. Photos of the crucibles studied in this paper are shown in Figure 3 (full documentation in the Appendix), while their 3D models will feature in the typology-oriented paper.

During the 2018-2019 field season, and a 2019 visit to the MAH in Geneva, all crucibles were studied using handheld X-ray fluorescence (HH-XRF: Bruker S1 Tracer III-SD, operating at 40 kV and 5 µA, without filter). By comparing spectra obtained for different surface areas of the same crucible (ceramic fabric, vitrified areas, copper corrosion products), a qualitative assessment of its use can be obtained (cf. Rademakers and Rehren 2016). These results (included in the Appendix) are helpful in selecting a suitable location to sample the crucibles for detailed, quantitative micro-analysis (cf. below), but are equally useful

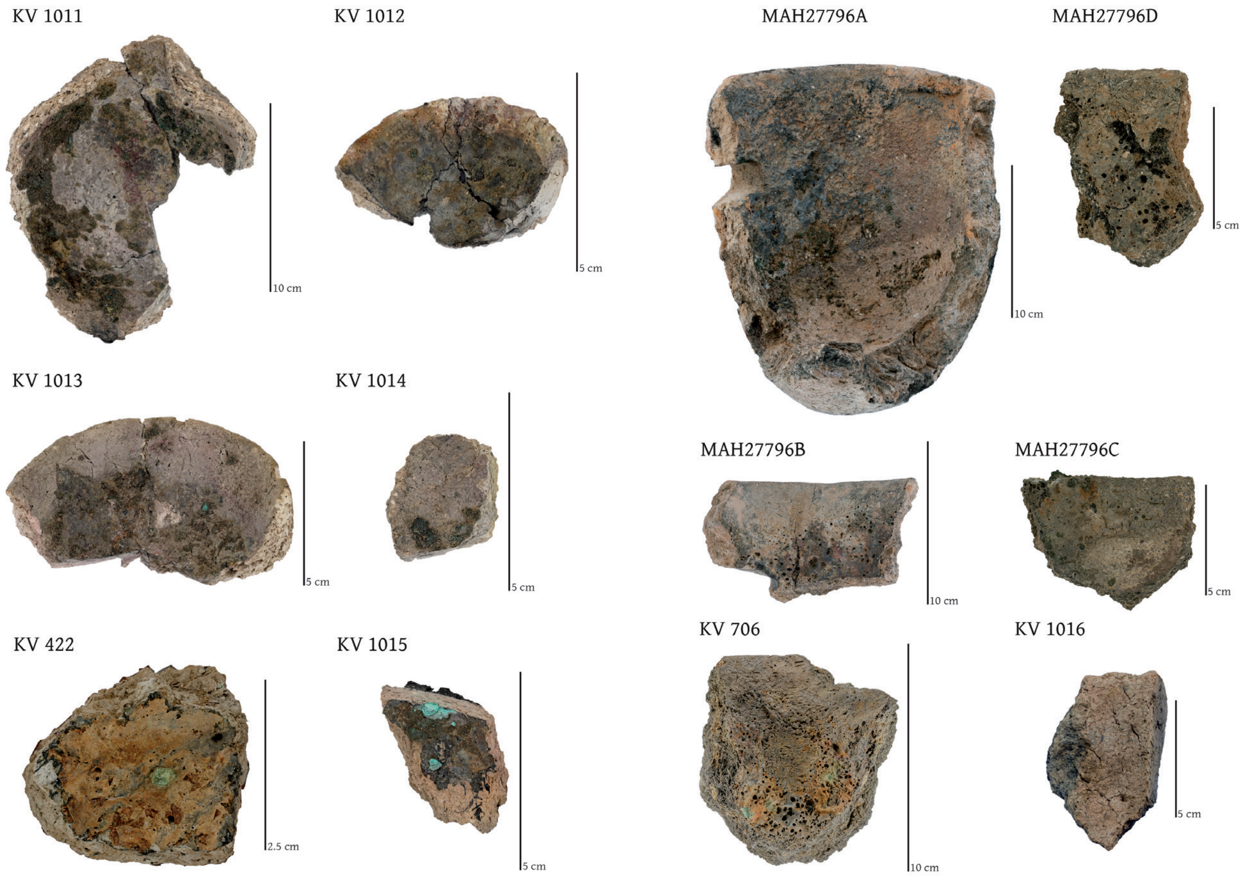

Figure 3. Internal view of crucibles analysed in this study. More detailed photography is included in the Appendix.

in their own right, enabling a qualitative comparison[4] of metallurgical contamination across all areas of the preserved crucible fragment (which cannot all be sampled), supporting their overall interpretation.

In the next step, crucible samples were cut using a rotary saw mounted to a Dremel tool (Bosch®). Care was taken to obtain samples representative of the entire cross-section, which may include one or two ceramic layers, an external vitrification zone, internal 'crucible slag' and/or dross[5]. Furthermore, preference is given to sampling lower body areas where possible, as these tend to provide a better reflection of the reducing crucible process conditions (cf. Rademakers and Rehren 2016).

From these samples, thin sections have been prepared for petrographic analysis (six crucibles) and polished blocks for metallurgical analysis (seven crucibles). Petrographic analysis is an established technique used to characterise ceramic fabrics, by identifying their constituent minerals, porosity, texture and microstructure (e.g., Quinn 2013). These inform on fabric preparation, vessel manufacture and firing

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[4] Some elements tend to be over- or underestimated through surface XRF analysis and relative proportions of alloy components cannot be confidently assessed in this way (cf. Kearns *et al.* 2010; Rademakers and Rehren 2016).

[5] This follows terminology outlined by Rademakers (2015, 50-51) and Rademakers *et al.* (2018b, 1649-1651). 'Crucible slag' is defined as the interaction layer formed through the disintegration and vitrification of the crucible interior (typically accelerated by fuel ash contributions) and its interaction with the crucible charge. It is typically superimposed on a bloated zone, marking the disintegration of the underlying ceramic. The term does not imply any specific metallurgical process, such as smelting, but is used as a generic name to describe this internal crucible zone. Crucible dross, by contrast, consists of various metal oxides which float on top of the crucible charge during the metallurgical process, with little or no (molten) ceramic component. Such dross layers are sometimes referred to as crucible slag in the literature (e.g., Craddock 2013; Tylecote 1986). Here, however, the term crucible slag refers strictly to the slagged ceramic formed at the crucible interior, while dross is a more generic term for what is sometimes called 'refining slag' in the literature (e.g., Craddock and Meeks 1987, Karageorghis and Kassianidou 1999; Merkel 1990; Pernicka 1999). Dross can be found deposited on both crucible exteriors and interiors.

conditions. Application of this technique is relatively rare for crucibles (examples from outside the Nile Valley include Evely *et al.* 2012 and Sahlén 2013), and has been used only recently for pottery in the Nile Valley more generally (e.g., Badreshany *et al.* 2022; Ownby *et al.* 2014; Spataro *et al.* 2015). Thin sections were prepared following standard methods and studied under plane and cross polarised light (PPL and XPL) using a Nikon Eclipse 50i POL Polarising microscope with Deltapix Invenio 5DII camera and a Leica DM750P transmitted light microscope fitted with a Leica Flexacam C3 at the KU Leuven, Department of Earth and Environmental Sciences. An overview of the fabrics is shown in Figure 5, with additional images and full descriptions in the Appendix.

Polished crucible sections were prepared by mounting in epoxy resin and polishing using increasingly fine abrasive paper, finished using a 1/4 µm diamond paste (cf. Rademakers *et al.* 2018b). Following carbon coating, they were analysed at the British Museum Department of Scientific Research using a scanning electron microscope (SEM: Hitachi S-3700N) in high vacuum mode, equipped with an Oxford Instrument energy dispersive X-ray spectroscopy microanalysis system (EDS: INCAx-act Silicon Drift Detector) for compositional analysis (using AZtec version 5.1 software, Oxford Instruments, factory calibration), operating at 20 kV with a working distance of 12 mm and live time of 90 seconds. The detection limit for most elements and oxides is below 0.5 wt% (often 0.1 wt%), while accuracy and precision are usually better than 10 % for concentrations down to 1 wt% (and progressively lower for lower concentrations – cf. analysis of reference materials reported in the Appendix). Bulk chemical composition for different zones (ceramic fabric, external vitrification, crucible slag, dross) was determined after Rademakers *et al.* (2018b), by averaging the analysis of five areas (magnification: ×100, frames *c.* 1.3x0.9 mm – sometimes smaller or fewer when zone size is limited). These results are reported as normalised weight percentages of oxides in the Appendix. Furthermore, point analysis was carried out to determine the composition of various micro-phases (reported as element weight percentages). Comparing bulk composition of the ceramic, vitrified exterior and crucible slag, combined with micro-phase analysis of crucible slag, enables the best possible reconstruction of the crucible charge. Nonetheless, it must be emphasised that metallurgical crucible processes are highly heterogeneous in nature, and their presentation in the crucible sherds can be extremely variable down to the micro-scale. As such, a single sample is usually not entirely representative of the metallurgical process conducted in a crucible. This must always be kept in mind when interpreting analysis results (cf. Rademakers and Rehren 2016).

Results

This section highlights the most relevant data for understanding crucible technologies. While initially intended for inclusion here, the complete overview and interpretation of analytical results is provided in the Appendix, to be used as a companion to the main text. An overview of key metallurgical features for each crucible is provided in Figure 4.

There are a few important factors used to assess metallurgical techniques reflected by each crucible. A crucial consideration for each crucible is the relative enrichment of particular elements between the ceramic (baseline), external vitrification and crucible slag. This follows the methodology outlined by Rademakers *et al.* (2018b), using ratios of metal oxides relative to alumina (see also Rademakers and Rehren 2016). Other significant process indicators are metallic prills embedded in the crucible slag and the metal oxides formed at high temperature in the crucible slag, dross and external vitrification layers. Post-depositional oxidation has led to the selective corrosion of many metal prills (as well as sulphide phases) in the crucible slag, creating a bias with respect to their original composition – these are therefore omitted from consideration for technological reconstructions.

The base of the crucible fabric is probably derived from a clay-silt deposit (possibly Nile alluvium),

Find number	**Copper alloy processed**	**Dimensions**	**Notable features**
MAH 27796 (A-D)	Cu-As-Sn	Diameter: 20-26cm Height: 21cm Charge volume: *c.* 260-450ml	Complete profile Clear 'water line' Developed crucible slag Casting traces Dross deposits
KV 1011	Cu-As-Sn	Diameter: 13.5cm Height: 5.5cm Charge volume: *c.* 150ml	Complete profile Spout Limited crucible slag External applied layer (vitrified) Dross deposits Highly reducing (Fe prills) Cu-As-Sn prills (with Fe) Cu-Fe-S inclusions Associated Fe-Co-Ni-As
KV 1012	Cu-As-Sn	Diameter: 6cm Height: 3cm Charge volume: *c.* 15-25ml	Complete profile Very small Developed crucible slag Internal repair layer (re-use) Cu-As-Sn prills (with Fe and Ni) Abundant Fe- and Ni-rich Cu-As prills As-rich prills: **probably active alloying** Cu-Fe-S inclusions Associated Fe-Co-Ni-As
KV 1013	Cu-As-Sn	Diameter: 10cm Height: 4cm Charge volume: *c.* 60ml	Complete profile High Fe and S in crucible slag Abundant Fe-rich Cu-As-Sn prills Extremely As-rich prills: **evidence for active As alloying** Traces of Ni (and Pb) in alloy Highly reducing conditions
KV 1015	Cu-As-Sn	n.d.	Body fragment External applied layer Developed crucible slag Extremely As-rich prills: **evidence for active As alloying** Cu-Fe-As sulphides (with Se)

Figure 4. Overview of key crucible features.

Find number	Copper alloy processed	Dimensions	Notable features
KV 1014	Cu-As-Sn	n.d.	Body fragment (close to rim?) External applied layer Limited vitrification Corroded dross deposits S-rich dross with Se and Te
KV 1016	Unknown	n.d.	Rim fragment Charred plant remains in fabric No vitrification No internal contamination
KV422	Cu(-As)	n.d.	Body fragment External (glaze) layer with cuprite Internal glaze (as external) Cu-Fe sulphide inclusions Highly reducing conditions
KV706	Cu-As(-Sn) (+Au)	n.d.	Rim fragment Charred plant remains in fabric Highly reducing conditions Au-rich alloy prill Possible recycling of gilded alloy

Figure 4 (cont.). Overview of key crucible features.

including minerals and rock fragments originally formed from the weathering of granite-type rocks. Well-sorted organic temper[6] has resulted in high porosity, improving the refractory properties of the crucibles. Several crucibles have been fitted with an additional external layer of a different clay type, which appears fused or completely vitrified due to high temperature exposure. An overview of the crucible fabrics and their variability is presented in Figure 5, and more exhaustive petrographic descriptions are provided in the Appendix.

In all crucibles, alumina and silica are the main constituents of the ceramic fabric, accounting for 85-96 wt% of all oxides, while iron and calcium oxide are present at *c.* 1-3 and 0.5-2 wt% respectively (excluding KV 706, with 4.2 wt% iron and 3 wt% calcium oxide). Figure 6 provides a reduced overview of the 'bulk' compositional data, re-normalised (on the left) to the silica, alumina and fuel ash components (lime, soda, magnesia and potash) and (on the right) to the silica, alumina and iron oxide components. This illustrates the compositional homogeneity of the crucible fragments studied here. Furthermore, it demonstrates the enrichment of both external vitrification and internal crucible slag zones by fuel ash components (derived from charcoal combustion), conducive to ceramic vitrification. Finally, there is a notable increase in iron oxide in some of the external layers (reflecting the application of a different secondary layer) as well as in some of the crucible slag layers (reflecting either an internal secondary layer or a source of iron within the crucible charge). These observations are interpreted in more detail for each crucible in the Appendix, and discussed further below.

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[6] The short, fine fibres and their even distribution indicate that dung may have been mixed into the fabric during their preparation (in contrast to the often coarse organic temper noted in the Pi-Ramesse crucibles: Aston *et al.* 2007, 518-519), although dung temper remains difficult to identify confidently (e.g., Amicone *et al.* 2021).

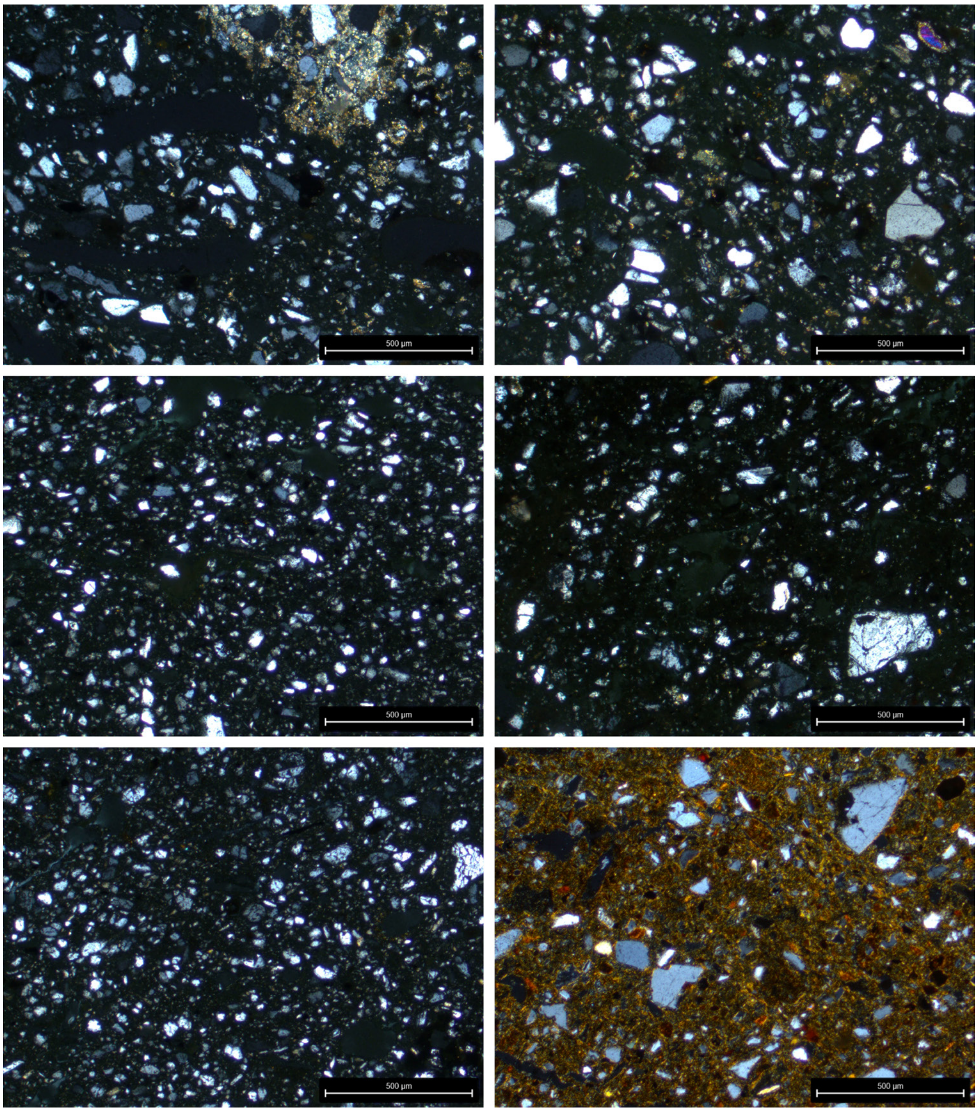

Figure 5. Overview of (core) ceramic fabric of the crucibles in cross-polarised light (XPL). Left to right, top to bottom: KV 1011 and KV 1014, KV 422 and KV 706, KV 1015 and KV 1016.

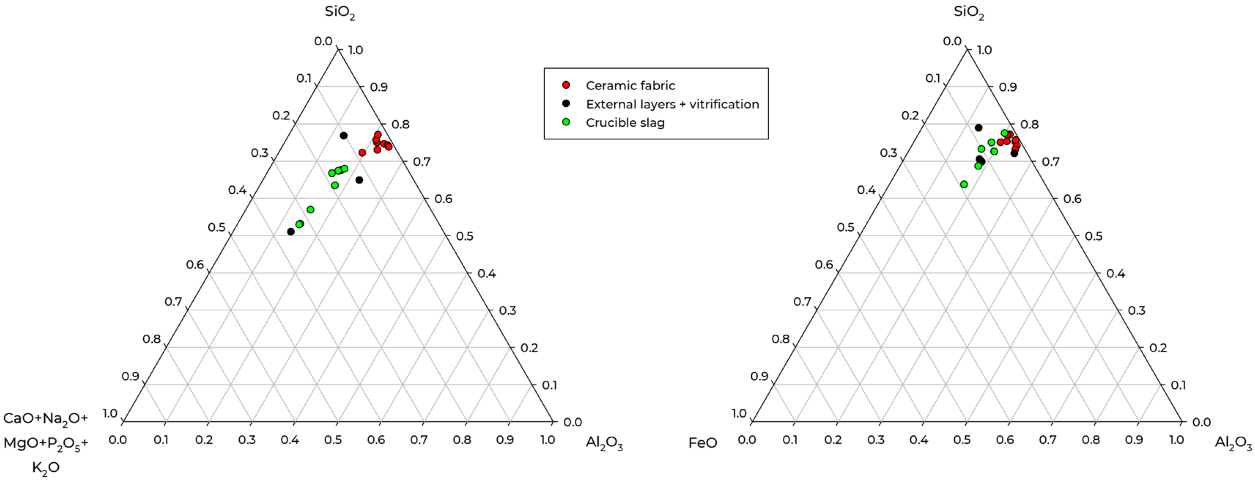

Figure 6. Ternary diagrams comparing the compositions of the ceramic fabrics, external vitrification layers and crucible slag.

Discussion

Metallurgical processes

While these crucibles reveal a somewhat homogeneous fabric selection (discussed below), their varied typology may indicate different metallurgical processes. Yet there is continuity in the main alloy components (copper, tin and arsenic) and a recurrence of similar minor elements (cobalt, nickel and lead) as well as sulphide inclusions throughout the assemblage (e.g., Figure 7), suggesting these processes were at least related. This is expected for an assemblage associated with a most likely short-lived casting event. Given that great heterogeneity can exist within crucibles, one should not expect the same presentation of a metallurgical process in each crucible (fragment). Rather, an attempt should be made to reconcile divergent samples as indications of a single process where possible, before interpreting these as multiple processes (Rademakers and Rehren 2016). Nonetheless, the variations in crucible shapes, sizes and layering may imply different operations were conducted in this context.

High-arsenic prills (illustrated in Figure 8) found in two crucibles (and strongly elevated arsenic in another crucible) prove that an arsenic-rich component was added to the crucible charge. This follows the same reasoning employed for identifying active tin alloying, whereby arsenic (like tin) would be lost preferentially upon remelting rather than concentrated in copper up to such high concentrations (Rademakers *et al.* 2018a and references therein). These (tiny) prills show an intermediate stage of the process frozen in the crucible slag, rather than the intended end product, which would have had the lower arsenic (and iron) content reflected by the majority of prills and casting spills (as well as contemporaneous finished objects).

The first question, then, is whether an arsenic-rich component was alloyed with copper metal, or rather co-smelted as part of a mixed arsenic-copper ore charge. The latter interpretation has been suggested for a Middle Kingdom crucible from Buhen[7] (Davey *et al.* 2021), but the evidence here is quite different. In the Kerma crucibles, no substantial silica enrichment is noted for the crucible slag, while the relative enrichment in sulphur is much higher in most fragments. If a mixed arsenic-copper ore was co-smelted at Kerma, it was not the same as tentatively identified at Buhen. Moreover, the relatively limited slag formation in the Kerma crucibles strongly points to a secondary alloying procedure rather than the smelting of complex ores. Indeed, smelting (at a relatively small scale) is not expected as a practical choice in the context of this urban workshop, as significant alloy volumes were needed for plate casting and the delivery of large volumes of ore to the religious precinct may be considered unlikely. Finally, there is the presence of tin to be considered. As tin is not usually found associated with sulphur[8], it was either added as a separate component (metal or cassiterite), or was part of scrap tin bronze being re-melted (to which arsenic was added, with sulphur, iron and/or copper). In any scenario, an active alloying procedure (with tin and/or arsenic) rather than (co-)smelting is implied.

This leaves the question as to what the crucible charge components might have been. An important clue is the presence of arsenic in the sulphide phase (in KV 1015, together with high-arsenic prills). This points to the introduction of arsenic already bound to sulphur, as any free sulphur in the crucible would preferentially bind to copper rather than arsenic. Arsenic must therefore have been introduced as mineral orpiment (As_2S_3), realgar (AsS), tennantite ($Cu_6[Cu_4(Fe,Zn)_2]As_4S_{13}$) or arsenopyrite (FeAsS), or alternatively as a 'speiss' (FeAs) product (cf. below), and almost completely dissociated from sulphur during crucible

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[7] Alternatively, the iron enrichment of the Buhen crucible slag could be interpreted as a remelting of unrefined copper, while the (relatively limited) silica enrichment might be attributed to fuel ash. In that case, the crucible may equally represent alloy remelting or fresh alloying – a possibility considered by Davey *et al.* (2021, 7).

[8] Tin can be found in stannite (Cu_2FeSnS_4), but this mineral is not attested in the geology of Egypt and Sudan – in contrast to the cassiterite (SnO_2) deposits of the Eastern Desert – or known as an exchange commodity.

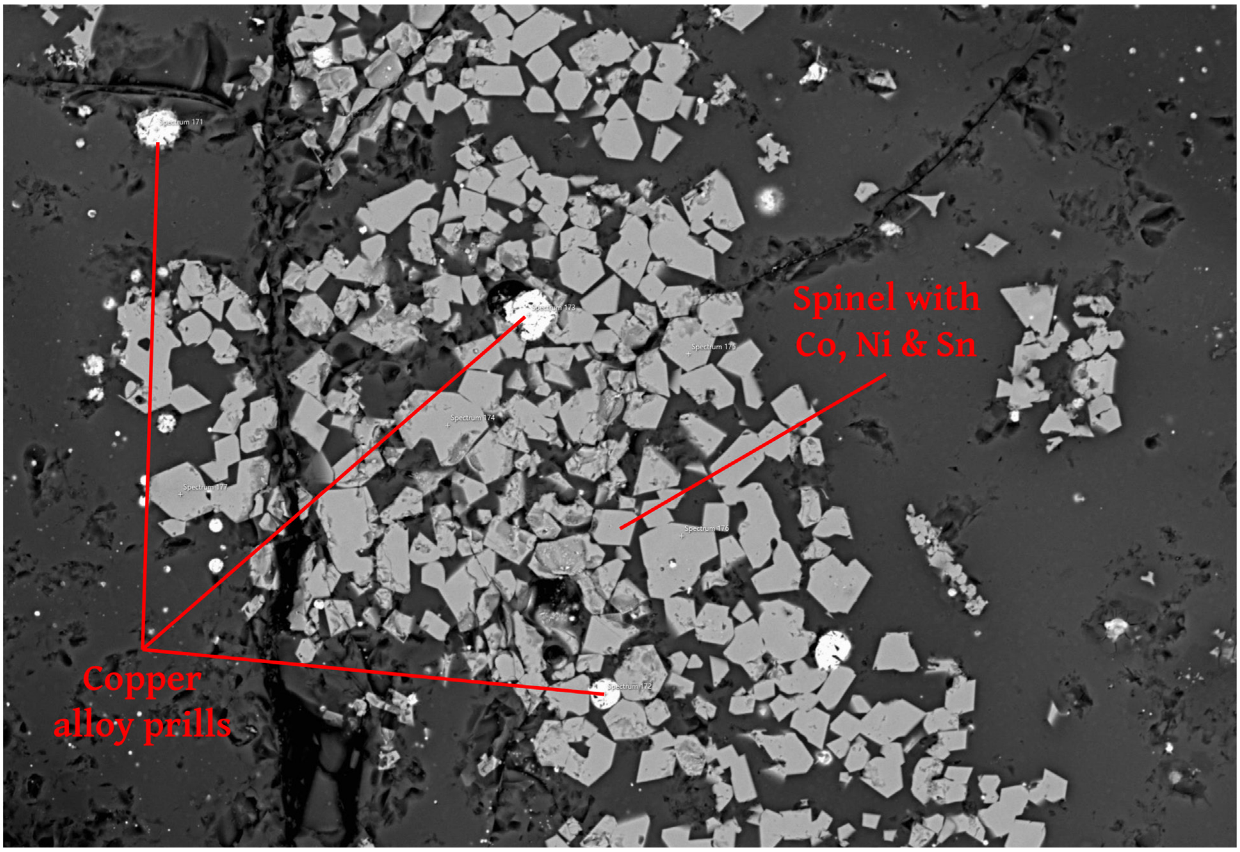

Figure 7. Crucible slag layer in KV 1012 showing arsenical copper prills (2-5 wt% As) with 2.5-5 wt% iron and < 1 wt% nickel, surrounded by spinel (magnetite: Fe_3O_4 with *c.* 1 wt% cobalt, 1.5-2 wt% nickel and up to 0.4 wt% tin) resulting from the (rare) local oxidation of copper alloy prills (width of image *c.* 0.35mm).

melting. Given that substantial sulphur and iron contamination are noted in many crucibles, particularly in dross deposits such as those from KV 1014 and the relatively large deposits above the 'water line' in KV 1011, arsenopyrite addition is a distinct possibility. However, the absolute sulphur content of the charge cannot be confidently assessed as the crucible slag volume is small and sulphur may have been removed as a gas (SO_2) or dross. Indeed, the noted sulphur contamination within the crucibles may equally be explained by sulphur inclusions introduced during 'speiss' alloying.

Before delving into the hypotheses of arsenic additives further, it is worth considering existing literature on arsenical copper. While the recognition of arsenical copper alloys in the Nile Valley is not new (e.g., Garland and Bannister 1927; Lucas 1962), arsenic was usually considered a natural contaminant from the copper ore rather than an actively added component – putting any intentionality of arsenical copper alloys at the copper ore selection level. This idea was challenged (for ancient Egypt) on the basis of elemental analysis by Cowell (1987) and more recently by Rademakers *et al.* (2018c; 2021) based on trace element and lead isotope ratio analysis of copper alloys, ores and smelting waste. The latter suggests an incompatibility between currently known copper ores exploited in Sinai and the Eastern Desert and raw copper from smelting sites – both poor in arsenic – and the arsenic contents in finished copper alloy objects. The (limited) current evidence thus indicates the absence of arsenic in primary metallurgy (preceding crucible melting), although possibly exploited Nubian copper ores remain poorly characterised (Rademakers *et al.* 2022). This stands in contrast to other Bronze Age settings where arsenical (and other) copper alloys were often produced directly from complex ore smelting (cf. Rademakers *et al.* 2018c, 186). This provided strong indications for secondary arsenic alloying, yet more direct proof was lacking thus far (and expected from crucible analysis: Rademakers *et al.* 2021, 24).

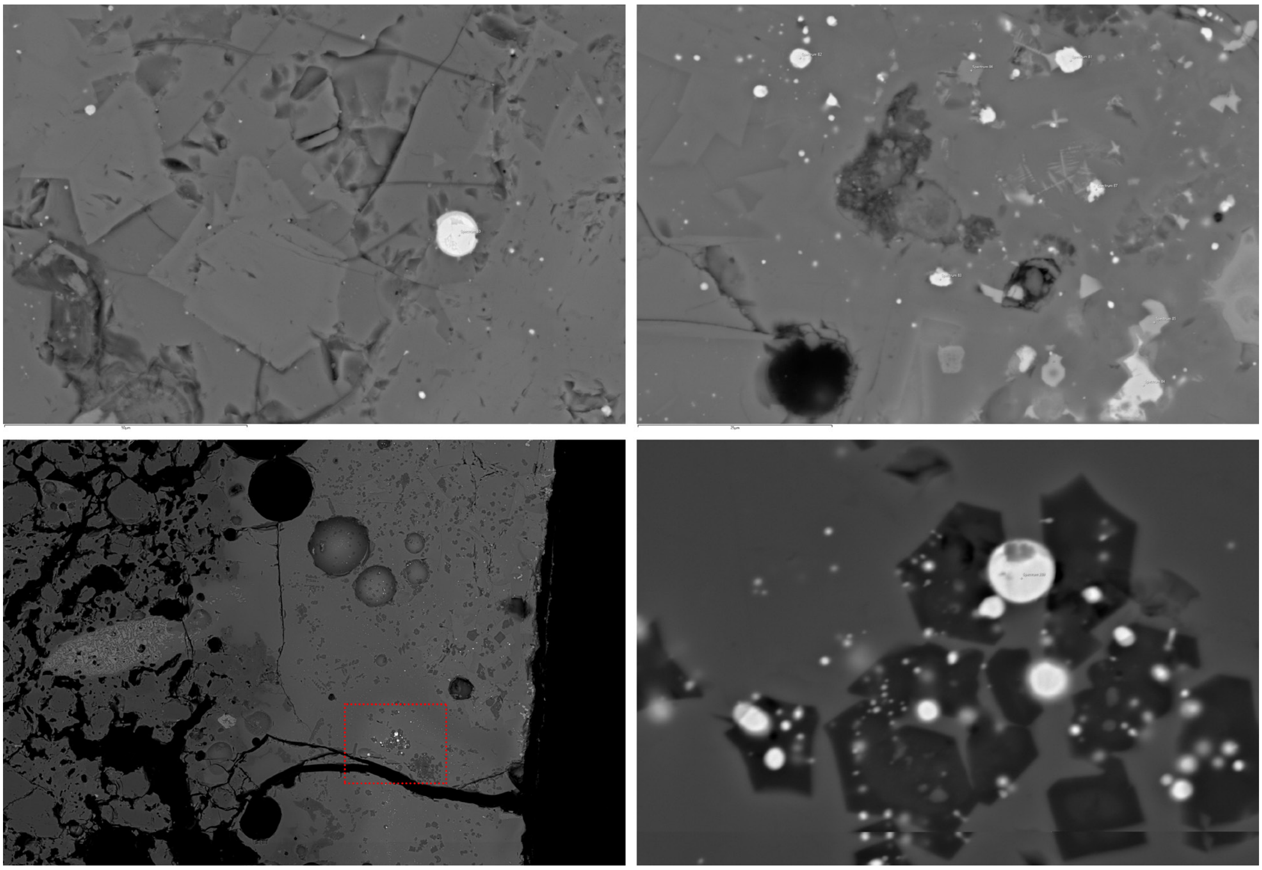

Figure 8. Top left: crucible slag layer in KV1013 including prill with *c.* 41 wt% arsenic and 31 wt% iron (width of image *c.* 130µm). Top right, crucible slag layer in KV1013 including prills with *c.* 20-24 wt% arsenic, 1-4 wt% iron and 2-3 wt% tin (width of image *c.* 80µm). Bottom left: crucible slag layer in KV 1015 and its transition from the ceramic fabric on the left (width of image *c.* 0.85mm). Bottom right: close-up of bottom left image showing small copper prills with *c.* 21 wt% arsenic and 2 wt% iron (width of image *c.* 0.05mm).

Existing literature on secondary arsenic alloying in archaeology is limited. On the one hand, research has focused on the direct co-smelting of (oxide) copper ore and (sulphide) arsenic ore, either experimentally (e.g., Lechtman and Klein 1999; Rostoker and Dvorak 1991) or through analysis of archaeological smelting waste (e.g., Lechtman 1991; Zwicker 1980). On the other hand, secondary alloying has been proposed through an intermediate product called 'speiss' – an apparently intentional (by-)product of copper or lead smelting, consisting of quasi-metallic iron arsenide (FeAs), known from Early Bronze Age Iranian sites such as Arisman (Nezafati *et al.* 2021; Rehren *et al.* 2012) and Tepe Hissar (Thornton *et al.* 2009). These could have been traded as ingots for arsenical copper production, and would have been superior to direct arsenopyrite mixing as most sulphur is removed during 'speiss' smelting (forming 'matte': copper-(iron-)sulphide). Archaeological finds of 'speiss' are rare across the ancient world (e.g., Doonan *et al.* 2007; Georgakopoulou 2018; Soles and Giumlia-Mair 2018) and no Early or Middle Bronze Age examples from Egypt or Sudan are known[9]. Direct identifications of 'speiss' addition to copper (in a crucible) have not been reported anywhere so far.

Could arsenopyrite have been alloyed directly with copper? Copper is expected to bond to sulphur more strongly than iron in a (smelting) system containing copper, iron, and sulphur (Willis and Toguri 2009). This is considered the rationale behind 'speiss' production as an intermediate metallurgical operation

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[9] Yet see likely exceptions from 15th dynasty Tell el-Dab^{c}a (Philip 2006, Reg. no. 1330 = Vienna ÄS2909) and 18th dynasty Tell el-Amarna (Charles 1995).

to facilitate secondary alloying, as the co-smelting of copper oxide ore and arsenopyrite (or the direct addition of arsenopyrite to copper metal) would be far less efficient (Rehren *et al.* 2012). The decomposition of arsenopyrite (into elemental arsenic and FeS at *c.* 650°C: Hayward 1940) and its interaction with molten copper would form copper sulphide, given copper's strong sulphur affinity at high temperature (e.g., Cox *et al.* 1949; Ellingham 1944; Marakushev and Bezmen 1971), and result in a type of 'matte' phase. Yet this particular system has never been described in terms of its thermodynamics. Experimental data suggest the 'matte' may incorporate large amounts of iron (copper-iron sulphides: Rostoker and Dvorak 1991), and a liquid copper bath might further influence process kinetics and element partitioning. In the context of crucible co-smelting, higher arsenic retention and lower iron contamination of the resulting alloy is noted compared to furnace smelting (Lechtman and Klein 1999). But rather than co-smelting oxidic copper ore with arsenopyrite (as in the Peruvian examples discussed by Lechtman and Klein), the addition of arsenopyrite to metallic copper in a crucible is considered here – a method for which no experimental data is currently available.

One could consider a theoretical example: when mixing 200ml (1790g) copper and 50ml (303.5g) arsenopyrite in a crucible, the charge would contain *c.* 104g iron, 140g arsenic and 60g sulphur. If all the sulphur binds to copper, this results in a loss of *c.* 240g copper into a pure Cu_2S 'matte'. This would leave 1550g of copper to bind with 140g arsenic, forming an 8.3wt% arsenical copper if no arsenic is lost. 300g 'matte' would partition from the melt as a separate floating phase (to be manually removed prior to casting, or partially deposited on the crucible walls), and possibly enter the crucible slag to some degree. Iron may be partially included in the metal alloy (as noted in some of the arsenic-rich prills) but is expected largely to enter the sulphide phase too (substituting for copper, as witnessed in Kerma crucibles and by Rostoker and Dvorak 1991) or perhaps be partially oxidised (rarely observed in the Kerma samples). The resulting 'matte' of *c.* 300g would correspond to a volume of *c.* 50ml, or *c.* 20% of the total charge volume. As a comparison, the mass of $(Cu,Fe)_2S$ in the crucible slag of KV 1013 can be roughly estimated at 0.05g (*c.* 3 wt% of crucible slag layer, with an estimated volume of *c.* 7ml), which is tiny compared to the estimated charge mass of 500g in that crucible. If direct arsenopyrite addition was indeed practiced, significant 'floating matte' formation and removal probably took place.

The above is of course a simplified theoretical estimate. 'Matte' development may have been more severe than these estimates suggest, making direct arsenopyrite alloying highly impractical – or perhaps less severe than estimated. Ongoing experimentation by the authors suggests it is indeed a feasible technique, whereby an arsenical copper alloy is formed, and 'matte' formation does not hinder immediate casting. Regardless, we cannot confidently assess the 'matte' or dross volume associated with the Kerma crucibles, as this may have been largely removed prior to casting. Mixing with 'speiss' would surely have resulted in a 'cleaner' alloying operation, yet limited 'matte' inclusions in the 'speiss' could equally explain the crucible slag contamination attested here. Indeed, the use of 'speiss' would have introduced similar iron contamination requiring removal. Therefore, it is difficult to prove which of these was added. Furthermore, the presence of tin in the system (either initially present as part of tin bronze or added as tin metal or cassiterite) could have influenced the alloying process. We thus suggest that either 'speiss' or arsenopyrite was used as an active alloy component to create ternary alloys (under strongly reducing conditions to avoid tin and arsenic loss) at the Middle Kerma casting workshop. This was either added to an existing (scrap) tin bronze, or to raw copper with the further addition of tin (as a metal or cassiterite). High tin contents in the casting spills rather indicate the latter, but conclusive evidence is lacking for now. The active selection of such a ternary alloy may reflect a variety of considerations, ranging from physical properties sought for successful plate casting to the resulting alloy colour and hardness (among others). These questions are the subject of ongoing investigation[10] and not further discussed here.

Sources of metal

This paper does not intend to discuss the provenance of copper used at Kerma. For an overview of five casting spills[11] as well as various copper alloys not related to the large cast plates, the reader is referred to Rademakers *et al.* (2022). It can be noted that the casting spills related to the mould structure have lower cobalt, nickel and antimony concentrations compared to some of the crucible slag prills. This may be related to their dilution during the metallurgical process, as these elements may have been associated with the arsenic source (arsenopyrite or 'speiss') as well as the base copper – most analysed crucible prills have low concentrations of these elements. Selenium and tellurium are expected to fractionate into the sulphide phase during smelting and remelting and thus constitute only traces in the cast metal (as sulphide inclusions: Rehren 1991). The alloy in the spills corresponds well with the crucible prills, although with lower arsenic contents – which may reflect losses during spilling (similar variability has been observed experimentally by the authors). Rademakers *et al.* (2022, 16) suggested that these spills reflect the variable addition of arsenic and tin to the same base copper type, resulting in slight variations between crucible charges. The crucible analysis presented here supports this interpretation.

As far as the arsenic source is concerned, we can only guess for now. Rademakers *et al.* (2022, 16) note that lead isotopic ratios for the casting spills fall along a trend line which may reflect a variable shift from the copper to the arsenic or tin source's lead isotopic signature, yet it is impossible to confidently identify a source this way. As discussed by Rademakers *et al.* (2021), any search for potential arsenic sources is hampered by very scanty literature, and the extent of their possible exploitation in the past impossible to assess without detailed archaeological fieldwork. Possible sources such as Wadi Tarr in Southern Sinai and Umm Semiuki in the Eastern Desert can be cited (in an Egyptian context), but the volumes mined were in all likelihood too small to support large scale alloying in the Nile Valley, and the exact minerals exploited remain unclear[12] (e.g., Abdel-Motelib *et al.* 2012; Hauptmann *et al.* 1999; Ilani and Rosenfeld 1994; Pfeiffer 2013; Rademakers *et al.* 2021). Furthermore, if arsenopyrite was used for direct alloying or as the basis for 'speiss' production, a much wider range of source candidates from the Egyptian and Nubian Eastern Deserts can be cited. The vast majority of gold deposits worked throughout pharaonic history derive from the intimate intergrowth of sulphides such as pyrite and arsenopyrite with gold in quartz veins – although gold exploitation prior to the New Kingdom was focused on weathered alteration zones rather than the actual quartz veins (Klemm and Klemm 2013, 38-45, 614). Arsenopyrite is reported at many gold mining sites in the Eastern Desert, but direct evidence for its mining is not available. Given the important mining activity attested at such sites throughout the Kerma period and the Old and Middle Kingdom in Egypt (as well as earlier and later), it is likely that miners encountered and tested such minerals. Potential sites of interest with attested Kerma presence include Umm Fahm and Duweishat (Klemm and Klemm 2013), but

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[10] Preliminary alloying and casting experiments suggest an important influence from the arsenic component improving cast-ability and freezing range, different from tin alloys, which could have been crucial for the large plate casting (Verly *et al.* 2022). The combined effect of arsenic and tin may further affect alloy casting behaviour.

[11] These casting spills were previously analysed by Schweizer (Bonnet 1986, 22), who initially interpreted the *c.* 1% arsenic in the metal as a contaminant from direct arsenic-rich copper ore smelting. The composition of various metal objects discussed by Rademakers *et al.* (2022) are not expected to be fully reflected in these crucibles, as that dataset is biased towards smaller objects. The exact composition of the cast plates will always remain unknown, as these were almost certainly repurposed over time and made their way into the circulating metal stock.

[12] Sulphur-poor arsenic minerals such as koutekite (Cu_5As_2) and domeykite (Cu_3As) are found associated with native copper and covellite (CuS) at Wadi Tarr (Ilani and Rosenfeld 1994), where a trench of just 20m long and 1-2m wide represents the only documented mining evidence. The lack of iron and sulphur in these minerals may make them less likely candidates in this context still. The mineralisation at Umm Semiuki is complex too (cf. Rademakers *et al.* 2021, 25) and arsenopyrite is apparently rare in the ore deposit (Shalaby *et al.* 2004).

whether arsenopyrite was extracted there, besides gold and possibly copper, remains uncertain for now. Similarly, no evidence from the Nile Valley for the production of 'speiss' is currently known, but this cannot be taken as evidence against production. Regardless, the large-scale production of copper from Sinai and Eastern Desert deposits and its use as arsenical copper alloys for many centuries suggests that a source of arsenic would have been quite readily available to Egyptian and Nubian metallurgists. Rather than something upon which they had to rely through long-distance exchange, it may have been obtained from the Nile Valley and its hinterlands.

Similar to this issue is the question of tin provisioning. While tin may have entered the Kerma crucibles as scrap bronze (which simply pushes the question back one step), the relatively high tin contents in some of the casting spills (up to 7.6 wt%) are suggestive of fresh tin addition. While no tin metal finds are known from Kerma or Old to Middle Kingdom Egypt (and very few from New Kingdom Egypt: Rademakers *et al.* 2023), it cannot be excluded that this was somehow obtained at Kerma. If the Eastern Desert deposits are considered potential sources for arsenic (and perhaps copper, if less likely at Kerma: Rademakers *et al.* 2022), however, they may equally have provided tin in the form of cassiterite (SnO_2). Its direct addition for the production of bronze alloys in New Kingdom Egypt has already been argued by Rademakers *et al.* (2018a; 2023), and might be a pathway for the production of the ternary bronzes at Kerma. Conceptually, the addition of alloy components in mineral form is not that far-fetched (see also Rademakers *et al.* 2018a; 2021; 2023), and may have been a long-standing tradition in the Nile Valley facilitating the transition from arsenic minerals (perhaps treated to create 'speiss') to cassiterite – with a period of overlap. These remain tentative suggestions which cannot be developed further here, but may be tested by the future analysis of other crucibles from workshop contexts at Kerma and elsewhere.

Crucible types

From a bulk compositional perspective, the casting workshop crucible assemblage is highly uniform (cf. Figure 6), although petrographic study reveals some variation between most crucibles (cf. Appendix). It reflects the selection of a relatively refractory base ceramic, rich in silica (*c.* 73 wt%) and alumina (*c.* 22 wt%) with low iron oxide (*c.* 1.5 wt%) and lime (*c.* 1 wt%) content. This composition compares best to Nile clay fabrics, as opposed to so-called Marl pastes as differentiated by the 'Vienna System' (Nordström and Bourriau 1993) for pottery found in the Egyptian Nile Valley (Bader 2001, 20). Petrographically, however, the matrix of the paste seems of a 'calcareous' nature, as expected for Marl fabrics. As the petrographic identification of matrix compositions as more calcareous or iron-rich is influenced to a large extent by oxidation processes (cf. full description of KV 1014 in the Appendix), these petrographic observations cannot be used to identify the fabrics as 'Marls'.

The selected paste has natural non-plastic inclusions (e.g., quartz grains and rock fragments) that provide mechanical stability, while the addition of significant organic temper, resulting in high porosity, further improved their isolating properties and fracture resistance (e.g., Freestone and Tite 1986; Hein and Kilikoglou 2007; Hein *et al.* 2013; Müller 2017). This has resulted in only limited (but measurable) crucible slag formation in most crucibles. The fabrics differ from typical domestic pottery and were very likely developed by the metallurgists, who would have shaped the crucibles by hand.

Further comparative research is needed to clarify to what extent the fabrics share characteristics with other metallurgical crucibles found in the Nile Valley. Nile silt is frequently used in crucible fabrics elsewhere, e.g., at 12-15th dynasty Tell el-Dab^{c}a (Philip 2006) and 19-20th dynasty Pi-Ramesse (Rademakers *et al.* 2018a). However, none of these crucibles have been analysed using thin section petrography, and only the Pi-Ramesse assemblage using SEM-EDS (polished blocks). As at Kerma, zircon was encountered in the Pi-Ramesse crucibles (Rademakers 2015, 577), while monazite was not noted. Both are typical accessory

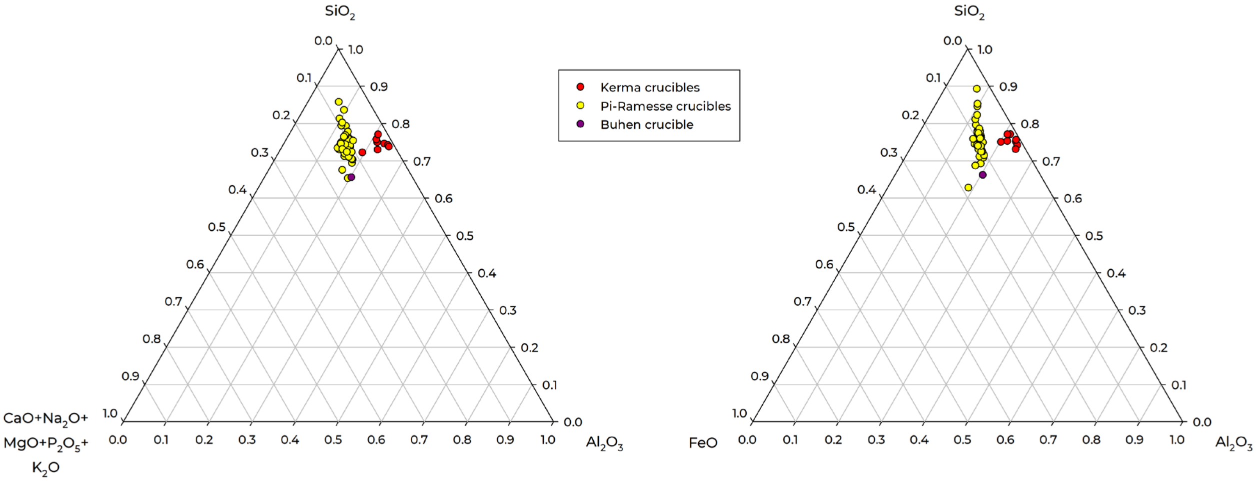

Figure 9. Comparison of ceramic fabric composition between metallurgical crucibles from Kerma (this study), Middle Kingdom Buhen (Davey *et al.* 2021) and New Kingdom Pi-Ramesse (Rademakers *et al.* 2018a).

minerals in igneous rocks, and could point to the contamination of the Kerma crucible slag by the addition of placer cassiterite (monazite can be found in placer deposits of Eastern Desert granites (e.g., at Igla and Nuweibi), associated with other heavy minerals such as cassiterite, ilmenite and zircon). However, both are equally encountered in (Nile) sediments: zircon is not uncommon in Nile fabrics (Bourriau *et al.* 2000) and monazite is found in the so-called black sands (beach placers) on the Mediterranean coast (e.g., Abdel-Karim *et al.* 2016; Said 1990). Given that these minerals are mostly found in the ceramic, they reflect the crucible fabric selected at Kerma – perhaps a local Nile silt variety – rather than its charge.

A comparison with the very limited analytical data published for crucibles from the Nile Valley shows that Kerma crucibles are more refractory than those from Pi-Ramesse, as reflected by their higher alumina content and lower concentrations of iron, calcium and other metal oxides which generally lower the ceramic melting temperature. Interestingly, the Middle Kingdom crucible from Buhen is compositionally far more similar to New Kingdom crucibles from Pi-Ramesse (cf. Figure 9). This may indicate a technological link between practices documented at Buhen (based on only one crucible, of course) and Pi-Ramesse. More detailed analysis of other crucible assemblages is needed to assert wider patterns in fabric selection, the possible mobility of craftspeople and technological exchange along the Nile Valley reflected in these crucibles.

Despite an overall similarity in the base paste, at least three different crucible ‘systems’ can be distinguished within the Kerma assemblage. Firstly, there are the large crucibles of type MAH 27796 (which include KV 1016 and KV 706). These would have held a relatively large volume of copper alloy, and several can be directly associated with the large casting mould. Based on volume estimates, at least 20 of these would have been needed for almost simultaneous casting – an extraordinary feat with myriad practical implications not discussed here (further discussion in the full publication of the 2018-2019 re-excavation – see also the melting batteries at Pi-Ramesse: Pusch 1990; 1994). These crucibles are thick-walled with high porosity derived from organic (likely dung and sometimes possibly hair) temper mixed into the paste (cf. Figure 10), and have two openings facilitating their manipulation. Their element composition has only been determined for KV 706, which has slightly higher iron content compared to the other crucibles. It appears that variations in the temperature and redox-conditions during the metallurgical process resulted in slightly different firing colour (and degree of vitrification) of these larger fragments.

The second main group consists of various smaller crucible types, each with a highly similar core fabric: KV 1011-1015 and KV 422. These are all more thin-walled and grey-fired. A further distinction

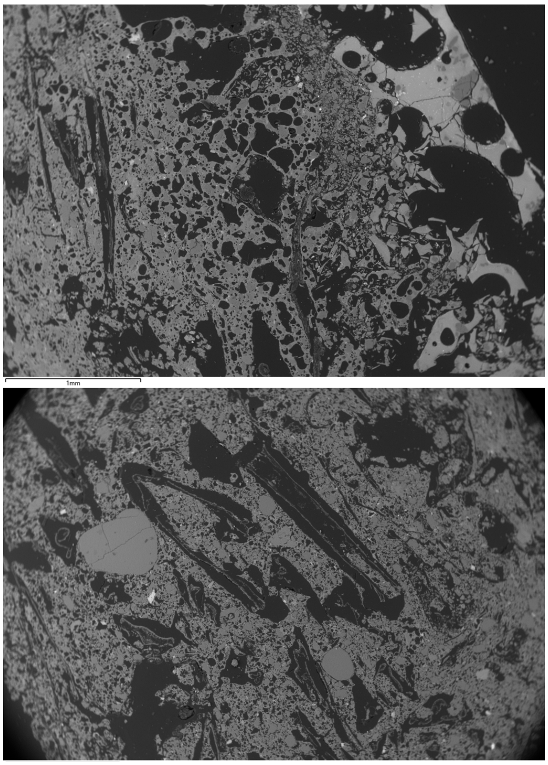

Figure 10. SEM-BSE images of KV 706 (width of images *c.* 4mm). Top: crucible slag formation at interior, with underlying bloated ceramic. Bottom: porosity from burnt-out organic temper, with residual phytoliths.

can be made on the basis of their shape (cf. below), but also on the use of external layers. Within this group, KV 1012 and KV 1013 have no external layer applied and a more oxidising external environment is reflected in their cross-sections. In contrast, KV 1011, KV 1014, KV 1015 and KV 422 all have an additional layer applied to their external surface. These additional layers appear to be the same in KV 1011, KV 1014 and KV 1015 (example shown in Figure 11), while KV 422 (not related to the Middle Kerma casting) is different. Within the former group, the added layer (which did not always cover the entire external surface) is completely vitrified in KV 1011 but only partially sintered/vitrified in the other two (both of a different shape). In KV 422, the external layer appears to have been applied to the crucible interior as well, resulting in the formation of a similar external and internal glaze.

The application of these external layers may appear a surprising technological choice at first glance. Their fabric is, in refractory terms, quite different and 'inferior' to that of the core ceramic material (strongly enriched in iron and other elements lowering its melting temperature, with more mafic minerals and rock fragments compared to the other fabrics). A vitrified and 'wet' external layer might even appear detrimental to the handling of these crucibles at high temperature – something intended at least for KV 1011, as indicated by the casting spout. This type of external layer has not been previously identified using micro-analysis for crucibles from the Nile Valley, but similar lining of already refractory crucibles (sometimes domestic pottery) with an iron-rich layer is known in Roman crucibles (e.g., Gardner *et al.* 2020; König and Serneels 2013; Rademakers 2015). These layers may have fulfilled different functions such as insulating, distributing heat and reducing thermal shock (Bayley and Rehren 2007). While layered crucibles are known from Old and Middle Kingdom Egypt (e.g., at Ayn Soukhna and Wadi el-Jarf: Verly *et al.* in preparation), the layer fabrics in those crucibles tend to be more similar to those of the core. The identification of layered crucibles at Kerma thus highlights another technological tradition previously unknown for the Nile Valley.

Apart from the small opaque inclusions frequently noted in all fabrics, larger opaque fragments were only noted petrographically in KV 1016 (not analysed by SEM-EDS). However, in KV 1011, KV 1012, KV 1014 and KV 1015 fragments of up to a few mm were noted by SEM-EDS, consisting of porous iron silicate of vitreous appearance with partially decomposed quartz – which might be the same as the large opaques in KV 1016. Based on SEM-EDS analysis, these look similar to partially vitrified ceramic fragments and more specifically fragments of the externally applied layers. This might indicate a form of grog tempering (using old crucible fabrics), although its identification must remain tentative for now.

In terms of overall shape, the second main group exhibits at least two types. Firstly, there is the relatively large, shallow and open shape of KV 1011, which also has a clear spout. Secondly, there are apparently hemispherical shapes of different size exemplified by KV 1012 and KV 1013. These may have had a spout at the missing end, but this cannot be determined. Finally, KV 422 represents another

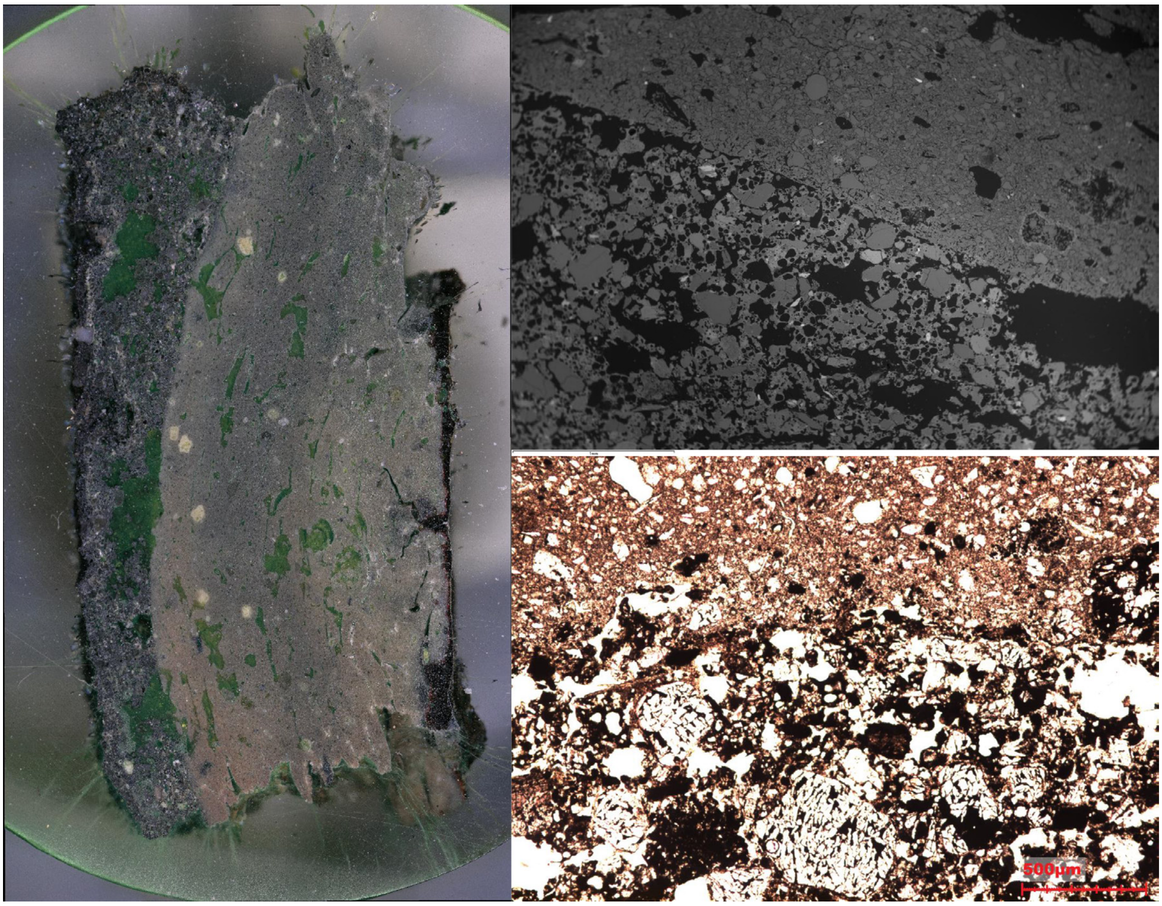

Figure 11. Mounted cross-section of KV 1015 (left) with abrupt transition between externally applied layer, shown in SEM-BSE image (top right, width of image *c.* 4mm) and PPL micrograph (bottom right, width of image *c.* 2.5mm).

small type, possibly similar to the latter. These shapes, particularly the smaller types, do not support a sufficiently large volume for them to be credible candidates for supplying large plate casting. Rather, they probably indicate ancillary activities (e.g., the casting of smaller tools needed at this workshop) or perhaps a segmented production chain. For example, it may be possible that (concentrated) alloys were first manufactured in smaller crucibles and then added to the larger crucible types as 'clean' master alloys for casting. However, a more detailed analysis of larger crucible assemblages is needed to confidently assert this.

A final interesting feature to note is the apparent repair of crucible KV 1012. Following a first phase of metallurgical use (resulting in crucible slag development at the interior), a new layer of the same fabric type was applied to the entire crucible interior, over the rim and smoothed down on the external surface. When not viewed in cross-section, this layering is almost impossible to detect. Similar crucible repair by relining the interior has been noted at New Kingdom Pi-Ramesse (Rademakers 2015, 168-171) and Amarna (Rademakers forthcoming), although it seems to be a rarity. Here, it is particularly surprising to see such a small crucible being repaired, as making a new one would not require significantly more raw materials, and there is a relative interior volume loss. This observation, like the possible use of 'crucible grog', may reflect particular attitudes of the metallurgists at Kerma regarding the long-term care of their crucibles, which were a metallurgist's most crucial tool.

Conclusion

This paper presents the first analytical study of crucibles from ancient Kerma. This detailed analysis provides strong evidence for an active alloying practice, as part of secondary metallurgy rather than through ore selection for primary metallurgy. This involved the melting of copper in a crucible, to which a high arsenic component (‘speiss’ or arsenopyrite) was added to create an arsenic-rich alloy. Rather than a binary alloy, however, ternary alloys were made by the further inclusion of tin. It is uncertain at this stage if tin was added separately (as metal or cassiterite) or by melting an existing tin bronze to which arsenic was added. However, high tin contents in the casting spills indicate the former.

At any rate, the metallurgists at Kerma did not ‘simply’ melt down existing alloys to obtain sufficient alloy volumes to cast large plates. Rather, they made use of all available means to create fresh alloys, including tin as a relatively novel alloy component. This may illustrate the transitional stage of a new technology being adopted, while still relying on the more familiar arsenical copper system. Indeed, the physical properties afforded by arsenic in the alloy may have been crucial to the success of this casting process. Beyond technical considerations, the alloy may have served to showcase the flexibility and intimate knowledge of these metallurgists, creating a mixed alloy of unusual appearance for conspicuous display of these plates, probably reworked into architectural features.

Although the motives may remain obscure, the analysis of only a small set of crucibles shows their power to improve our understanding of metallurgical technologies at Kerma and beyond. It is crucial to study this archaeological material in further detail at a much wider range of sites in the Nile Valley to understand how copper alloys were produced in different contexts over time. It is likely that the secondary alloying of arsenic also took place downstream in Egypt, as predicted from the analysis of copper alloy artefacts. Crucible studies thus hold the potential to reveal hitherto unknown alloying technologies and clarify patterns observed in metal assemblages. Apart from alloying techniques, the crucible fabrics and typologies can illuminate the particular practices of craftspeople operating in different regions and build a framework to investigate their mobility and wider technological interaction over time. In the particular setting of Kerma, these results underline the metallurgical skills associated with a unique casting technology.

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