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Small Size, Large Scale
Roman Brass Production in Germania Inferior

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A new type of Roman crucible is attributed to brass making on the evidence of chemical and microscopic analysis. Clearly, being technical ceramic used in a high temperature process, these vessels differ significantly in their design from known Roman copper-alloy melting crucibles. Upon scientific analysis, the size, shape and fabric characteristics were found to match the specific thermodynamic requirements of cementation for brass production, while several other possible interpretations were convincingly excluded.

Introduction

The production and use of brass on a regular scale apparently only began in the 1st century BC, although several much older brass objects are known (Craddock, 1978). This is most probably related to the invention of the cementation process, allowing the controlled production of brass instead of the co-smelting of naturally mixed copper-zinc ores. For a detailed discussion of most aspects of early brass technology, and a full bibliography, the reader is referred to the recent edition of the BMOP 50 (Craddock, 1998).

In contrast to sound numismatic and object-based evidence for the emergence and spread of brass (Caley, 1964; Craddock, 1978; Dungworth, 1996; Hook & Craddock, 1996), very little archaeological evidence for Roman brass making is known. Bayley (1984, 1998) published some Romano-British crucible fragments related to brass production, and only recently Picon, Le Nezet-Celeston & Deskat (1995) postulated a large brass making workshop near Lyon. Around the same time several hundred small vessels from Xanten, probably predating the official foundation of Roman Colonia Ulpia Traiana (CUT) in Germania inferior, were identified as brass making (as opposed to brass melting/casting) crucibles. Their morphology, analysis and interpretation are presented here.

Roman Brass Working Crucibles

A wide range of Roman crucibles is known for bronze and brass casting. Sizes, shapes and fabrics vary widely, depending on the needs and possibilities of the local coppersmiths. No typological differences are known between bronze and brass melting crucibles. Typically, they are about fist-size, bag- or pear-shaped and have walls about 10 mm thick. Some are covered by a second, less refractory clay layer, either around their base to improve heat resistance (J. Bayley, pers. comm.), or covering the top suggesting the former presence of a lid or a mould luted on to enable secure casting (cf. Eckert, 1990).

A New Crucible Type

Against this background, fragments of hundreds of crucibles from the CUT are to be seen, not matching any of the typical Roman crucibles. They originate from a small pit within the excavation site 79/4 at insula 37, measuring less than 1 square metre. The dark grey, heavy soil of this pit contained (beside the crucible fragments) much charcoal, traces of iron and copper alloys, and burnt bones. According to the accompanying pottery and numismatic evidence, this feature dates to the very beginning of the 1st century AD (Boelicke, pers. comm.).

The entire material is very homogeneous in shape, size and fabric. Two main fragment types are evident (Figure 1), one being from small cup-shaped vessels, the other from flat caps or lids with a solid central hub. A range of cups were measured and found to have an average diameter of about 30 mm and a depth of 20 mm. Cups and lids match perfectly, and many vertically split examples were found, showing the lids overlapping the walls. From the measured internal sizes an average volume of about 15 to 20 cm^3 can be calculated. The vessels are of a very thin, porous and brittle fabric of grey (outside) to black (inside) colour. The walls are usually only 2–3 mm thick. Visual inspection of the fabric revealed a slight overall vitrification of the outer surfaces, but no such signs in the interior.

Analysis

Polished thin sections and bulk chemical analyses by ICP-OES were made of several fragments from

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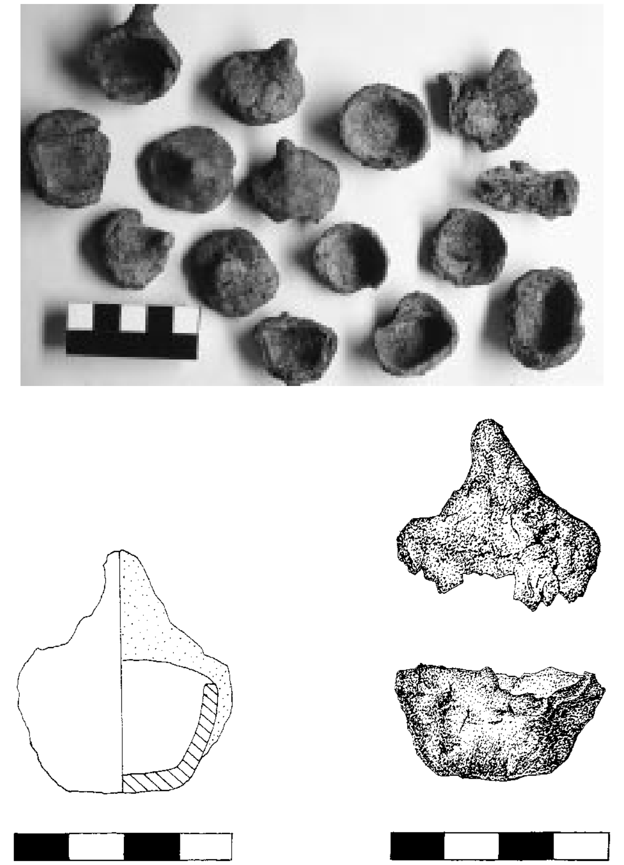

Figure 1. (a) A few caps and cups of brass making crucibles from Xanten, Germany (scale in cm). (b) Reconstruction of brass making crucible from Xanten, Germany. The caps and cups are made from the same clay (scale in cm). Drawing by K. Engel.

different individual vessels, following standard routines. The analyses are given in Table 1, revealing a moderate, though significantly high zinc content of the fabric, but almost no copper. The high proportion of silica is due to abundant quartz grains. The relatively low concentration of alumina, together with considerable amounts of iron oxide and alkalis, indicate that a clay of only limited refractoriness was used. For comparison, an analysis of a local, ordinary brass melting crucible is also given in Table 1, with almost the same ceramic composition, but a wall thickness of more than 1 cm. There is no reason to assume a non-local origin or a specific selection of the clay used in making the small crucibles under study here. As far as the trace elements are concerned the low lead content is notable. At first glance this may indicate the use of refined zinc oxide, i.e., an artificial material rather than natural calamine ore, which often contains abundant lead phases. However, it was found that during cementation experiments lead (from cerussite or galena) enters preferably the metal phase (Maréchal, 1938), and not the ceramic. In view of the more noble character of lead as compared to zinc, this is no surprise.

The microscopic investigation of the samples confirmed the macrostructure of the vessels as established by visual inspection. Cups and caps were made from the same clay, luted together while still plastic, and heated from the outside. The slight 'overglaze' appearance of the exterior, however, turned out to be the result of a total vitrification of the clay matrix, with well-rounded vesicles throughout the fabric. Only the quartz grains appear unreacted, thus providing some structural stability. The volume percentage of quartz in the matrix is hard to determine, owing to the pumice-like texture; a rough estimate is made to about 50 vol% free SiO_2. All other compounds including zinc oxide are concentrated in the vitrified matrix. It is important to note that no inclusions of metal were found in the samples except one heavily corroded speck of copper-rich substance, adhering internally to one fragment of a cap.

Interpretation

The fragments studied belong to a vessel type without known parallels in Roman archaeology. They are evidently related to a high temperature process involving the presence of significant amounts of zinc, but lacking the feature of metal droplets trapped in a slag layer in the interior of the vessel, typical for casting crucibles. Apparently, this process required closed vessels of a particularly small size, but without much need for mechanical strength or special refractoriness.

Table 1. ICP-OES analysis (in wt%) of four different brass cementation crucible fragments (72a–h). Data for one melting vessel (71) are given for comparison

	SiO_2	Al_2O_3	FeO	MgO	CaO	Na_2O	K_2O	ZnO	Cu	Pb
72a	75·6	12·1	3·6	1·3	1·5	2·3	3·7	0·71	0·01	0·04
72b	73·4	11·3	4·5	1·1	1·7	1·8	3·2	2·15	0·01	0·05
72f	75·8	10·4	3·6	1·3	1·0	1·3	3·2	2·54	0·10	0·04
72h	77·9	10·3	3·5	1·3	0·9	1·2	3·2	1·20	0·07	0·02
71	78·0	10·8	3·7	0·7	0·5	2·4	3·9	0·02	0·01	0·05

The high content of ZnO in the top four analyses is significant for the brass cementation, in particular in combination with low copper concentrations. Analyses carried out by W. Steger.

The interpretation given here is that these vessels served as reaction containers to produce brass by the cementation process, involving the solid state reduction of zinc ore to zinc vapour which subsequently reacted with copper to form brass. The discrepancies between the crucible fragments studied here and typical Roman crucibles have been noted already; all peculiarities are to be explained by the special requirements of the process.

An Outline of Brass Cementation

The theory and practice of brass cementation have been studied in depth by various scholars (Percy, 1861; Maréchal, 1938; Caley, 1964; Werner, 1970; Haedecke, 1973; Grothe, 1973). It is widely, though not universally, accepted that there is an upper limit of zinc uptake in brass around 30 wt% Zn, which matches nicely the composition of the vast majority of ancient brass objects analysed so far. During the reaction of zinc oxide with carbon in the presence of copper metal, zinc vapour is formed and immediately picked up by the copper to form brass. Conversely, high-zinc brass emanates zinc vapour to the ambient atmosphere when heated until an equilibrium is reached between metal and gas composition. The maximum amount of zinc contained in the metal is mainly controlled by the temperature, the partial pressure of zinc vapour in the vessel, and the redox conditions. The transformation of zinc oxide into metal requires strongly reducing conditions similar to iron smelting. The heat balance of the process is highly negative, i.e., the heat consumption of the reducing process is not at all compensated by the oxidation of the carbon. A minimum temperature of about 900°C, i.e., near the boiling point of zinc, is necessary to keep sufficient zinc in the vapour phase and enable it to contact and enter the solid copper metal. An upper temperature limit, however, is given by the fact that the added copper (sheet, shot or filings) should stay solid throughout the process to offer a large recipient surface. For a copper alloy of about 20 wt% zinc this upper limit is approximately 1000°C, while the melting point decreases to about 900°C when the zinc content rises to 30 wt%. Since brass cementation involves the formation of a zinc vapour, this has to be kept inside the crucible in order to react with the copper present and to separate it from any atmospheric oxygen to avoid re-oxidation. These two factors, the need to provide a suitable partial pressure of zinc vapour and to keep it apart from oxidizing gasses like CO_2 and O_2, require the use of closed reaction vessels, i.e., neatly lidded crucibles.

Vessel Peculiarities

The small size, thin walls and closed lids of the vessels discussed here are all due to the negative heat balance of the cementation process. Haedecke (1973) in his experiments always measured 100 to 150°C lower temperatures within the charge than outside. In smelting furnaces the heat consumption of metal reduction is compensated by the burning of additional charcoal for heat generation. Also, the combustion of some of the carbon monoxide to carbon dioxide in the upper parts of the furnace, outside the reduction zone, preheats the charge. Owing to the volatile nature of metallic zinc above about 900°C, this does not work here: the zinc vapour would rise and immediately re-oxidize in the upper, less reducing parts. Hence, closed reaction vessels are needed and the necessary heat has to be supplied from outside the vessel. Small, thin-walled vessels are the ideal solution for this. Small vessels have a large surface relative to their volume, which is important, since heat transfer is a function of the surface, while the heat consumption depends on the volume of the charge. As ceramics generally act as bad heat conductors, thin walls are less obtrusive against heat flow than thicker ones, and hence profitable.

The very homogeneous, almost superficial vitrification of the outer surface of the vessels excludes that they stood in a bed of charcoal, the typical Roman method to heat melting crucibles to temperatures up to 1000–1100°C. This direct contact between charcoal and crucible typically results in a vitrification and softening of the outer ceramic which then often takes impressions of charcoal lumps. This, however, would destroy the extremely thin-walled crucibles, and therefore appears highly unlikely as a heating mode. The optimum temperature range for the cementation process is somewhat lower, engulfed by the boiling point of zinc and the melting point of brass, i.e., roughly between 900 and 1000°C. Any heat above that has to be avoided, because otherwise the vessels would collapse or explode (see below), and the forming alloy could melt, reducing the zinc-receptive surface of the copper drastically. On the other hand, too low a temperature would prevent the formation of sufficient zinc vapour, also resulting in unsatisfactory brass production. To ensure working in this relatively narrow temperature range without the danger of overheating, an indirect heating device seems to be the best way. Such indirect heating systems, where a separate combustion chamber supplies a constant stream of hot air, were well known in the Roman world and are supposed for our process here as well. The lack of contact with the charcoal prevented the formation of an outer slag by fluxing ceramic material with charcoal ash, thus minimizing the macroscopically visible heat effect. This model is further augmented by the central protrusion of the caps, forming a nice grip to handle the crucibles with pincers. Heating and cooling curves of indirectly heated furnaces are rather slow, which are therefore preferably run continuously over a longer period than just one process cycle. Thus, the use of pincers is required even to put in the cold, fresh vessels, and to remove them later without the need to let the entire furnace cool down.

The light grey colour of the clay appears to be in contrast to its relatively high iron content. Microscopy and microanalysis of the vitrified matrix, however, revealed that the clay was fluxed by zinc oxide, while the iron seemed to occur as tiny, partially submicroscopic metallic particles scattered throughout the colourless glass. As soon as the (coarsely simplified) reaction $ZnO+C$ to $Zn+CO$ starts, which transforms two solid phases into two gaseous ones, the pressure within the vessels must have increased dramatically, forcing metallic zinc vapour into the matrix, where it acted as a reducing agent to transform iron oxide into metallic iron and zinc oxide, which dissolved into the glass. Only the interior surface of the vessels is discoloured black from residual charcoal dust. The uptake of zinc oxide by the ceramic is limited, of course, by the amount of iron oxide present; any surmounting zinc vapour will just pass through the ceramic and disappear with the fumes. Therefore, the total zinc content of the ceramic seems small compared to later brass making crucibles operating under different conditions (Th. Rehren, unpubl. results), easily rising there to more than 10 wt%.

Other Possibilities

Small sizes of vessels are often indicative of the working of gold or silver, and, indeed, several crucibles of almost identical shape and size were positively attributed to gold casting (Bayley, pers. comm.). These Dark Age vessels from Wales, however, have "*a single spout pulled out from the clay of the bowl*" (Alcock, 1963: 142), i.e., they are not entirely closed. Such a closed situation, evident from the Xanten vessels, then could indicate the parting of gold and silver by the chlorine process (Bayley, 1991; Meeks *et al*., 1996). In our case the small size of the vessels is balanced by their huge quantity, and neither chlorine nor gold or silver were found in them at any level elevated above the geological background. This possibility has therefore to be excluded. Other, even more unlikely, possibilities related to closed crucibles include the solid state carburization of bits of iron (excluded by the small size, not suitable to hold even tiny blades, etc.) or the production of liquid crucible steel (excluded by the low refractoriness of the fabric and the wrong cultural context).

Apart from metallurgy, zinc oxide in antiquity played an important role in medicine. During the 1st century AD classical writers repeatedly mention the use of zinc oxide as a pharmaceutical. Dioscorides, for instance, mentions it in his *De Materia Medica* as a by-product of brass melting, and copper, lead and silver smelting. Even the oxidative burning of some ores to get zinc oxide is mentioned. The installations used to collect it from the fumes, however, are either thin iron rods introduced into the flues or a separate collection chamber on top of the furnace proper. In any case, oxidizing conditions are maintained, quite contrary to our closed crucibles. The further treatment of the zinc oxide were cold processes to obtain various pasty or powdery ointments: again nothing matching our high temperature vessels.

Conclusion

The attribution of the crucibles to brass cementation appears as the most likely scenario, matching perfectly the process as reconstructed from what we already knew about Roman brass making in general and the new evidence presented here from Xanten. A charge, probably consisting of zinc oxide (either calcined natural calamine, i.e., zinc carbonate, or artificial zinc oxide retrieved from lead smelting furnaces), ground charcoal and copper metal, was enclosed in hundreds of small, lidded crucibles. After drying, these containers were placed into a stream of hot air, possibly in an indirectly heated furnace. The mechanical strength of the softened ceramic material is considered too low to hold the pressure developing during the reaction, which was released instead through pores or cracks and by diffusion. During this, some zinc vapour was forced not only into the copper metal to form brass, but also into the ceramic matrix. The absorbing capacity of this matrix, however, was limited, resulting in a relatively low zinc content of the used fabrics.

Outlook

It has to be noted that most of the features considered here as necessary and indicative for this process are absent from another Roman ceramic ensemble recently attributed to the same process (Picon, Le Nezet-Celestin & Desbat, 1995). In contrast, some other cementation crucibles from Romano-British excavations, dating to the mid-1st century AD, are of a small size similar to the Xanten examples, and also very friable and rich in zinc (Bayley, 1998: 11, Plate 2), but again of a different design. Only future work, including much more archaeological material related to brass production, but also theoretical and experimental approaches, will resolve the full range of varieties used by the Romans for this process. The outline of this process and its possible relation to cadmea as discussed by Bayley (1998: 9–11), however, is already confirmed by the recent interpretations.

Open questions in relation to the vessels described here include the difficulty of explaining why the sealed crucibles did not explode upon heating, and which kind of raw materials were used. Furthermore, the situation of these vessels in a civilian, probably even non-Roman settlement is noteworthy, and contradicts the often assumed, military-controlled, state monopoly of brass production during the 1st century AD.

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