

TAYNTON BRASSMILL, NEWENT,
GLOUCESTERSHIRE
EARLY 17TH-CENTURY BRASS MANUFACTURE

TECHNOLOGY REPORT

David Dungworth and Roger Wilkes



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SUMMARY

A selection of refractory and metallic debris from the site of an early 17th-century brass mill have been examined. Chemical analysis of the metals shows that most are brasses with less than 30wt% zinc (small proportions of other elements were also detected), consistent with the production of brass using a cementation process. The crucibles examined showed elevated concentrations of zinc which led to the formation of a zinc spinel which gave rise to a distinctive pale blue colour. The interior vitrified surface of the used crucible showed elevated concentrations of elements (Mg, Ca and Ba) likely to have been impurities in the zinc ore used.

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THE BRASSMILL AT TAYNTON

The samples of crucible and amorphous copper alloy lumps (see below for further details) were recovered from the surface of ploughed fields (centred on NGR SO 7336 2072) in the parish of Taynton, Gloucestershire by Don Sherratt (Green Croft Farm, Taynton). This material appears to have been generated by a brass mill in the early 17th century. Numerous 16th- and 17th-century artefacts have been recovered from the same field (Gloucestershire Historic Environment Records 4008), including three large Swedish copper coins. The *Calendar State Papers Domestic* for Charles I (16/205/11) refer to a Gloucester pinmaker (Tilsley) who obtained his brass from Taynton in about 1632 (see also Caple 1995, 227). The Victoria County History also records the presence of several 'Dutchmen' (possibly brass manufacturers) in the parish during the 1630s and 1640s (Jurica in preparation).

POST-MEDIEVAL BRASS MANUFACTURE

Brass is an alloy of copper and zinc but before the mid 18th century zinc metal was a rarity in Europe and the alloy was made by a cementation process. Zinc is a very volatile metal and under normal smelting conditions it would be lost as a vapour. The cementation process used crucibles in which copper metal and zinc ore were mixed with charcoal; the crucible was then sealed and heated. The charcoal reduced the zinc ore to metallic zinc which was then partially absorbed by the copper to produce brass (Bayley 1998; Day 1998; Hawthorne and Smith 1979, 143–144).

Theoretical and experimental investigations of cementation brass have shown that it is difficult to obtain more than c28wt% of zinc in the finished brass (Haedecke 1973; Newbury *et al*/2005). The chemical analysis of medieval and earlier brass artefacts confirms that very few examples contain more than 28wt% zinc (Blades 1995). During the 16th and 17th centuries most brass appears to have contained less than 28wt% zinc (eg Dungworth 2005), but a small proportion of brass artefacts were made which contained higher proportions (up to 33wt%) of zinc (Mitchiner *et al*/1985). Some of these artefacts may have been produced using an improved cementation process. Mitchiner *et al* (1985, 153) suggest that brasses with 28–33wt% zinc could be made using the cementation process but with granulated rather than lump copper — the increased surface area of the copper allowed the absorption of more zinc. In the 18th century Champion began to produce metallic zinc in England using a combined smelting and distillation technique which allowed the manufacture of brasses with any zinc content (Dungworth and White 2007).

The production of copper and brass in Britain during the later Middle Ages appears to have been at a modest level with most demand for these metals being met by imports from the rest of Europe (Blair and Blair 1991, 83–4; Goodall 1981; Hammersley 1973). Concerns over strategic resources led to the encouragement of English copper and brass

industries in the 16th century. The Company of Mines Royal was established to mine copper, silver and gold (Donald 1955; Hamilton 1967) while the rights to produce brass were assigned to the Mineral and Battery Works Company (Donald 1961; Hamilton 1967). Subsequent events suggest that most of the brass was intended to be used for the manufacture of cards (that is wire brushes) for the wool-processing industry.

In the 1560s the Mineral and Battery Works Company attempted to produce brass from copper and calamine (probably the zinc ore smithsonite, $ZnCO_3$) at Tintern Abbey, on the Welsh side of the River Wye (Donald 1961, 86–94). In February of 1568 it was reported that the Mineral and Battery Works Company had produced brass at Tintern but the product proved to be unsuitable for battery (forging). The Company quickly shifted its attention to the production of high quality iron that could be drawn into wire (for the manufacture of wool cards). The reasons for the poor performance of the Company's brass manufactured at Tintern in 1568 are far from clear. In reporting the production of brass it was stated that one hundredweight of copper increased in weight by 28–35 pounds. Assuming that all of the increased weight was due to the zinc absorbed during the cementation process, the brasses produced would have contained 20–24% zinc (Donald 1961, 91). This is somewhat less than the best brass of the period but would have been sufficient for most purposes. It is possible that the calamine used contained too much lead, sulphur (especially if some of the ore was the zinc sulphide sphalerite rather than the carbonate smithsonite), or some other undesirable impurity which might have made the resulting brass brittle.

In the 1580s the Company leased the privilege to manufacture cementation brass to a group (including John Brode) based at Isleworth who succeeded in producing 25 hundredweight of brass from 18 hundredweight of copper (implying a zinc content of 28wt%). Despite protracted legal disputes between Brode, his partners and the Company, substantial quantities of brass appear to have been manufactured at Isleworth in the late 16th century. During the early 17th century the Company leased privileges for the manufacture of brass to a number of individuals and groups. In 1630 William Webbe and William Blaydon were granted the privilege along the River Severn from Bristol to Shrewsbury (Hammersley 1977, 40). In the following year the privilege was transferred to Charles Powell, Thomas Jennings and Richard Ley and battery works are recorded in the Forest of Dean. In 1639 Powell's lease was transferred to Thomas Weston and it is further recorded that there were works in which brass and brass wire were made (Hammersley 1977, 40).

AIMS

The archaeology of the brass manufacturing industry of the 16th and 17th centuries is very poorly known. Some details are known from contemporary historical sources but these tend to provide most detail where disputes ended in court proceedings and do not provide sufficient detail on technical aspects of the industry. While the analyses of 17th-century coins and tokens undertaken by Mitchiner *et al* (1985) provide important information about the use of brass and other copper alloys in this period, it is not certain whether brass used for coins was produced in Britain or imported from continental Europe.

The examination and analysis of the brass-working debris from Taynton was undertaken to answer the following questions:-

- 1) was brass produced or simply melted at Taynton?
- 2) what sort of brass was produced or melted at Taynton?

DESCRIPTION OF ANALYSED MATERIAL

The material comprises base fragments of two crucibles (Figure 1) and 15 amorphous lumps of copper alloy (Figure 2). The two crucibles each have a base diameter of 95mm, a wall thickness of 40–60mm and survive to a maximum height of 110mm (neither crucible, however, survives to anything like its full height). The first crucible has a buff-coloured fabric (I6) while the second has a pale blue fabric (I7). The visual appearance of the crucibles suggests that I6 had not been used while I7 had been used to make or melt brass. The colour of many crucibles and other materials used in the manufacture of brass (cf Dungworth and White 2007; Freestone 1988) often have a pale blue or purple colour due to the presence of zinc-aluminium spinels (gahnite) which contain small amounts of Fe²⁺ (see below).

The processes which lead to the formation of the amorphous copper alloy lumps are not certain but it is likely that most represent molten brass which was spilt during manufacture and or casting. The interpretation of any chemical analysis of such spillages needs to be approached with caution, however, due to the well-known volatility of zinc in brasses. When a brass is melted a proportion of its zinc tends to be lost as vapour. Any brass spillages, therefore, may contain less zinc than the brass that was manufactured.



Figure 1. Crucibles 16 (left) and 17 (right)



Figure 2. Amorphous copper alloy lump sample 4

METHODS

All samples were analysed to determine their chemical composition. The samples were cut using a diamond saw and embedded in epoxy resin. The samples were polished and coated in carbon to allow examination with a scanning electron microscope (a FEI Inspect F) which was operated at 25kV and 1nA using both secondary and back scattered electron detectors. The chemical composition was determined using an Oxford Instruments X-ACT SDD detector. Four separate areas (typically 0.5mm by 0.5mm) from each sample were analysed, and the results reported below represent the average values. The results were calibrated against a series of certified reference materials which allowed the determination of accuracy (Table 1). In addition, repeat analyses (of certified reference materials) provided data which was used to estimate precision.

Table 1. Accuracy, precision and detection limits (wt%)

Sample	S	Fe	Ni	Cu	Zn	As	Sn	Pb
accuracy	0.05	0.01	0.05	1.5	0.1	0.05	0.3	1.1
precision	0.05	0.03	0.05	0.2	0.05	0.05	0.1	0.1
detection limit	0.05	0.05	0.05	NA	0.1	0.1	0.1	0.2

Samples from each crucible were powdered and analysed using a Phillips 1840 X-Ray Diffractometer to determine the crystalline phases present.

RESULTS

The metallic lumps

All of the copper alloy lumps have dendritic microstructures indicating that they have been cast but have not been worked. The microstructures are consistent with the lumps having been formed due to accidental spillage of material during manufacture. Most of copper alloy lumps are brasses but a range of chemical compositions are present (Table 2). Sample 11 is clearly very different to the other samples — it contains very low levels of alloying elements (zinc, tin and lead) but does contain high concentrations of sulphur. This sample appears to be a form of crude copper. It is unlikely to represent the copper used as a raw material in brass manufacture as it contains much more sulphur than the finished brass. Of the remaining samples, only 9 and 10 can be regarded as not being brasses. Sample 9 contains less than 6wt% zinc, 2wt% lead and no detectable tin; in terms of its colour and physical properties this would be an impure copper. The low zinc content of this sample could simply be the result of the volatility of zinc. If a high-quality cementation brass with c28wt% zinc were kept molten for long enough, the zinc content would diminish. Alternatively, this low-zinc alloy might have been deliberate. Mitchiner *et al.* (1985, 151) report that 44% of the mid-seventeenth-century tokens that they analysed

were low zinc brasses with $5.0\pm 0.6\text{wt}\%$ zinc (that is similar to sample 9). Sample 10 contains 11wt% zinc and 5wt% lead — a composition which has no exact parallels among contemporary analysed metals (Dungworth 2005; Mitchiner *et al* 1985).

The remaining 13 samples have zinc contents (15–30wt%) that are consistent with the manufacture of cementation brass. The highest zinc contents (28–30wt% zinc) represent highly efficient cementation brass production. The presence of several samples with zinc contents slightly in excess of 28wt% might indicate the use of small particles of (perhaps even granulated) copper rather than copper lumps.

Table 2. Chemical composition of amorphous copper alloy lumps (wt%)

Sample	S	Fe	Ni	Cu	Zn	As	Sn	Pb
1	<0.05	0.39	0.16	72.5	19.1	0.22	2.6	6.2
2	0.10	0.07	<0.05	81.6	15.2	<0.10	<0.1	2.7
3	<0.05	0.09	<0.05	75.1	21.0	0.08	0.4	3.6
4	0.05	0.34	0.15	68.9	25.4	0.22	1.8	3.1
5	<0.05	0.30	0.06	68.8	28.1	<0.10	0.4	1.7
6	0.07	0.22	0.19	75.9	18.8	0.28	1.9	1.6
7	<0.05	0.23	<0.05	73.0	24.4	0.12	<0.1	1.5
8	<0.05	0.05	<0.05	69.2	28.2	0.13	0.2	1.6
9	0.09	<0.05	<0.05	91.3	5.8	<0.10	<0.1	2.0
10	0.08	0.14	<0.05	84.2	11.0	<0.10	0.3	5.3
11	0.64	<0.05	0.05	95.5	1.0	<0.10	0.2	0.7
12	0.07	0.10	0.09	75.6	21.1	0.10	0.4	2.1
13	<0.05	<0.05	<0.05	71.4	26.0	0.11	<0.1	2.2
14	<0.05	0.06	0.20	67.1	29.6	0.15	0.1	3.2
15	0.09	0.22	0.19	75.8	18.5	0.28	2.0	2.0

The lead and tin contents of the Taynton brasses are frequently higher than contemporary analysed artefacts (Cople 1995; Dungworth 2005; Mitchiner *et al* 1985). Four of the Taynton brasses (samples 1, 4, 6 and 15) are distinguished by having more than 1wt% tin, while all of the others have less than 0.5wt%. These brasses also tend to have higher concentrations of nickel, arsenic and iron. While the trace element differences might indicate the use of different copper sources, the difference in tin content is more likely to be a result of deliberate alloying.

The brasses all contain small amounts of lead which is present as a separate phase along grain boundaries. It has been suggested that some lead in cementation brass might derive from the calamine used as both zinc and lead ores are often found together (Cople 1995, 223). The examination of the crucibles (see below) shows that these had absorbed zinc but not lead suggesting that the calamine used contained little or no lead. Instead the lead would appear to have been a deliberate addition. At high concentrations, lead can cause copper alloys to crack during hot working but at lower concentrations it can improve machinability (CDA nd) and it is often found in drawn brass wire (Cople 1995).

The crucibles

The two crucibles are made of refractory clay, have been fired to high temperatures and have been contaminated with zinc (Table 3). The two crucibles are completely different colours and suggest that I6 was unused while I7 had been used. The colour difference, however, is not reflected in their bulk chemical compositions which are very similar. The blue colour of crucible I7 appears to be due to the presence of substantial amounts of the mineral gahnite (ZnAl_2O_4 , see Figure 3). The presence of gahnite is confirmed by the XRD analysis (Table 4). Pure gahnite is a green mineral and the blue colour of some gahnite (including that in zinc-smelting crucibles and other waste) is probably due to the presence of small amounts of Fe^{2+} (cf Batchelor and Kinnaird 1984).

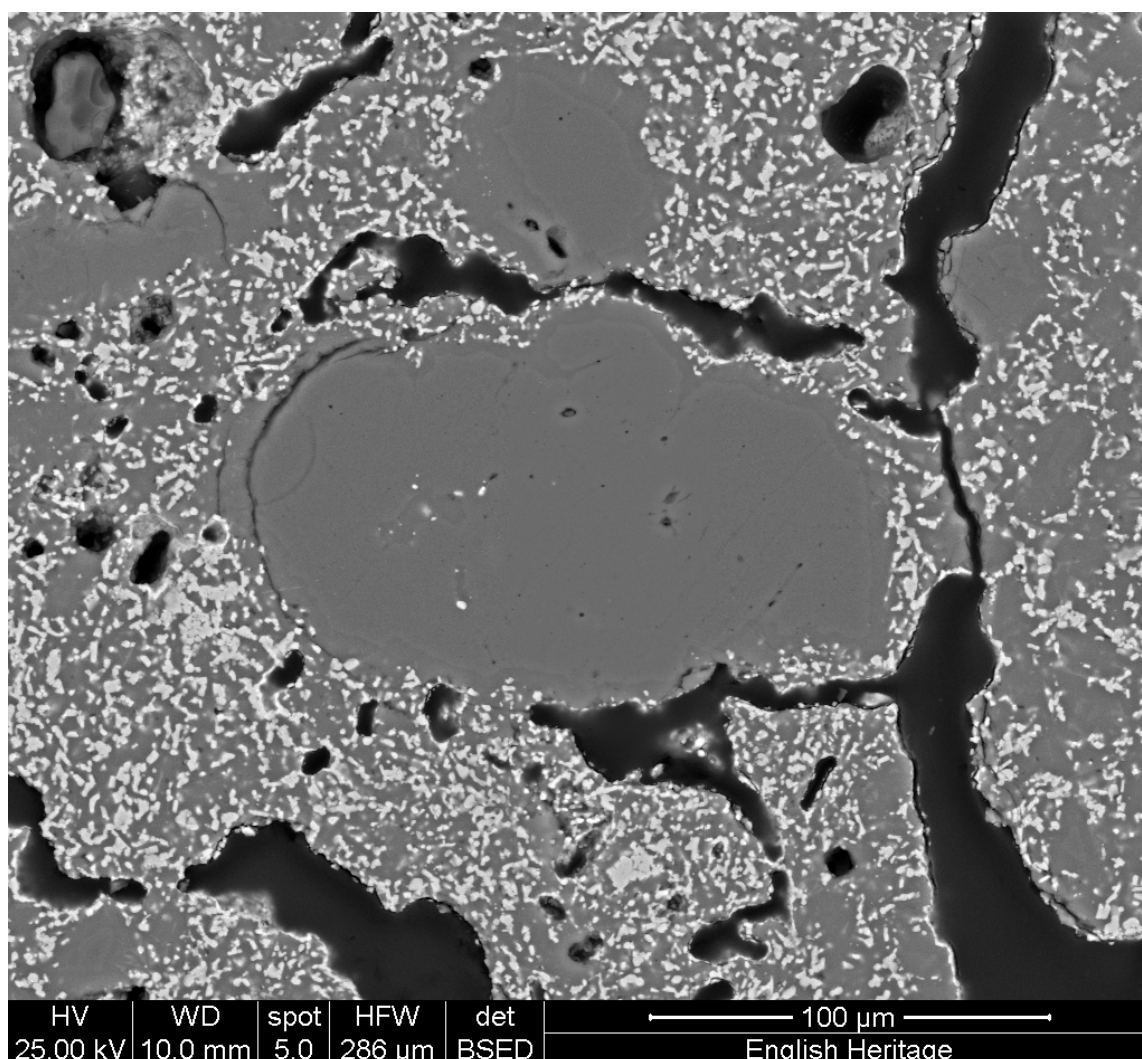


Figure 3. Scanning Electron Microscope image (back-scattered electron detector) of sample I7. The small bright euhedral crystals are gahnite (ZnAl_2O_4) while the larger, irregular, mid-grey crystals are silica polymorphs (XRD analysis indicates the presence of both quartz and tridymite)

Table 3. Chemical composition of the crucibles (wt%)

	zone	Na ₂ O	MgO	Al ₂ O ₃	SiO ₂	K ₂ O	CaO	TiO ₂	FeO	ZnO	BaO
16	bulk	<1.0	0.45	15.7	65.2	1.34	0.20	1.27	1.25	14.6	<0.2
16	grog	<1.0	0.34	18.3	56.6	0.86	0.16	0.94	1.11	21.7	<0.2
16	matrix	<1.0	0.44	15.1	72.5	1.66	0.26	1.00	1.10	7.9	<0.2
16	gahnite	<1.0	1.91	53.1	1.43	<0.1	0.10	0.41	7.76	35.2	<0.2
16	roeperite	<1.0	3.72	0.24	28.7	<0.1	<0.1	<0.1	6.66	60.3	<0.2
17	bulk	<1.0	0.43	14.0	65.4	0.88	0.45	0.99	1.68	16.1	<0.2
17	gahnite	<1.0	0.91	53.0	1.85	<0.1	0.15	1.06	2.27	40.6	<0.2
17	inner surface	<1.0	1.83	15.9	56.6	6.0	7.5	0.81	0.95	3.5	5.9
17	outer surface	<1.0	1.75	23.5	52.6	3.4	4.0	0.77	7.60	4.3	1.3

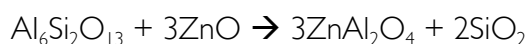
Table 4. XRD results

	Minerals
16	Quartz, Tridymite, Gahnite, Ropperite
17	Quartz, Tridymite, Gahnite

Analyses of individual gahnite crystals in sample 17 (SEM spot mode) indicates a composition close to ZnAl₂O₄ but with small amounts of iron (Table 3). The blue colour of gahnite associated with brass working suggests that the furnace conditions were strongly reducing (otherwise iron would not be present as Fe²⁺).

Crucible 16 contains almost as much zinc as crucible 17 but it is a buff colour rather than pale blue. It does, however, contain small pale blue inclusions. Examination of the microstructure of crucible 16 indicates that these inclusions have microstructures very similar to crucible 17. These inclusions are rich in zinc and contain abundant gahnite crystals. The remaining parts of crucible 16 contain less zinc and very few gahnite crystals. A significant proportion of the zinc present in crucible 16 is present in roeperite ((Zn,Fe)₂SiO₄) crystals which lacks the blue colour of gahnite. It is likely that crucible 16 was manufactured using a proportion of grog, that is ground up fragments of old crucibles. The firing of crucible 16 would probably lead to the diffusion of zinc from the grog to the remaining portions of the crucible fabric, and the transformation of some gahnite into roeperite). The complete absence of lead in the crucibles suggests that the calamine used did not contain significant amounts of lead.

It is likely that the raw clay for the crucible contained kaolinite (Al₂Si₂O₅(OH)₄) which during firing would dehydrate to form metakaolin (Al₂Si₂O₇). Firing to the very high temperature needed for use as crucibles would then lead to the formation of mullite (Al₆Si₂O₁₃) and cristobalite (SiO₂) (Dungworth 2003; Eramo 2005; Martín-Torres *et al* 2006). During use as brass-making crucibles, however, the high concentration of zinc probably led to the decomposition of the mullite and the formation of gahnite and silica (Vorob'eva *et al* 1971).



Both the interior and exterior surfaces of crucible 17 are vitrified surfaces. The interior surface contained numerous metallic droplets that are discussed below. The exterior vitrified surface shows significant elevation of iron oxide and alumina concentrations which probably indicates the use of coal rather than wood fuel (Dungworth 2003; 2010). The interior surface shows the elevation of a range of oxides (especially barium oxide) that may represent gangue minerals associated with the use of zinc ores in the production of cementation brass. It is puzzling that the interior vitrified surface of crucible 17 contains a lower concentration of zinc (3.5%) than the ceramic fabric (16.1%). It might be argued that the ceramic fabric was porous which would enable easy diffusion of zinc oxide vapour while the limited porosity of the interior vitrified surface would inhibit diffusion, however, zinc oxide from the charge inside the crucible could only enter the crucible fabric via the interior vitrified surface.

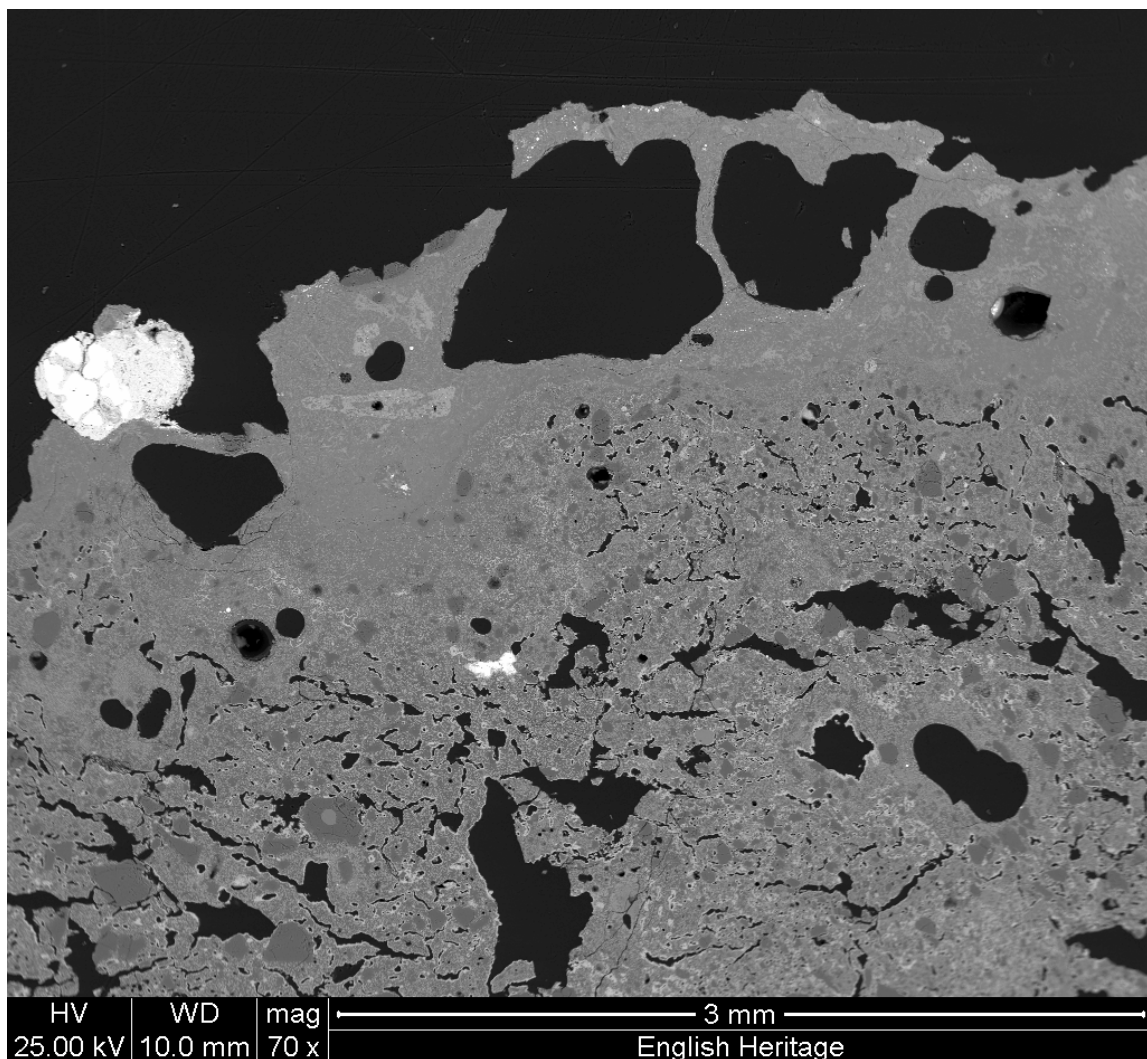


Figure 4. Scanning Electron Microscope image (back-scattered electron detector) of the interior vitrified surface of crucible 17. The bright region on the left is a copper alloy droplet (17.5 in Table 5)

Assuming that the zinc derives entirely from the manufacture and/or melting of brass, both crucibles are alumina and silica rich, with low levels of potassium, iron and other elements. The crucibles would be extremely refractory; leaving aside the zinc content, they have chemical compositions which are similar to contemporary glass-melting crucibles and would be capable of withstanding temperatures up to 1300°C (Dungworth 2003).

Crucible 17 contained numerous metallic droplets trapped within the ceramic fabric and within the inner vitrified surface (Figure 4) while crucible 16 contained no metallic droplets. The chemical composition of the metallic droplets in crucible 17 (Table 5) vary from almost pure copper to brass (the former are restricted to the inner vitrified surface) while the latter have often penetrated pores within the ceramic fabric). Varying proportions of lead are present in these droplets but tin is absent (or present at very low concentrations). Lead is generally absent (or at very low concentrations) in the droplets which contain low levels of zinc but is present in all of the brass droplets. The brass droplets mostly contain zinc concentrations close to the maximum that can be consistently reached using a cementation process. Droplet 17.12, however, contains a very high concentration of zinc, although such concentrations have been achieved on the microscopic scale during modern cementation experiments (Newbury *et al*/2005) and should not necessarily be interpreted as evidence for alloying using metallic zinc.

Table 5. Chemical composition of copper alloy droplets within crucible 17 (wt%)

Droplet	S	Fe	Ni	Cu	Zn	As	Sn	Pb
17.1	<0.05	0.28	0.08	69.6	25.4	0.12	0.3	5.7
17.2	<0.05	0.30	0.07	68.7	24.9	0.10	0.4	8.6
17.3	<0.05	0.34	<0.05	70.0	25.1	0.12	0.3	5.3
17.4	<0.05	0.30	0.05	70.0	25.2	0.13	0.3	5.4
17.5	<0.05	0.22	0.10	95.3	2.1	<0.10	<0.1	0.3
17.6	<0.05	0.62	<0.05	66.5	29.4	0.15	0.2	3.4
17.7	<0.05	0.60	<0.05	66.5	29.4	0.14	0.1	3.7
17.8	<0.05	1.43	<0.05	86.9	9.2	<0.10	0.1	0.3
17.9	<0.05	0.78	0.05	93.6	3.2	<0.10	<0.1	0.3
17.10	<0.05	0.24	<0.05	95.6	1.9	<0.10	<0.1	<0.2
17.11	0.05	0.78	<0.05	89.7	7.3	<0.10	<0.1	0.3
17.12	<0.05	0.46	0.05	60.5	36.7	0.11	<0.1	1.8
17.13	<0.05	0.68	0.12	72.3	23.3	0.11	0.4	3.1

It can be suggested that the copper alloy droplets in crucible 17 formed during the production of cementation brass and/or during the melting of brass. The low-zinc droplets could represent copper charged for cementation brass manufacture but which had only absorbed a small proportion of zinc. Alternatively the low zinc droplets could represent brass droplets which had lost a proportion of their zinc during melting due to the volatility of zinc. On balance the former model seems the more likely as any loss of zinc by volatilisation should have been accompanied by a loss of lead and there is no correlation between lead and zinc contents. In addition the composition of the inner vitrified surface

of the crucible (Table 3) suggests that the crucible was used for the manufacture of brass by cementation. The absence of lead from the crucible fabric or its vitrified surfaces suggests that lead was not present (as an impurity) in the zinc ore used in the cementation process. It is possible that lead was deliberately added to molten brass after the cementation process while the brass was molten (as discussed above a small proportion of lead may have added to make the brass more suitable for wire drawing).

CONCLUSIONS

The examination of the two crucibles from Taynton shows that one (17) was used in an environment where significant amounts of zinc had diffused into the crucible fabric and lead to the formation of gahnite ($ZnAl_2O_4$). The vitrified interior surface of this crucible contains a range of oxides (in particular magnesia, lime and barium oxide) that are compatible with the cementation manufacture of brass (these oxides being common gangue minerals associated with smithsonite zinc ores from the Mendips). In addition the crucible contained numerous small droplets of copper and brass. The second crucible (16) was made using a proportion of grog derived from old crucibles but it had not been used extensively (if at all) for the manufacture or melting of brass. The copper alloy droplets in crucible 17 and the separate amorphous copper alloy lumps have compositions that are typical of cementation brass production with zinc contents largely between 20 and 28wt%. Most of these brasses contain very low levels of tin but significant levels of lead. The lead appears not to be an accidental bi-product of using a zinc ore that also contained lead but instead was deliberately added to the cementation brass prior to final casting.

While the examination of a small sample of brass manufacturing debris from Taynton has provided the first material evidence for brass manufacture in the 17th century, there are several aspects of the material which remain uncertain. It is not clear why a proportion of the amorphous brass lumps are made from a copper which contains appreciable concentrations of important trace elements (especially nickel and arsenic). These trace elements are not as abundant in the metal droplets in the crucible. This discrepancy may simply have arisen because only one used crucible was available for study. The examination of a larger number of crucibles might show that copper with a range of trace elements (from several sources) was exploited. The copper and brass droplets in the crucible generally contain higher concentrations of iron (0.54wt%) than the amorphous lumps (0.15wt%). The reasons for this difference are not clear. Similar uncertainty surrounds the observation that the ceramic fabric of the crucible has absorbed a greater proportion of zinc than its interior vitrified surface. While these questions could be explored through further investigation of the Taynton samples, a more effective approach would be through stratified material from archaeological excavations.

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