
Trials and errors in search of mineral wealth: metallurgical experiments in early colonial Jamestown

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Introduction

There is little doubt that the main motivation that launched British explorers to the conquest and colonization of North America was the hope of finding and exploiting plentiful natural resources, together with the discovery of a water route to the East. Having witnessed the success of the 16th-century Spanish enterprise in the New World, British entrepreneurs could only hope that their American adventure would turn out to be just as profitable. In 1607, with their confidence boosted by previous descriptions of a fertile land, and a royal charter in hand, over one hundred men arrived at the Chesapeake Bay in Virginia, where they founded Jamestown - the first permanent English settlement in the New World.

In the ensuing months and years, famine, disease and continuing attacks by the indigenous population caused numerous casualties amongst the Englishmen who, also fearing Spanish attacks, had to build a fort and seek strategies for a peaceful co-existence and a productive settlement. In spite of their efforts, the colonists failed to accomplish their ambitions and struggled to manage the situation. In 1624, the English king, James I, revoked the charter and Virginia became a crown colony. Jamestown remained the capital of Virginia until the major fire of 1698, when this settlement was abandoned and the capital moved to Williamsburg (Kelso and Straube 2004)

Recent historical, archaeological and analytical work has provided convincing evidence that one of the major aspirations of the early settlers sent to Jamestown was, in particular, the exploitation of mineral wealth for metallurgical operations. Besides gold, it appears that the Virginia Company was also interested in locating zinc ores, scarce and poor in England, which could be used for the production of brass (Hudgins 2005, and this volume). Historical documents show that some of the main entrepreneurs behind the Jamestown colonization were the Society of Mines Royal and the Society of Mineral and Battery Works, who needed zinc to supply the growing demand for brass and to find an outlet for their large stocks of copper. Furthermore, thousands of off-cuts of copper sheet found in Jamestown

have been shown to constitute English scrap metal, which could have been brought for exchange with the indigenous populations but which also could have been used in experiments with zinc ores to produce brass (Hudgins 2005, and this volume).

A crucial piece of this emerging picture is contained in the industrial metallurgical remains excavated in Jamestown, mostly consisting of crucibles and some melted waste, together with a few pieces of distillation equipment (Kelso and Straube 2004: 157-161). Since this would be the equipment used in the trials of American minerals, it was hoped that their analyses could shed light on the actual experiments carried out in the first years following the colonists' arrival. The fact that most of these instruments appear unused is a strong indication that the hopeful metallurgists failed to find whatever they were after, and thus most tools were abandoned before putting them to any productive use. However, what can be said about the origins and quality of these crucibles, and what exactly were they used for?

This paper presents the results of preliminary analytical work carried out on a small number of crucible fragments from the site. Their study allows a detailed insight into some of the high-temperature metallurgical processes carried out in Jamestown. On this basis, it is possible to assess the skill and scientific knowledge of those conducting them, thus contributing to the history of early science in America, but also facilitating a reassessment of the often assumed laziness and incompetence of the Jamestown settlers (cf. Morgan 1975). The limited number of samples available for this study, however, allows only a very sketchy picture to be drawn; further work is needed to broaden the database and enable more meaningful interpretations

Methodology

Three crucibles were studied as standard polished cross-sections removed from the ceramic sherds, mounted in epoxy resin, ground and polished down to a 1 μ m finish to expose the section as a flat surface including both ceramic fabric and any residues adhering to it. These specimens were first examined in a reflected light optical microscope, and then analyzed using a Philips XL30 scanning electron microscope, with an attached energy-dispersive X-ray spectrometer from Oxford Instruments (SEM-EDS). All chemical compositions reported are from SEM-EDS, combined with oxygen by stoichiometry where appropriate, and normalized to 100 percent by weight (wt%) to correct for ceramic porosity and fluctuation of beam intensity. All the

micrographs shown are SEM backscattered electron images.

Crucible fabrics

Before addressing the residues within the vessels, it is worth dealing with the crucibles themselves as ceramics (Fig. 1). All three crucibles appear to have been made of a very lean clay, probably refined prior to tempering. The paste is typically tempered with spheroidal quartz (20 to 40% by volume, in a grain size of 200 to 500 μm) and very few other minerals are present. This is a technically significant feature, as quartz grains, unlike other minerals, would have a specific behaviour under high temperatures resulting in enhanced toughness and thermal shock resistance for the vessels (Freestone 1989; Tite et al. 2001).

In one of the vessels (JR124F/C1), some roundish argillaceous inclusions were also identified, about 1 mm large, compositionally identical to the ceramic matrix and also containing quartz grains. They are generally surrounded by a shrinkage void, and their presence clearly causes a random crack distribution within the paste, compared to the parallel elongate cracks of the other crucibles. These inclusions are interpreted as grog fragments, i.e. fragments of old crucibles crushed and added as temper, but the high vitrification of the paste makes it difficult to confirm this point.

The chemical compositions of the ceramic matrices show outstanding alumina levels (above 35 wt%, contrasting with contemporary Bavarian crucibles at about 30 wt%, or more common crucibles from other origins with alumina concentrations below 25 wt%; cf. Martínón-Torres and Rehren in press). Conversely, alkali, earth alkali and iron oxide concentrations are very low. (Table 1) This composition indicates an exceptional thermal refractoriness, much higher than that of most contemporary crucibles.

	Na ₂ O	MgO	Al ₂ O ₃	SiO ₂	P ₂ O ₅	K ₂ O	CaO	TiO ₂	FeO
JR 1024/C C 3	0.2	0.4	36.8	56.4	0.3	1.5	0.2	1.9	2.3
JR 124F/C 1	0.2	0.5	36.8	57.1	0.2	1.1	0.4	2.0	1.8
A 631/C C 2	0.1	0.5	35.6	57.8	0.4	1.2	0.4	2.2	1.7

Table 1. Chemical composition of the ceramic matrices of the three crucibles analysed. Results are averages of several area measurements per crucible section, normalised to 100 wt%.

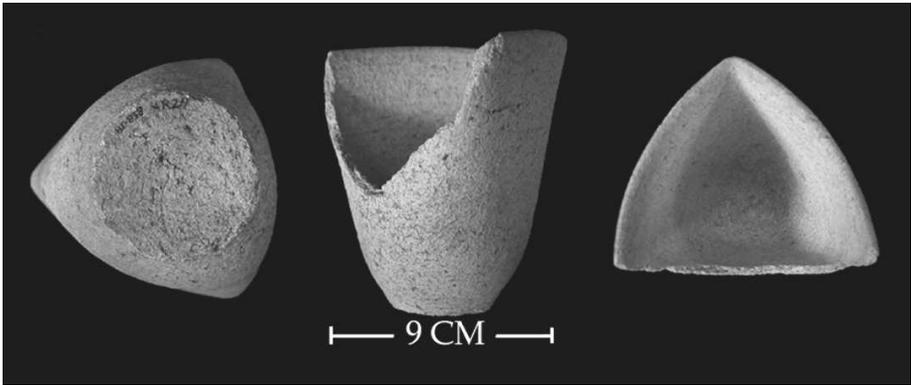


Figure 1. Example of a Hessian crucible recovered in Jamestown. The triangular mouth is typical of the period, as an easy technical solution creating three pouring spouts, but beaker-shaped Hessian vessels are also present in the assemblage. Photo by C. Hudgins

Altogether, these features allow us to track the origins of these crucibles to the German region of Hesse. Hessian crucibles were the most famous in the post medieval world (Cotter 1992; Stephan 1995). Recent research has identified Hessian vessels in a large number of sites (Martinón-Torres and Rehren in press), thus documenting the earliest international trade of scientific instruments (Martinón-Torres 2007). Based on comparative scientific analyses, it has been demonstrated that these crucibles possessed an outstanding technical quality, which justifies their historical reputation (Martinón-Torres et al. 2006). The stamp found in a crucible base in Jamestown had already indicated a German provenance (Kelso and Straube 2004: 190), but these analyses show that the unstamped crucibles also originated from Hesse. This seems logical, considering the involvement of German metallurgists in Elizabethan metallurgy in general, and the American enterprise in particular (Hicks, this volume), but it does also illustrate the high hopes of the metallurgists that brought to Jamestown the best crucibles available. Were the operators as proficient as the vessels they used?

Crucible 1

Sample JE 1024/CC3 is the rim sherd of a relatively large beaker-shaped crucible, with a diameter in excess of 100 mm, and a wall thickness of about 10 mm. The vessel shows signs of intense use, such as a high degree of vitrification of the matrix, extensive shattering of the quartz temper following thermal stress, and deep penetration of

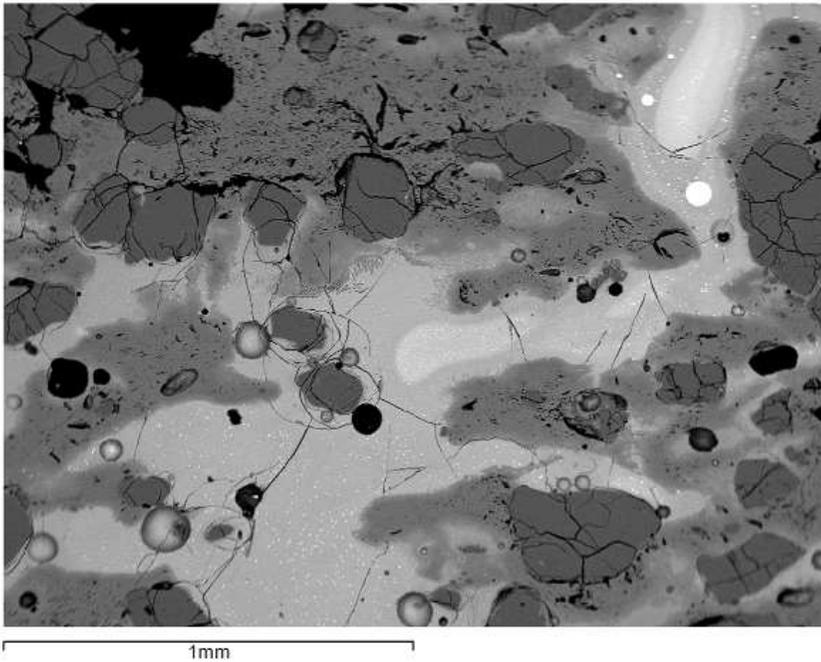


Figure 2. Cross section through the crucible JR 1024/CC3, showing the shattered quartz grains (dark grey) and the penetration of the slag (bright grey) through crucible cracks. Note the bright globules of copper metal, especially in the top right area of the slag.

slag into the fabric. It is this latter slag, also present as a thin film on the inside surface, that carries the most metallurgically relevant information. Most noticeable is its widespread contamination with copper, both as metallic prills and as dendritic copper oxide crystals (cuprite) precipitating from the melt (Figs. 2-3). Alloying metals such as tin, zinc and lead, however, are absent or only present in rather low concentrations. The slag composition itself is dominated by silica (>40 wt%), lime (>20 wt%), copper oxide (c. 15 wt%), alumina (c. 10 wt%) and minor amounts of iron oxide (c. 5 wt%) (Table 2). The alkali oxides are here present at less than 1 wt%. Within this glassy slag, two discrete phases are identified: one being the before-mentioned cuprite, the other clusters or aggregates of an alkali oxide alumina silicate of the approximate formula $(K,Na,Ca)AlSi_2O_6$, probably structurally related to the mineral leucite, $KAlSi_2O_6$ (Figs. 2-3). The occurrence of this phase is a result of a relatively high alumina and alkali oxide content, although the glassy matrix analyses indicate that the overall composition was richer in alumina than alkali oxides (Table 2).

In terms of metallurgical activity, one can assume that the crucible was used for melting and casting of copper, probably

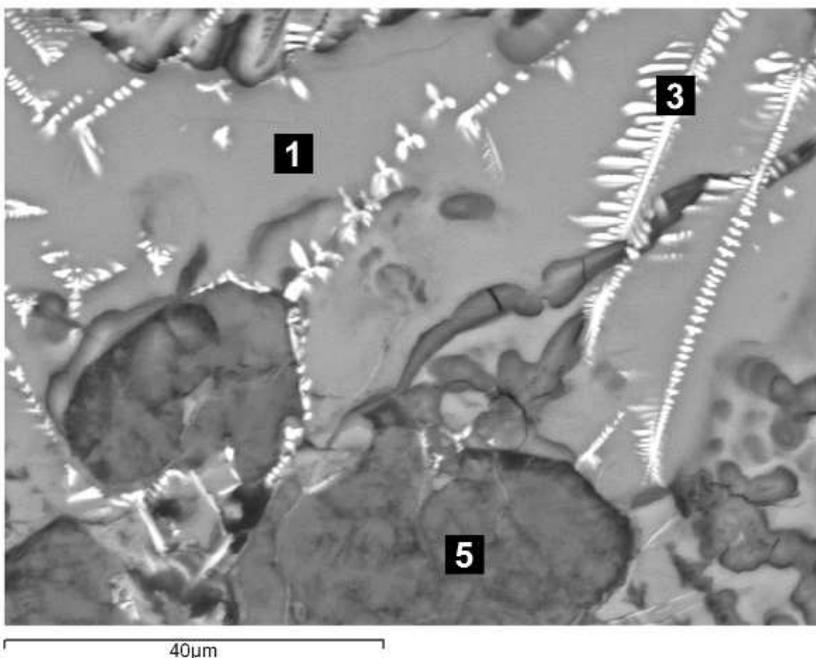


Figure 3. Detail of the slag on the surface of crucible JR 1024/CC3, showing dendrites of cuprite (brighy grey) and crystals of the leucite family (dark grey) in a copper- and calcium oxide-rich glassy matrix. SEM-EDS results for the areas marked are given in Table 2.

unalloyed or only weakly alloyed. This would have required a relatively high temperature, of around 1100 °C, which may have resulted in a high rate of fuel combustion. The residual charcoal ash would explain the elevated lime and alumina levels in the slag glass. It is not possible to ascertain at present whether the copper melted in this crucible would be part of the British scrap copper abundantly found in Jamestown. However, it appears reasonable that some of this copper could have been melted to meet ordinary needs at the settlement.

	Na ₂ O	MgO	Al ₂ O ₃	SiO ₂	P ₂ O ₅	K ₂ O	CaO	TiO ₂	MnO	FeO	CuO	ZnO	As ₂ O ₃	PbO
1*	-	2.1	9.0	44.6	0.9	0.8	21.6	1.5	0.5	4.7	14.7	-	-	-
2	-	2.2	9.2	43.7	0.99	0.8	21.8	1.0	0.6	4.7	15.1	-	-	-
3*	-	1.3	6.6	27.7	0.6	0.8	10.1	0.5	0.2	2.0	50.3	-	-	-
4	-	0.7	3.9	17.9	0.2	0.7	5.9	-	-	1.3	69.0	0.4	-	-
5*	6.0	0.2	21.8	54.0	3.8	3.5	1.8	0.3	-	3.8	2.6	-	0.2	2.1
6	6.6	-	23.9	57.8	2.5	3.5	0.3	-	-	1.5	1.6	-	-	2.3

Table 2. SEM-EDS analytical results for the slag penetrating crucible JR 1024/CC3. Analyses highlighted with an asterisk refer to phases marked in Figure 3, while the rest were performed on similar phases in other regions of the sample.

Crucible 2

The residues in crucible JR 124F/C1 speak of a somewhat more complex story. This is a small body sherd, with a thickness of 7 mm. The section through this fragment exposed several of the green prills contained in this crucible. They are completely corroded, but the largest one preserves an as-cast dendritic alloy structure in the corrosion products. Area analyses of various parts of this prill reveal a predominance of tin oxide (50-75 wt%) and 10-30 wt% copper oxide; the balance being silica, lead oxide, iron oxide and phosphorous oxide. The immediate contact area between the prill and the ceramic consists of a layer of cuprite and copper chloride, probably re-deposited material from the prill proper. The present microstructure indicates a major loss of copper metal due to corrosion, while the tin content is less affected by this and thus appears relatively enriched. This is a widely known phenomenon and prevents any reliable identification of the original alloy composition from the analysis of corroded prills; the only possible statement is that the alloy originally must have been a tin bronze with a certain amount of lead in it.

In itself, this residue would suggest that the crucible was used for melting a leaded bronze. The melting and casting activity is also indicated by the loose metal prill JR 69E/224, also analysed by us (Fig. 4). This has a metallographic structure very similar to the corroded prills in the crucible, with an as-cast dendritic pattern of copper-rich alpha phase, interstitial tin-rich alpha-delta eutectic, and numerous isolated droplets of metallic lead and copper sulphide. Area analyses for this prill gave an average tin content of 18 wt%, with just under 2 wt% lead and the balance being copper. The area proportion of the eutectic in this sample is very similar to the equivalent material in the corroded prills from the crucible, suggesting that the overall composition of the alloy worked in the crucible could have been very similar to the composition of the larger prill. Like the copper in the crucible discussed above, bronze could have been needed for many implements in the settlement. It is worth noting, however, that a tin content of just under 20 wt% is relatively high for ordinary bronze; remarkable also is the large amount of sulphide inclusions present in this prill.

There is a further complication in the interpretation of this crucible, manifest in the continuous black film covering the inner surface (Fig. 5). This is characterised by high amounts of zinc and iron oxide, of around 20 wt% each, as well as a significant lime enrichment (Table 3). The contrast in composition between this slag layer and the bronze prills is remarkable, and may indicate the consecutive use of the same crucible for two different operations, resulting in two disconnected use

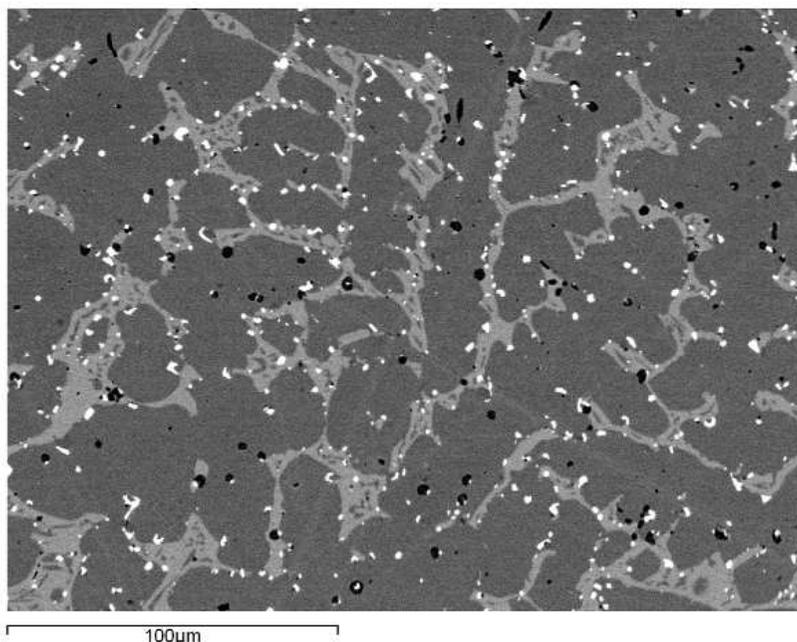


Figure 4. Microstructure of the loose bronze prill (JR 69E/224), showing a developed dendritic structure very similar to that of the relictic structures of the corroded prills within crucible JR 124F/C1 (not shown). The bright globules are metallic lead, the dark ones are copper sulphide.

traces: while the bronze prills most likely result from a simple casting operation, it is tempting to suggest that the zinc- and iron-rich material could possibly indicate a brass cementation attempt, involving low-quality zinc ore.

Before the distillation of zinc was introduced into Europe in the eighteenth century, the only way of making brass, both in Europe and the colonies, was the method known as cementation. In this process, copper metal and zinc minerals are heated together with charcoal in a crucible, so that zinc forms as a vapour and is absorbed by the copper metal, thus creating the alloy. The slag forming within these crucibles is usually rich in zinc oxide, together with iron oxide (typical impurity of zinc ores), and lime from the charcoal ash (Martín-Torres and Rehren 2002). For a metallurgist in Jamestown, the only way to test the quality of a potential zinc ore would be to carry out cementation experiments and assess the resulting brass, given the impossibility to smelt zinc alone. This, together with the documented interest of the Virginia Company in locating American zinc for making brass in Europe, reinforces the possibility that the metallurgists at Jamestown had been performing cementation tests using the copper metal brought from England (Hudgins 2005, and this volume). Thus the present

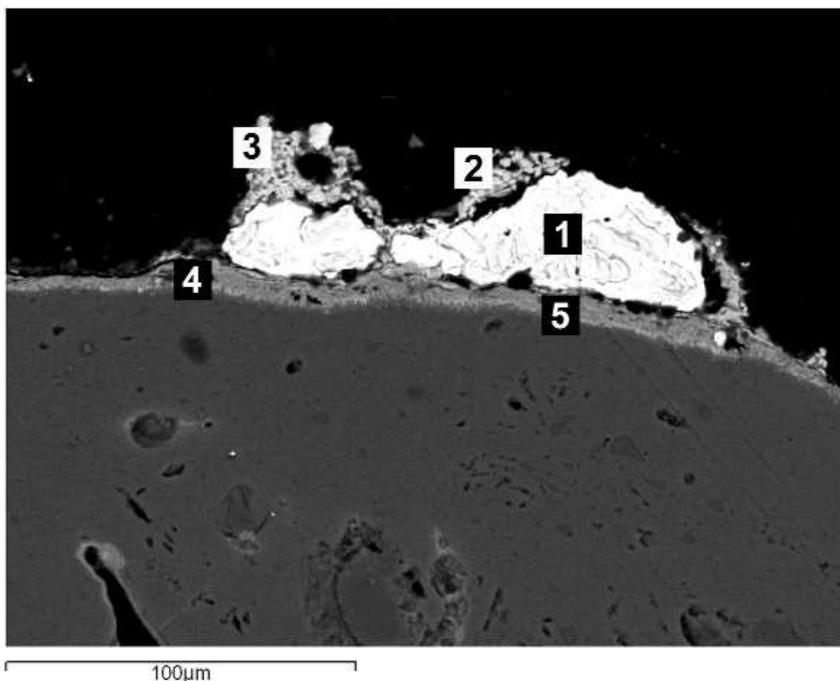


Figure 5. Detail of the zinc- and iron-oxide enrichment on the inner surface of the crucible (mid grey layer), where some corroded metal prills (bright) are sitting. SEM-EDS analytical results for the areas marked are given in Table 3.

crucible could constitute the first direct evidence of such practice, and the low quality of the zinc ore (and correspondingly high iron oxide levels), would be consistent with the fact that no good zinc ores are known to occur in this region.

More crucible analyses, however, will be needed in the hope of more incontestable evidence. The very limited zinc oxide contamination of the crucible fabric beneath the inner surface (less than 0.1 wt% ZnO on average, with the most contaminated areas reaching only 0.7 wt%) is unusual for brass cementation vessels, even accounting for the chemical refractoriness of the Hessian crucibles, and assuming a poor ore quality. The only possible explanation for this would be that the cementation was carried out with the crucible unlidged, so that zinc oxide escaped with fumes rather than reacting with the ceramic fabric. Yet from a technical viewpoint this would constitute bad practice, as it would also make it more difficult for the copper to absorb zinc (Rehren and Martínón-Torres in press). Finally, also peculiar is the fact that the same vessel would have been used for two such different operations (bronze casting and brass making),

	MgO	Al ₂ O ₃	SiO ₂	P ₂ O ₅	K ₂ O	CaO	TiO ₂	FeO	CuO	ZnO	SnO ₂	PbO
1	-	1.2	2.4	5.6	-	1.9	-	1.0	16.5	2.5	62.3	6.7
2	0.9	2.2	5.8	1.2	-	1.1	0.3	58.7	2.5	23.7	3.5	-
3	1.5	1.3	5.7	1.2	-	2.9	0.5	61.8	3.1	18.3	3.7	-
4	0.8	23.2	30.1	1.3	0.5	7.3	1.3	16.9	-	18.6	-	-
5	0.9	25.0	26.5	0.8	0.2	4.5	1.3	19.2	-	21.6	-	-

Table 3. SEM-EDS analytical results for the section of the inner surface of the crucible JR 124F/C1. The numbers 1-5 correspond to the areas marked in Figure 5.

especially when so many crucibles were available at the site; but this may be explained by the need to save them when the settlers still expected to conduct plenty of ore analyses.

Crucible 3

The third vessel is considerably different to the ones characterised before, both in its appearance and the adhering residues. Unlike the others, A631/CC2 is a very small crucible, with a base diameter of only 15 mm and a wall thickness of 5 mm. Although incomplete, its height is unlikely to have much exceeded the preserved 30 mm. The inner surface of this crucible is covered by a thin dark brown to black film, with a larger lump of glassy shiny slag preserved near the upper end (Figs. 6-7). Analysis of this material gave silica and iron oxide as main components, totalling 80 to 90 wt%, with soda and potash between 3 and 10 wt% each, and under 2 wt% alumina. Phosphorous oxide, chlorine and lime are each at or below 1 wt% (Table 4). The conspicuous absence of copper from this list rules out any relationship to copper-based metallurgy, while the presence of nickel oxide in some of the more iron-rich phases is remarkable.

The glassy matrix has almost 1.5 times more silica than iron oxide (45 and 30-35 wt%, respectively), and 17 wt% combined alkali oxides; however, in this relatively homogenous matrix float well-defined clusters and aggregates rich in free iron oxide. These aggregates are predominantly composed of haematite, Fe₂O₃, as indicated by their crystal shape and the bright red internal reflections. Discrete particles can have up to 15 wt% nickel oxide; these are probably nickeliferous magnetite, Fe₃O₄ (Fig. 7, Table 4). The co-existence of magnetite and haematite indicates highly oxidising conditions.

There is no published precedent for this microstructure that would guide in its interpretation. A connection to iron metallurgy is unlikely

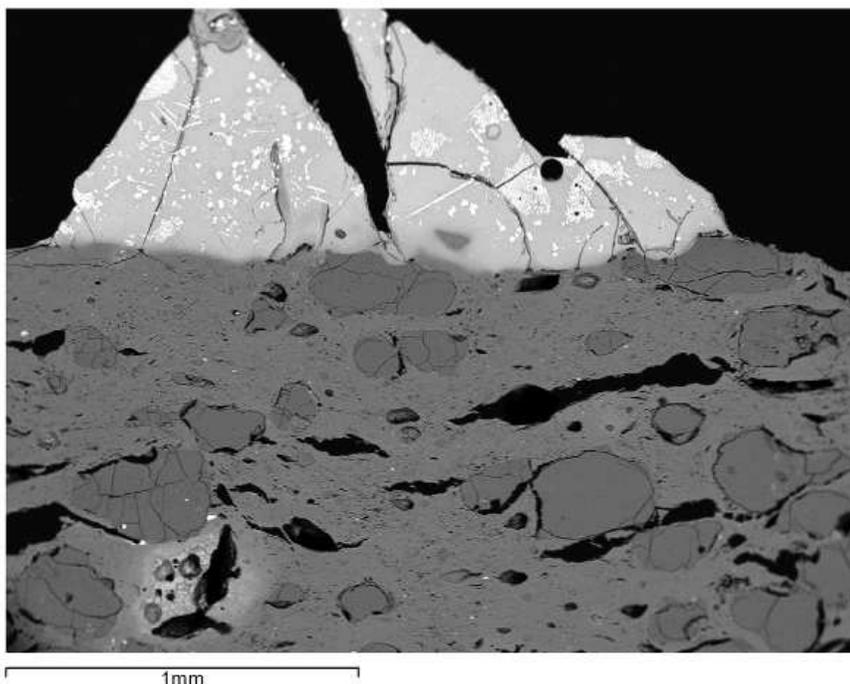


Figure 6. Section through the crucible A631/CC2, showing the quartz-tempered fabric (bottom) and slag remains adhering to the inner surface.

for a number of reasons: firstly, although the bulk chemical composition is somewhat similar to early modern iron slag, it is far too oxidised to constitute iron smelting slag; secondly, the use of a crucible is atypical for iron metallurgy in this cultural context, when crucible steel making was apparently restricted to Central and South Asia but unknown in Europe (Rehren and Papachristou 2003); and thirdly, the small size of the crucible is more indicative of a precious metal than a bulk material such as iron or steel. The processing of base metals, such as copper, tin or lead, is similarly unlikely: under the evident oxidising conditions these would have left clearly identifiable remains in the slag. A non-metallurgical use, for instance in glass or enamel making or working, is also unlikely, given the iron-rich composition. This leaves a connection to noble metals, i.e. silver and gold, as the most likely scenario, which is further corroborated by the small size of the vessel. The identification of distillation equipment in the assemblage, potentially used for mineral acids to be employed in the parting and refining of gold and silver, would also support this idea. Of the two noble metals, silver appears less likely here, as it is most often associated with copper and/or lead, either by nature or artificially alloyed, and none of these metals are present.

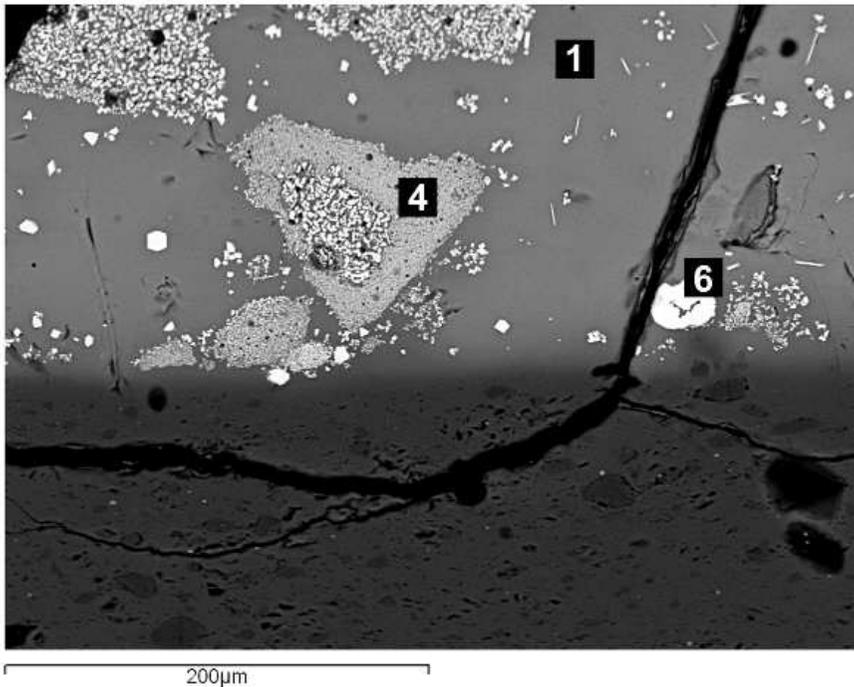


Figure 7. Detail of the interface between the slag and the crucible fabric A631/CC2, showing clusters of magnetite and haematite in a glassy matrix. SEM-EDS analytical results for the areas marked are given in Table 4.

	Na ₂ O	MgO	Al ₂ O ₃	SiO ₂	P ₂ O ₅	Cl	K ₂ O	CaO	TiO ₂	MnO	FeO	NiO
1*	9.8	-	1.4	46.9	1.1	1.0	7.7	0.7	-	-	31.5	-
2	10.7	1.6		41.8	1.3	1.0	7.4	1.1	0.3	-	34.8	
3	8.9	-	1.5	40.7	1.1	0.9	6.3	0.5	-	-	40.1	-
4*	5.5	-	1.5	23.6	0.6	0.9	3.7	-	-	0.5	63.1	0.7
5	7.5	1.0		27.9	0.7	0.9	4.6	0.7	-	0.4	56.4	
6*	-	0.9	-	-	-	-	-	-	-	0.4	83.2	15.6
7	-	0.4	-	0.5	-	-	0.3	-	-	0.6	97.0	1.2
8	0.5	-		0.9	-	-	0.2	-	0.7	-	97.8	
9	-	-		0.4	-	-	0.2	-	-	0.9	98.5	

Table 4. SEM-EDS analytical results for the crucible slag A631/CC2. Analyses highlighted with an asterisk refer to specific phases marked in Figure 7, while the rest were performed on similar phases in other regions of the slag.

How then could this slag relate to gold metallurgy? One possible scenario is the assay of an iron-rich mineral for gold, for instance "fool's gold" pyrite (FeS_2) or pyrrhotine (FeS), which often is nickeliferous. This would have been fluxed with silica, either naturally present in the minerals or added artificially as sand, together with some organic or inorganic salt, under oxidising conditions. A combination of saltpetre (potassium nitrate), rocksalt (NaCl) and/or sodium nitrate, would have been a very effective oxidising agent removing any sulphur from the system as gaseous sulphur dioxide and oxidising the iron up to haematite. This would explain the alkali levels in the slag. Depending on the actual relative proportions of materials present in the charge and the processing parameters, iron sulphide would initially be oxidised to Fe^{2+} , which would at high temperature react with the silica to form a fayalitic melt; with progressing oxidation, the Fe^{3+} would precipitate from this melt as magnetite or even haematite, giving rise to the euhedral crystals seen in the slag.

More importantly, the question remains open as to what should have happened to the presumed gold in this test. We have no indication for the former presence in the crucible charge of lead metal, the typical collector used in the assay of noble metals (Mongiatti et al. in press). We can only assume that the Jamestown assayer would have hoped to gain a noticeable gold-rich regulus straight out of this process, which would have led them to deem the use of a noble metal collector unnecessary. Alternatively, this would indicate a reluctance, or lack of relevant knowledge, to go through the full fire assay procedure including collecting the suspected noble metal in a lead bullion, followed by scorification and cupellation in order to retrieve the gold (Beaudoin and Auger 2004; Mongiatti et al. in press). Both possibilities are feasible under the specific circumstances of the early settlers in Jamestown, who would have been driven by the hopes for the mineral riches of the new continent, and who may not have included among their group an experienced assayer (but see one mentioned below). Again, only analyses of a larger sample may aid in resolving this question.

Discussion and conclusion

The three crucible fragments investigated in this pilot study represent three apparently different metallurgical activities, and they raise a number of interesting questions that should be addressed through further analyses. JE 1024/CC3 and JR 124F/C1 were probably used to melt and cast copper and bronze, respectively; however, there is the possibility that the latter had also been used for a different

activity, brass melting or even cementation. This would confirm the hypothesis that the Jamestown settlers were not only after noble metals, but also seeking zinc ores.

The third crucible, A631/CC2, is the most technically peculiar. Its slag composition is unique, and possibly indicates an attempt to assay iron-rich minerals, probably pyrite or nickeliferous pyrrhotine, for their gold content, using an alkali-rich oxidant and silica as a flux. While the apparent use of fluxes reveals some degree of technical competence, the residues do not appear to have a counterpart in any of the assaying recipes collected in contemporary treatises.

Assay remains are very rare in the archaeological record, and even more rarely published (Beaudoin and Auger 2004; Mongiatti et al. in press), but they contain potentially unparalleled insights into the early modern quest for the exploration, understanding and, ultimately, exploitation and domination of nature (Martín-Torres and Rehren 2005). These residues illustrate the seeds of modern analytical chemistry, and of our present understanding of natural elements.

The Englishmen who founded Jamestown arrived with the highest of hopes, and the best possible equipment, ready to seek out, test and exploit rich and abundant ores. It would seem unfair to accuse them of laziness and idleness (cf. Morgan 1975), as it seems probable that they did their best. Naivety and hope, however, were probably much more abundant than the ambitioned mineral wealth.

Historical records recount that a metallurgist named Beale, "an excellent trier of minerals", identified in Jamestown an exceptionally rich gold ore. When a sample was sent to London assayers for further analyses, "all turned to vapore" (ed. Barbour 1969: I, 111). In view of the somewhat unconventional metallurgical experiments documented in this study, and considering the frustrating scarcity of rich ores in Jamestown, this anecdote appears as no surprise. Back in England, this event must have recalled a previous scandal, a few decades earlier, when tonnes of worthless rock were shipped from Canada to London, based on erroneous assays by explorers who claimed the discovery of rich gold mines (Hogarth 1999; Symons 1999; Beaudoin and Auger 2004). It seems likely that these anecdotes inform about early scientific enterprises and technical competence as much as about human ambition and faith.

Acknowledgements

We are very grateful to Carter Hudgins and Bill Kelso, of the

Jamestown Rediscovery Project, for facilitating our access to the archaeological materials, together with relevant information. We are also indebted to Kevin Reeves, at the UCL Wolfson Archaeological Science Laboratories, for his precious technical assistance.

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