# Analysis and interpretation of some crucible fragments from Jamestown

**Marcos Martinon-Torres & Thilo Rehren**

Institute of Archaeology, University College London (22nd February 2005)

## Introduction

The crucibles recovered in Jamestown, Virginia, date to the first decades of the 17th century and formed part of the equipment used there during the initial phase of occupation. Given the involvement of German craftsmen in the Virginia Company, it had been assumed from the outset that the technical ceramics, including the crucibles, might have been brought from Germany, probably from Hesse. At least one of the preserved crucible bases shows two circular stamps containing two lines of letters, respectively reading PIV and GER, separated by a central dot. This stamp is in fact identical to that observed in crucibles excavated in Hesse and published by Stephan (1995: 36-37), demonstrating the place of origin unequivocally. But how about the other, unstamped, crucible fragments, and what had they been used for?

This brief report concerns three small crucible samples, JR 124F/C1 (henceforth abbreviated to C1), A631/CC2 (in short C2) and JE 1024/CC3 (in short C3), and one metallic prill, JR 69E/224. C1 is a small wall fragment of less than three by one centimetre area and about 7 mm thick; C2 is a base/wall fragment of a small crucible with a preserved height of three centimetre, a reconstructed base diameter of only 15 mm and a wall thickness of around 5 mm; C3 is a rim sherd of a relatively large beaker-shaped crucible, of almost five by five centimetre preserved size, an original rim diameter in excess of 100 mm, and up to 10 mm wall thickness. The following part will introduce the ceramic fabric of these crucibles and then focus on the metallurgical residues preserved in the samples.

## Methodology

The specimens used for this study are standard polished cross-sections, removed from the ceramic sherds and the metallic prill, mounted in epoxy resin, ground and polished down to 1 μm particle size to expose the section as a flat surface. These were first examined in a reflected light optical microscope, and then analysed using a Philips

1

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 and have been normalised to 100 wt%. Further details of the analytical methodology are given in Martinón-Torres 2005.

## Crucible fabrics

The analysis of the crucible fabrics is reported in detail in Martinón-Torres 2005, where these crucibles are presented within a wider historical and technological context, and their technical properties, provenance and socio-economic implications are discussed and compared to those of other crucibles.

All three crucibles have a white to grey paste with darker areas due to vitrification and contamination during use. The paste is typically tempered with 20 to 40 vol% of spheroid quartz in a grain size of 200 to 500 μm. In one of the vessels (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, although the high vitrification of the paste makes it difficult to confirm this point.

The ceramic matrix of the crucibles is extraordinarily lean and homogeneous, containing very few mineral inclusions. This suggests a thorough refining of the original clay prior to tempering with quartz sand. Similarly, the temper is consistently pure quartz, with no other minerals mixed. 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 of the vessels.

The chemical compositions of the ceramic matrices show outstanding alumina levels (above 35 wt%, contrasting with contemporary Bavarian crucibles at about 30 wt%, or crucibles from other origins with alumina concentrations below 25 wt%). In addition, 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.

Overall, the paste and temper are consistent with an origin of these crucibles from Hesse, probably Großalmerode (Table 1).

| | $Na_2O$ | MgO | $Al_2O_3$ | $SiO_2$ | $P_2O_5$ | $K_2O$ | CaO | $TiO_2$ | FeO |
|---|---|---|---|---|---|---|---|---|---|
| **Jamestown JR 124F/C1** | **0.2** | **0.5** | **36.8** | **57.1** | **0.2** | **1.1** | **0.4** | **2.0** | **1.8** |
| **Jamestown A631/CC2** | **0.1** | **0.5** | **35.6** | **57.8** | **0.4** | **1.2** | **0.4** | **2.2** | **1.7** |
| **Jamestown JR 1024/CC3** | **0.2** | **0.4** | **36.8** | **56.4** | **0.3** | **1.5** | **0.2** | **1.9** | **2.3** |
| **Großalmerode 52207V** | 0.1 | 0.5 | 36.9 | 56.8 | 0.2 | 1.5 | 0.3 | 1.9 | 1.9 |
| **Großalmerode 52205Z** | 0.1 | 0.5 | 37.5 | 56.5 | 0.4 | 1.2 | 0.3 | 1.9 | 1.7 |
| **Cripplegate WFG 18/129** | 0.1 | 0.5 | 36.9 | 56.8 | 0.3 | 1.2 | 0.2 | 1.8 | 2.2 |
| **Cripplegate WFG 18/116** | 0.5 | 0.5 | 36.6 | 57.0 | 0.3 | 1.2 | 0.2 | 1.8 | 2.0 |
| **Porto CI/91/3032/60** | 0.2 | 0.7 | 36.6 | 56.9 | 0.1 | 1.4 | 0.3 | 1.8 | 2.0 |
| **Oxford OX n001** | 0.2 | 0.5 | 36.5 | 57.1 | 0.2 | 1.4 | 0.2 | 2.0 | 2.0 |
| **Oxford OX 1413** | 0.4 | 0.2 | 36.4 | 58.0 | 0.2 | 1.9 | 0.2 | 1.6 | 1.2 |
| **Burgsteinfurt BGF 01** | 0.2 | 0.5 | 36.9 | 56.5 | 0.1 | 1.4 | 0.5 | 2.0 | 1.9 |
| **Burgsteinfurt BGF 02** | 0.1 | 0.5 | 36.4 | 57.2 | 0.3 | 1.7 | 0.4 | 2.0 | 1.6 |
| **Burgsteinfurt BGF 03** | 0.2 | 0.5 | 37.6 | 56.1 | 0.3 | 1.6 | 0.4 | 1.8 | 1.5 |

**Table 1.** Chemical composition of the ceramic matrices of the crucibles from Jamestown discussed in this report (top rows). Compositions of other post-medieval crucibles found in different sites of Europe and identified as Hessian are given for comparison.
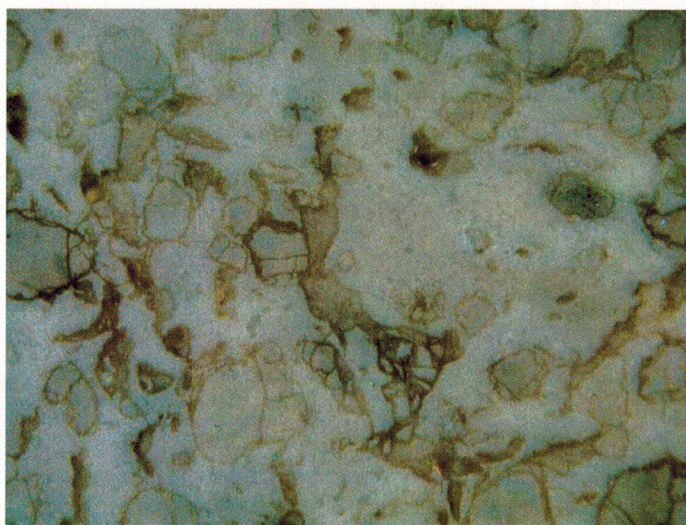


Figure 1. Detail of the fabric of the crucible, showing quartz grains and an argillaceous inclusion surrounded by a shrinkage crack (right), which is interpreted as grog (JR 124F/C1/s1, sXPL, 50x, long axis represents ~2 mm).
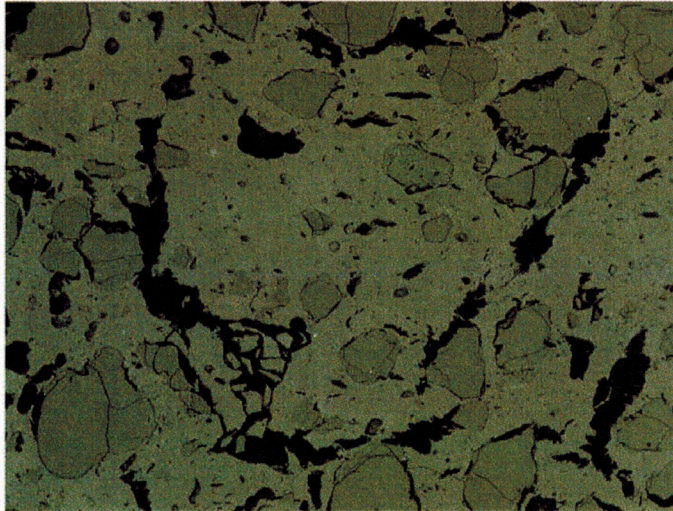
**Figure 2. BSE photomicrograph of the same fabric area (JR 124F/C1/s1, 50x).**
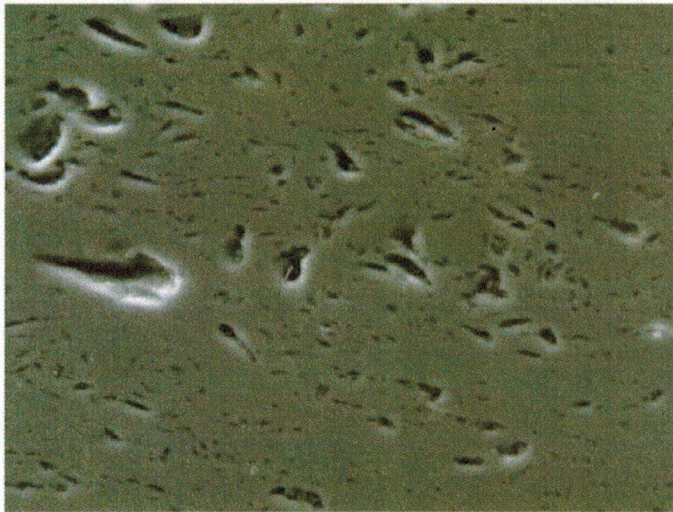
1mm



**Figure 3. Detail of the fabric of a crucible, showing complete vitrification in a very lean matrix. Note that larger voids are not shown in the image (A631/CC2/631/s1, SE, 800x).**

70μm

## Metallurgical remains

The three crucibles studied here appear to have been used for three quite different metallurgical operations, which is not surprising for a frontier settlement or foothold post in a new continent with immediate domestic needs to be served as well as ores and rocks to be tested for their potential metal content and usefulness. C1 has a few dark green corroded prills attached, indicating copper-based metallurgy, and a coherent black film covering the entire preserved inside; C2 has a small amount of glassy, dark brown slag attached near the upper end of the fragment and is otherwise only slightly discoloured on the inside, and C3 has a shiny darker vitrification film of variable coverage on its inside, with no immediately identifiable slag or corrosion.

4

### C1:  wall fragment (Figure 4)

The section through this fragment exposes one of the green prills contained in this crucible (Figure 5). It is completely corroded, but preserves an as-cast dendritic alloy structure in the corrosion products. Area analyses of various parts of the prill reveal a predominance of tin oxide (50 to 75%) and 10 to 30 wt% copper oxide; the balance being silica, lead oxide, iron oxide and phosphorous oxide (Table 2). The immediate contact area between the prill and the ceramic consists of a layer of cuprite (red copper oxide) 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.

The continuous black layer covering the inside of this crucible fragment is characterised by high amounts of zinc and iron oxide, of around 20 wt% each, as well as a significant lime enrichment (Figure 6, Table 3). The contrast in composition between the slag film and the bronze prills is remarkable, and may indicate consecutive use of the same crucible for two different operations, resulting in two disconnected use traces. The zinc- and iron-rich material could possibly indicate some brass cementation attempt, involving low-quality calamine ore; the bronze prills most likely result from a simple casting operation. This latter activity is indicated by the loose metal prill JR 69E/224 (Figure 7). This has a metallographic structure very similar to the corroded prills in the C1 sample, 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 (Figure 8-Figure 9). Area analyses for this prill gave an average tin content of 18 wt%, with just under 2 wt% lead and the balance being copper (Table 4). The area proportion of the eutectic in this sample is very similar to the equivalent material in the corroded prills from C1, suggesting that the overall composition of the alloy worked in the crucible could have been very similar to the composition of the larger prill.

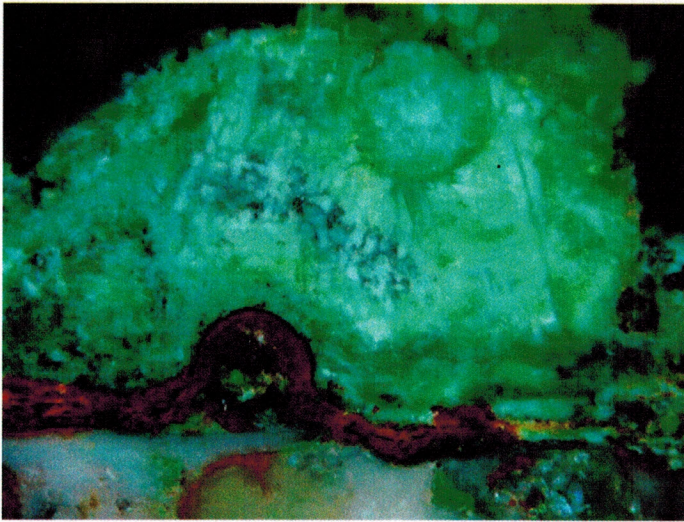Figure 4. Crucible fragment JR 124F/C1.



Figure 5. Two views of the section through one of the corroded prills adhering to the inner surface of the crucible. Note the relics of the dendritic microstructure, and the copper chloride at the interface between metal and ceramic. SEM-EDS analytical results for the spots and areas marked are given in the table below. Spectrum 5 is a large area covering most of the prill (JR 124F/C1, top XPL, bottom UPL, 100x, long axis represents ~1 mm).
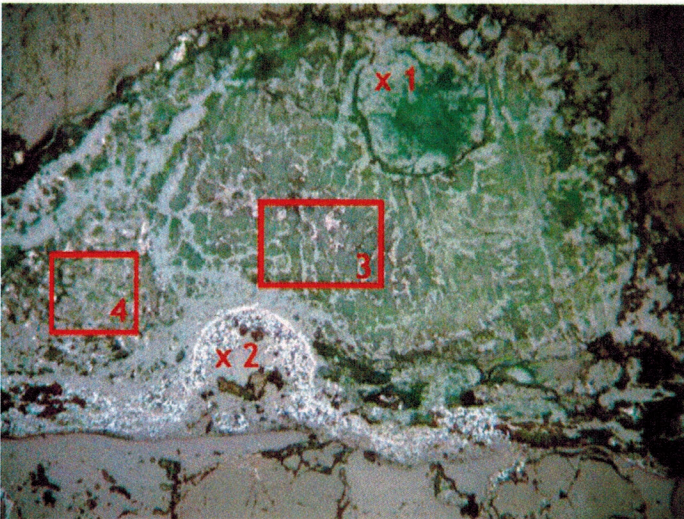
|   | Al$_2$O$_3$ | SiO$_2$ | P$_2$O$_5$ | Cl | FeO | CuO | ZnO | SnO$_2$ | PbO |
|---|---|---|---|---|---|---|---|---|---|
| 1 | - | - | - | - | - | 99.9 | <0.1 | - | - |
| 2 | - | - | - | 18.4 | - | 79.1 | - | - | 2.5 |
| 3 | - | 4.5 | 1.5 | - | 2.7 | 10.6 | - | 76.2 | 4.5 |
| 4 | - | 5.5 | 2.0 | - | 3.5 | 10.5 | - | 74.5 | 3.9 |
| 5 | 1.1 | 4.5 | 1.6 | 2.5 | 3.1 | 32.3 | - | 50.9 | 4.0 |

**Table 2. SEM-EDS analytical results of the corroded metal prill shown in Figure 5.**
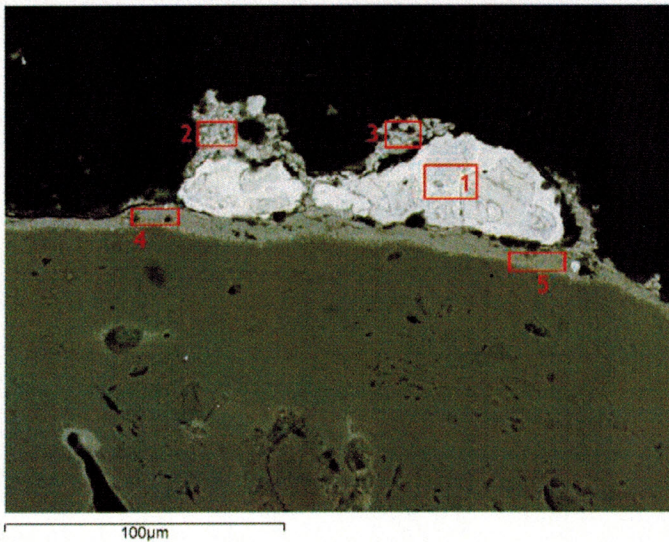


**Figure 6. Detail of the zinc- and iron oxide-rich enrichment on the inner surface of the crucible, where some corroded metal prills are sitting. SEM-EDS analytical results for the areas marked are given in the table below. (JR 124F/C1/s1, BSE, 500x).**

|   | 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 of the section of the inner surface of the crucible shown in Figure 6. Note the enrichment in zinc and iron oxides in the light grey surface layer beneath the corroded metal prills (spectra 4-5).**
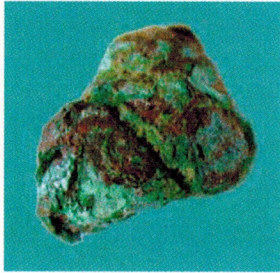
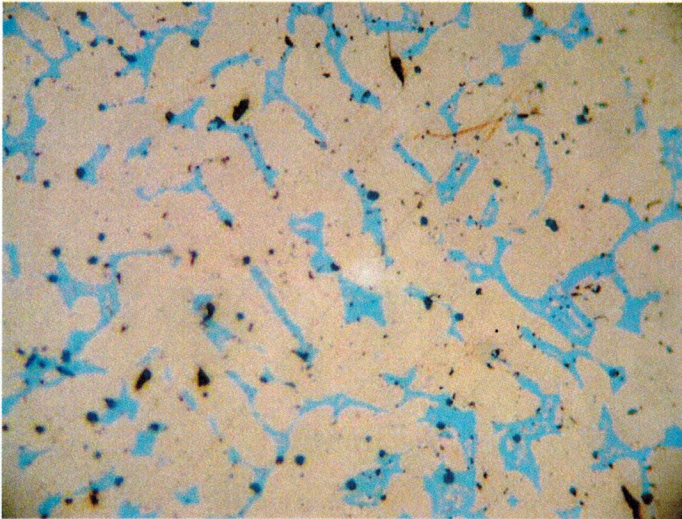Figure 7. Metallic prill JR 69E/224. Its width is ~10 mm.



Figure 8. Detail of the microstructure of the prill, showing large dendrites of alpha (golden) and interdendritic spaces of alpha-delta (grey), as well as discrete globules (dark spots) (JR 69E/224/s1, UPL, 500x, long axis represents ~200 μm).
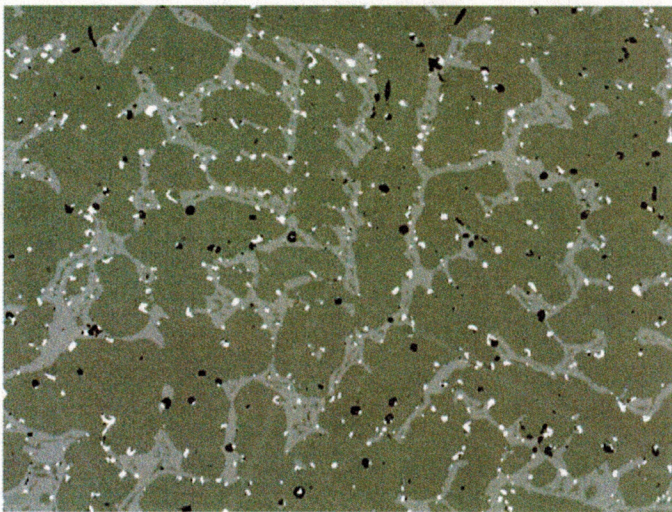


Figure 9. BSE photomicrograph of the same pril. SEM/EDS analytical results are given in the following table (JR 69E/224/s1, 500x).

100μm

|  | S | Fe | Cu | Sn | Pb |
|---|---|---|---|---|---|
| **Dendrite** | - | - | 84.8 | 15.3 | - |
| **Dendrite** | - | - | 85.4 | 14.7 | - |
| **Dendrite** | - | - | 85.4 | 14.6 | - |
| **Inter-dendrite** | - | - | 68.0 | 32.0 | - |
| **Inter-dendrite** | - | - | 68.4 | 31.6 | - |
| **Dark globule** | 17.8 | - | 77.0 | 5.2 | - |
| **Dark globule** | 18.4 | 1.4 | 80.2 | - | - |
| **Bright globule** | - | - | 22.7 | 3.6 | 73.7 |
| **Bright globule** | - | - | 22.2 | 3.8 | 74.0 |
| **Full area** | - | - | 80.1 | 18.2 | 1.7 |

**Table 4. SEM-EDS analytical results for the metal prill.**

### C2: small crucible base fragment (Figure 10)

The inner surface of this crucible is covered by a thin dark brown to black film, with a larger lump of glassy shiny slag being preserved near the upper end (Figure 11, Tables 5 and 6). 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 one percent by weight. 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 as much 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, $Fe_2O_3$, as indicated by their crystal shape and the bright red internal reflections (Figure 12). Discrete particles can have up to 15 wt% nickel oxide; these are probably nickeliferous magnetite (Figure 13, Table 5). The co-existence of magnetite and haematite indicates highly oxidising conditions, quite unlike typical iron smelting slag.

There is no published precedent for this assemblage which would guide in its identification. A connection to iron metallurgy is unlikely for a number of reasons; firstly, and although the bulk chemical composition is somewhat similar to early modern iron slag, it is far too oxidised to be 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 & 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 of the slag. This leaves a connection to noble metals, i.e. silver and gold, which is further corroborated by the small size of the vessel. Of these two, silver is less likely as it is most often associated with either copper or lead, or both, either by nature or artificially alloyed, none of which are present.

How then could this slag relate to gold metallurgy? One possible scenario is the assaying 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, together with some organic or inorganic salt, under oxidising conditions. Saltpetre, a combination of sodium and potassium 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. Depending on the actual relative proportions of materials present in the charge and the parameters of processing, 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. It remains open, however, to imagine what should have happened to the presumed gold in this test. We have no indication for the former presence in the crucible charge of either of the possible collectors, matte or lead metal, and have to assume that the Jamestown assayer would have hoped to gain a noticeable gold-rich regulus straight out of this scorification process. This either indicates a confidence in a rather rich ore to be tested, or a reluctance to go through the full fire assay procedure including scorification, collecting the suspected noble metal in a lead bullion, and then subjecting this to cupellation in order to retrieve the noble metal. Both possibilities are feasible under the specific circumstances of the early settlers in Jamestown, which may not have included among their group an experienced assayer with the complete laboratory equipment required for full fire assays, and would have been driven by high hopes for the mineral riches of

their new environment; after all, one of their key objectives, set by the Virginia Company, was to find gold, and a jeweller as well as a number of 'refiners' are known among the group arriving in early 1608 (www.apva.org/history/1stsup.html).
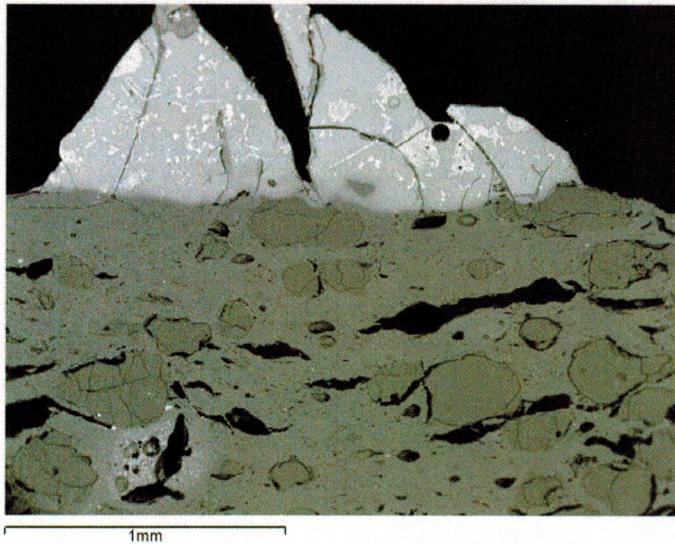


Figure 10. Crucible sample A631/CC2/631.



Figure 11. Section through the same crucible, showing the quartz-tempered fabric with one small molten mineral (bottom) and slag remains adhering to the inner surface (top) (A631/CC2/s1, BSE, 50x).
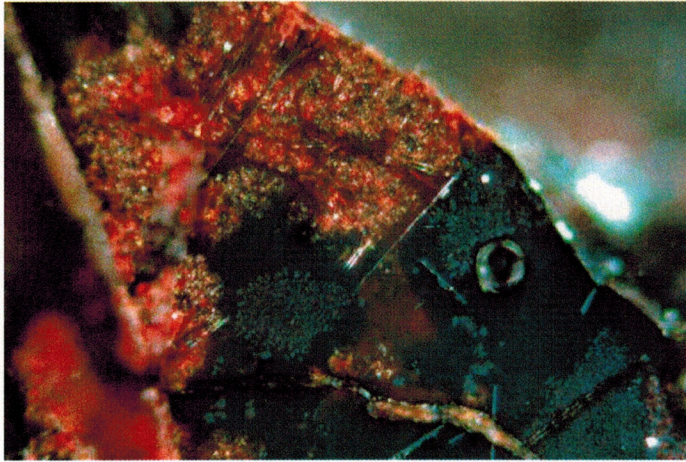
Figure 12. Detail of the slag adhering to the inner surface of the crucible. Note the very glassy appearance and the clustering of bright crystals with red internal reflections (A631/CC2/s1, top XPL, bottom UPL, 200x, long axis represents ~500 μm).
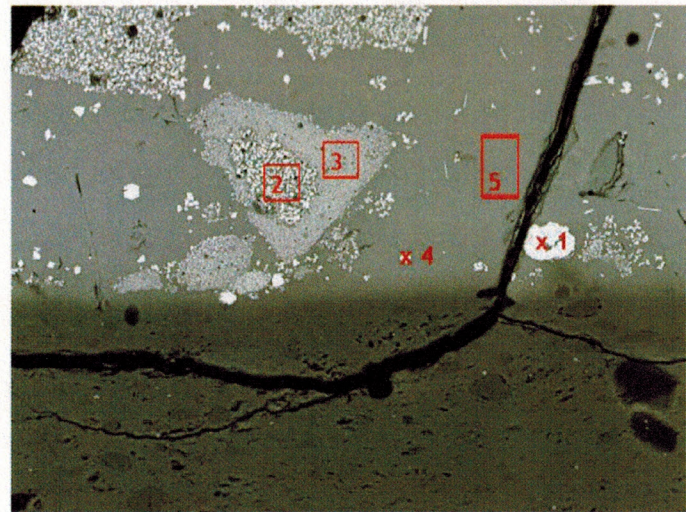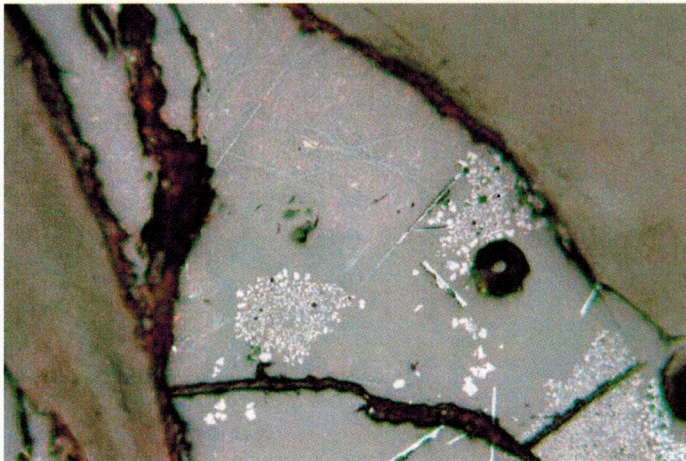


Figure 13. BSE photomicrograph of the interface between the slag and the crucible fabric. SEM-EDS analytical results for the spots and areas marked are given in the following table. Spectrum 6 is a large area of the slag. (A631/CC2/s1, 300x).

|   | 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 | - | 0.9 | - | - | - | - | - | - | - | 0.4 | 83.2 | 15.6 |
| 2 | 5.7 | - | 1.6 | 19.9 | 0.4 | 0.7 | 3.1 | 0.2 | 0.4 | - | 68.0 | - |
| 3 | 5.5 | - | 1.5 | 23.6 | 0.6 | 0.9 | 3.7 | - | - | 0.5 | 63.1 | 0.7 |
| 4 | - | 0.4 | - | 0.5 | - | - | 0.3 | - | - | 0.6 | 97.0 | 1.2 |
| 5 | 9.8 | - | 1.4 | 46.9 | 1.1 | 1.0 | 7.7 | 0.7 | - | - | 31.5 | - |
| 6 | 8.9 | - | 1.5 | 40.7 | 1.1 | 0.9 | 6.3 | 0.5 | - | - | 40.1 | - |

Table 5. SEM-EDS analytical results for the slag shown in Figure 13
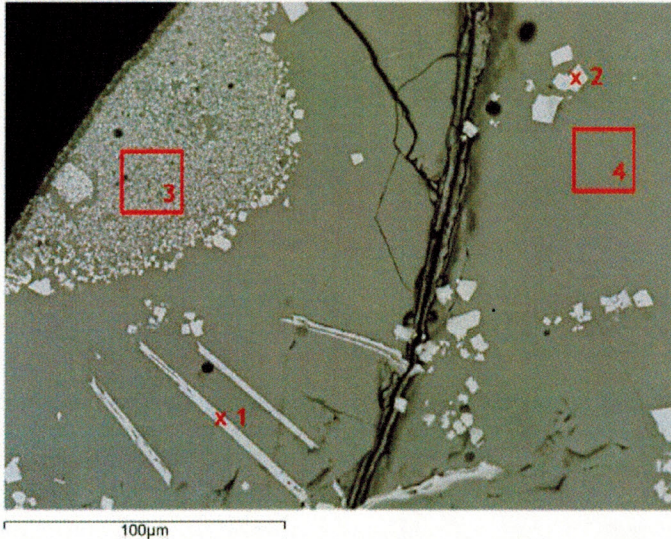


Figure 14. Detail of the same slag. SEM/EDS analytical results are given in the following table (A631/CC2, BSE, 500x).

|   | Na$_2$O | Al$_2$O$_3$ | SiO$_2$ | P$_2$O$_5$ | Cl | K$_2$O | CaO | TiO$_2$ | MnO | FeO |
|---|---|---|---|---|---|---|---|---|---|---|
| 1 | 0.5 | - | 0.9 | - | - | 0.2 | - | 0.7 | - | 97.8 |
| 2 | - | - | 0.4 | - | - | 0.2 | - | - | 0.9 | 98.5 |
| 3 | 7.5 | 1.0 | 27.9 | 0.7 | 0.9 | 4.6 | 0.7 | - | 0.4 | 56.4 |
| 4 | 10.7 | 1.6 | 41.8 | 1.3 | 1.0 | 7.4 | 1.1 | 0.3 | - | 34.8 |

Table 6. SEM-EDS analytical results for the detail of the slag shown in Figure 14.

### C3: large crucible rim fragment (Figure 15)

This set of three matching fragments is from a rather large vessel and 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 slag into the fabric (Figures 16-20). It is this latter slag, also present as a thin film on the inside surface, which carries most metallurgically relevant information. Most noticeable is its widespread contamination with copper, both as metallic prills and as dendritic cupper oxide crystals precipitating from the melt. Alloying metals, i.e. tin, zinc and lead, however are absent or only present in rather low concentrations. The slag composition

itself is dominated by silica (40 to 45 wt%), lime (20 to 25 wt%), copper oxide (c. 15 wt%), alumina (c. 10 wt%) and minor amounts of iron oxide (c. 5 wt%). The alkali oxides, which were so prominent in the slag matrix of the previous crucible, are here present at less than one percent by weight only (Table 7). Within this glassy slag separate two discrete phases; 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, $KAlSi_2O_6$. The occurrence of this phase is a result of a relatively high alumina and alkali oxide content; the prevalence of alumina over alkali oxides in the glassy matrix indicates that the overall composition was richer in alumina than alkali oxides. A similar phase, also separating from a surrounding slag matrix, is known from smithing and some smelting slags in iron metallurgy, again reflecting bulk chemical peculiarities more than process parameters. The chemical interpretation in those cases is often that the high alkali content of fuel ash contributes to the formation of this phase.

In terms of metallurgical activity, one can assume that the crucible was used for melting and casting of copper, probably 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 and hence increased fuel ash contamination of the crucible surface.



Figure 15. Crucible rim fragment JR 1024/CC3. Note the shiny glaze and relatively smooth surface resulting from contamination and vitrification during use.

14
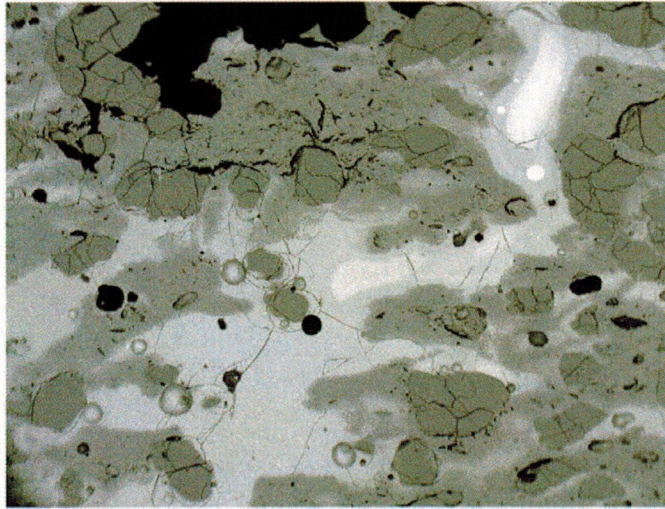
1mm

Figure 16. BSE photomicrograph of the crucible JR 1024/CC3, showing the penetration of slag, very rich in lime and copper oxide, through the fabric cracks (JR 1024/CC3/s1, 50x).
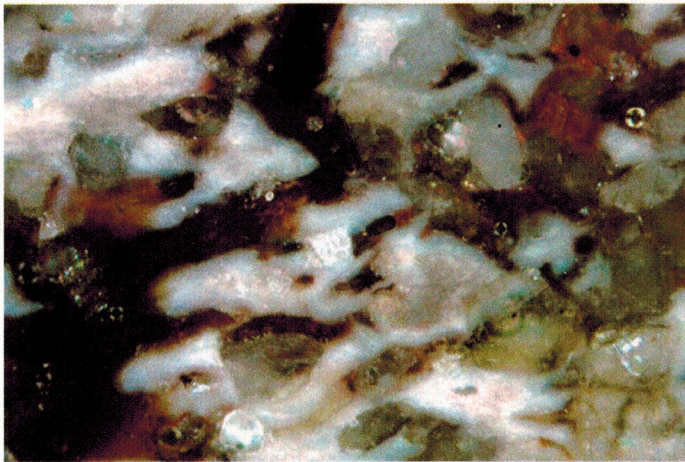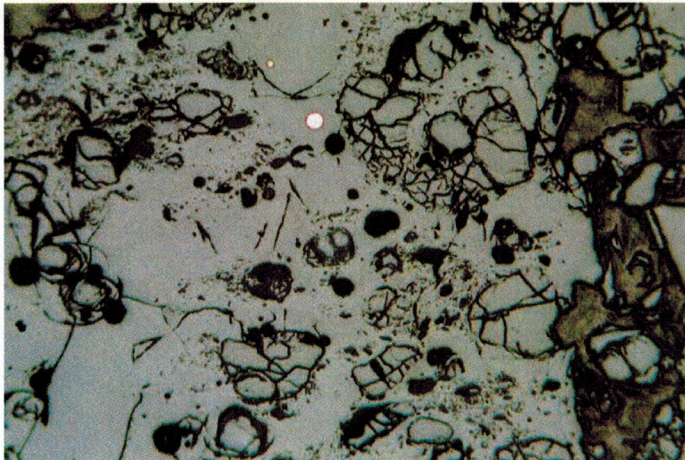


Figure 17. Section through the ceramic fabric of the crucible, showing the intense penetration of glassy slag (top image, brown) containing metallic prills, and the severe shattering of the quartz grains (bottom image) (JR 1024/CC3/s1, top XPL, bottom UPL, 50x, long axis represents ~2 mm).
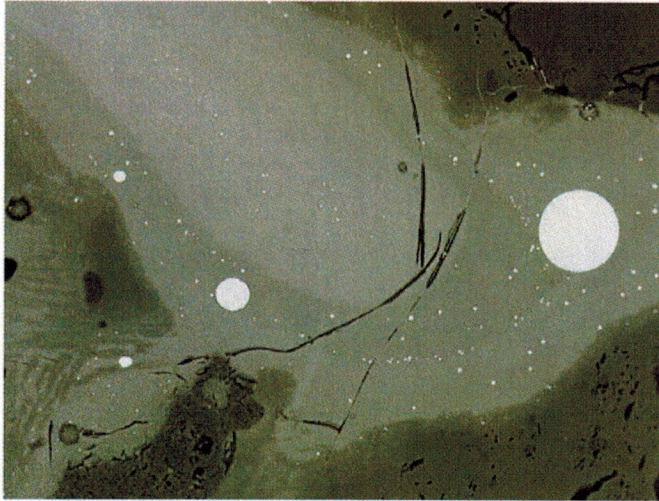
**Figure 18. Detail of the slag penetrating into the ceramic fabric, very rich in lime and copper oxide. Note the abundance of minuscle copper prills (bright) (JR 1024/CC3/s1, BSE, 250x).**
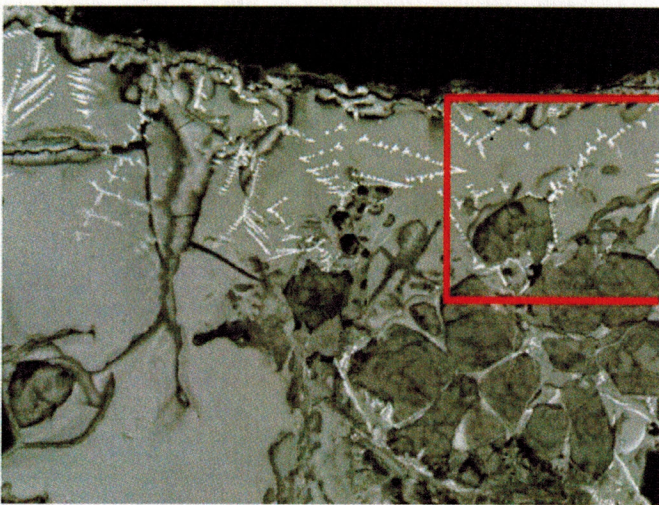


**Figure 19. Detail of the slag on the inner surface of the crucible. The marked area is shown in the following figure under higher magnification (JR 1024/CC3/s1, BSE, 600x).**
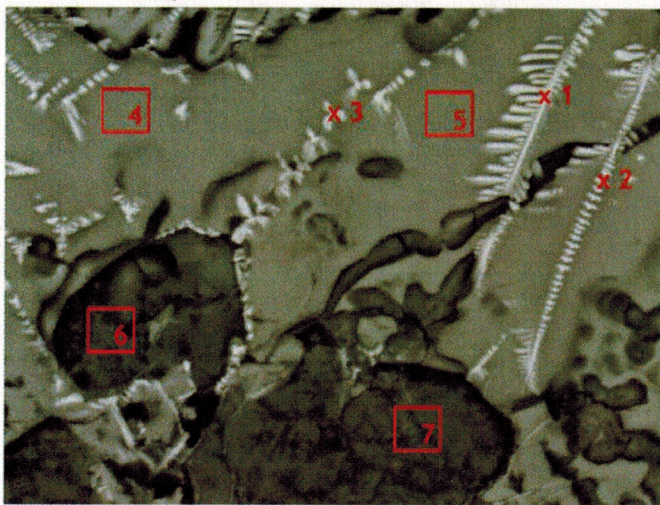


**Figure 20. Detail of the slag shown in the previous figure. SEM/EDS analytical results are given in the following table (JR 1024/CC3/s1, BSE, 1400x).**

16

|   | 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 | - | 1.3 | 6.6 | 27.7 | 0.6 | 0.8 | 10.1 | 0.5 | 0.2 | 2.0 | 50.3 | - | - | - |
| 2 | - | 0.7 | 3.9 | 17.9 | 0.2 | 0.7 | 5.9 | - | - | 1.3 | 69.0 | 0.4 | - | - |
| 3 | - | 1.6 | 7.9 | 37.9 | 0.7 | 1.2 | 15.4 | 0.7 | 0.3 | 3.3 | 31.1 | - | - | - |
| 4 | - | 2.1 | 9.0 | 44.6 | 0.9 | 0.8 | 21.6 | 1.5 | 0.5 | 4.7 | 14.7 | - | - | - |
| 5 | - | 2.2 | 9.2 | 43.7 | 0.99 | 0.8 | 21.8 | 1.0 | 0.6 | 4.7 | 15.1 | - | - | - |
| 6 | 6.0 | 0.2 | 21.8 | 54.0 | 3.8 | 3.5 | 1.8 | 0.3 | - | 3.8 | 2.6 | - | 0.2 | 2.1 |
| 7 | 6.6 | - | 23.9 | 57.8 | 2.5 | 3.5 | 0.3 | - | - | 1.5 | 1.6 | - | - | 2.3 |

**Table 7. SEM-EDS analytical results from the detail of the slag shown in Figure 20**

## Conclusion

The three small crucible fragments investigated in this pilot study represent three apparently different metallurgical activities. C1 and C3 both were probably used to melt and cast bronze and copper, respectively; however, there is the possibility that C1 had been used previously for a different activity, brass melting or even cementation. As has been shown repeatedly in the past it is near impossible to reconstruct with any certainty the original alloy composition from the remains left behind in the crucible (e.g.Dungworth 2000); however, the bronze prill JR69E/224 is of a composition fully compatible with the remains in C1. The 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. C3 was used at a higher temperature and with a much cleaner copper metal; whether this places this crucible in a direct relationship to the copper sheet off-cuts from Jamestown is at present difficult to say.

The third crucible, C2, is the most technically interesting one. 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. Assay slags are very rare in the archaeological record, and only partially published (Beaudoin and Auger 2004), but contain potentially unparalleled levels of information regarding past metallurgical practices in a given site and context.

## References

Beaudoin, G. and Auger, R., 2004. Implications of the mineralogy and chemical composition of lead beads from Frobisher's assay site, Kodlunarn Island, Canada: prelude to Bre-X?, *Canadian Journal of Earth Sciences*, **41**, 669-681.

Dungworth, D., 2000. A note on the analysis of crucibles and moulds, *Historical Metallurgy*, **34**, 83-86.

Martinón-Torres, M. 2005. *Chymistry and Crucibles in the Renaissance Laboratory: An archaeometric and historical approach.* PhD thesis. London: Institute of Archaeology, University College London.

Rehren, Th. & Papachristou, O., 2003. Similar like white and black: a comparison of steel-making crucibles from Central Asia and the Indian subcontinent. In: Th Stoellner, G Koerlin, G Steffens & J Cierny (eds), *Man and Mining - Mensch und Bergbau. Studies in Honour of Gerd Weisgerber*, (=Der Anschnitt, Beiheft 16, Bochum), 393-404.

Stephan, H.-G., 1995. *Großalmerode: Ein europäisches Zentrum der Herstellung von technischer Keramik. Die Geschichte der keramischen Gewerbe in Großalmerode und Epterode und die Entwicklung ihrer Produktion vom 12. bis zum 19. Jahrhundert. Teil II: Technische und Baukeramik, Tonpfeifen, Knicker, Steingut, Porzellan, Aspekte von Handel, früher chemischer Industrie, Bergbau und Gewerbegeschichte.* Großalmerode (Germany): Glas- und Keramikmuseum Großalmerode.