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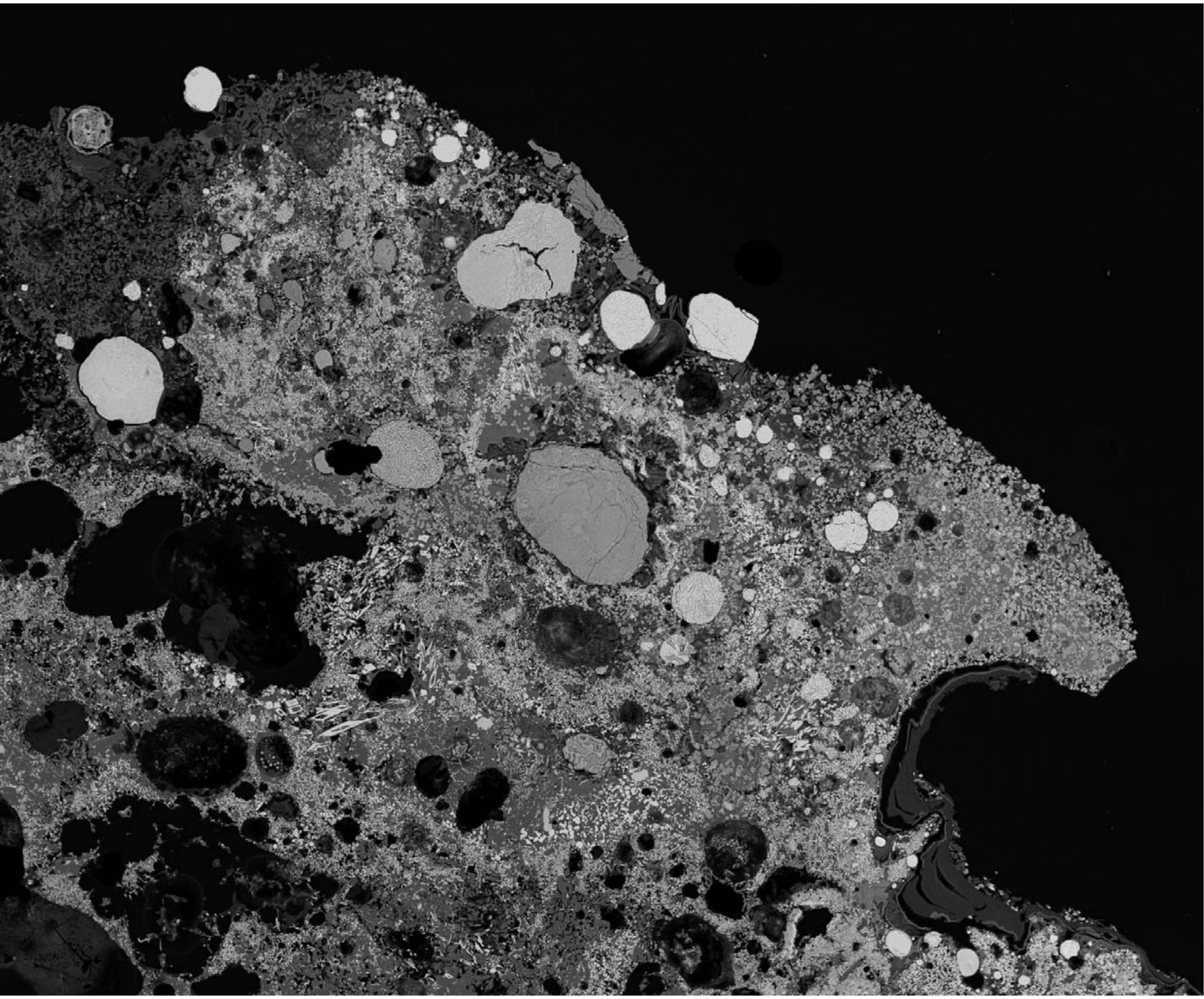
PARK FARM EAST, ASHFORD, KENT ANALYSIS OF CRUCIBLES FROM THE IRON AGE SETTLEMENT

TECHNOLOGY REPORT

Victoria A L Lucas and Sarah Paynter



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PARK FARM EAST
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Analysis of Crucibles from the Iron Age Settlement

Victoria Lucas and Sarah Paynter

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SUMMARY

Four vessels from the Iron Age settlement of Park Farm East were submitted for assessment with regards to their supposed function and use as crucibles. Three of the four vessels were shown to have been crucibles employed in the melting of what was probably a reasonably high tin bronze.

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DATE OF RESEARCH

March 2010

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INTRODUCTION

The site of Park Farm East of Ashford, Kent is located to the east of the A2070 road. To the West the site is bounded by the Ashford to Hastings railway line, to the south by the Kingsnorth to Cheeseman's Green road and by agricultural land to the north and east. The underlying geology is a Cretaceous Wealdon Clay containing quantities of iron pan and flint pebbles. Excavations were undertaken, in 2003, by Wessex Archaeology who were commissioned by CgMs Consulting on behalf of Taylor Woodrow Developments Ltd. Three principle phases of activity were identified: a mid to late Iron Age farmstead, a late Iron Age rectilinear enclosure system with evidence for industrial activity and continued settlement and alteration of the enclosure into the early Roman period. This report deals with the analysis of crucibles recovered from Late Iron Age to early Roman contexts from the site. According to the post-excavation assessment (Wessex Archaeology 2004) pottery preservation was very poor due to acidic burial conditions and the ceramics suffered from heavy abrasion of exposed surfaces.

METHOD

The crucibles were examined by two analytical methods; surface EDXRF (energy dispersive X-ray fluorescence) analysis and quantitative SEM-EDS (scanning electron microscopy energy dispersive spectroscopy). The crucibles were also visually examined and photographed prior to sampling.

Surface EDXRF Analysis

Surface EDXRF analysis was carried out (using an EDAX Eagle II at 40kV) in order to give an indication of the types of alloys melted in the crucibles and to eliminate vessels unlikely to have been used as crucibles. This method is ideal for preliminary analysis as it is quick and non-destructive. Surface EDXRF is not quantitative and the proportions of alloying elements detected are unlikely to be representative of those in the original metal (due to differential volatilisation and oxidation of the alloying elements and subsequent corrosion) (Dungworth 2000a).

SEM-EDS Analysis

The crucibles were examined in detail using quantitative SEM-EDS analysis. The analysis conditions were 25kV, 100 seconds of live time, at a 10mm working distance (FEI Inspect F with Oxford Instruments X-act SDD detector and INCA software). The back-scatter electron detector produces images based on atomic number; whereby different atomic numbers give different shades of grey on the image, allowing easy identification of different inclusions or phases in the crucible fabric or metal. Bulk analysis of the fabric was carried out at 150x magnification; small features of interest, such as inclusions, were analysed in spot mode.

RESULTS

Visual Examination

Eight fragments representing three separate crucibles (SFN: 10032, 10007 and 10005) were submitted for analysis as well as a complete thumb-pot (SFN: 20010) of unknown function.

Figure 1 shows a plan view of three adjoining fragments of crucible from 10007 showing the reduced fired grey interior surface

Crucibles 10007 (CN: 11867) and 10005 (CN: 11881) are very similar, both in terms of form and fabric. They have grey reduced fired interior (Figure 1) and exterior surfaces with extensive bloating and red vitrification of the rim and upper outside surfaces (Figure 2); this indicates exposure to high temperatures and that they were heated from above (Bayley and Rehren 2007). Both fabrics contain a lot of quartz of variable coarseness, which comprises approximately 80-90% (by volume) of the crucible fabric. There are also relatively large amounts of copper alloy adhering to the upper portion of the interior surface of several of the fragments from these crucibles. 10005 has a pouring spout which appears to have been pinched out from the rim of the crucible (Figure 3); this is where the greatest quantity of copper alloy is found. The fragments are likely to be sherds from the small triangular-plan crucibles typical of the Iron Age (Gregory 1991, 139, type B, Paynter 2002, Wainwright, 1979, 125-149) (Figure 4). The reconstructed crucibles have an internal diameter of about 90mm at their widest point, and a maximum depth of approximately 70mm. When in use they appear to have been filled to within about 20-30mm of the rim.

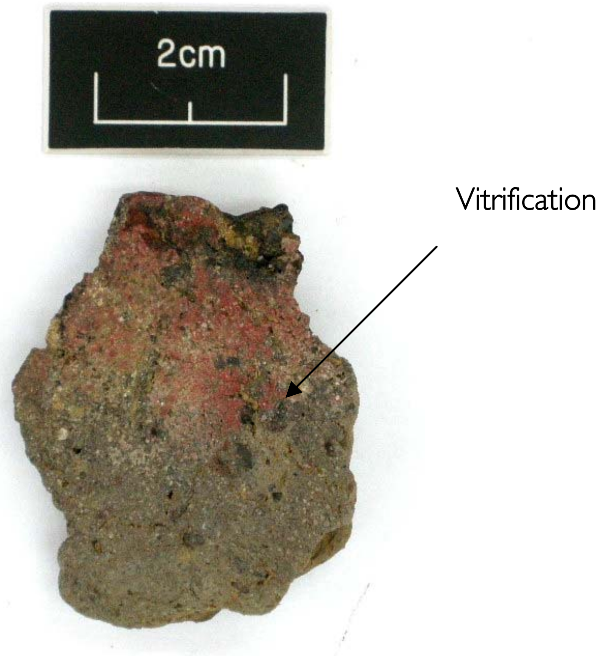

Figure 2 shows a fragment from 10005 showing the exterior surface and a typical red vitrified rim

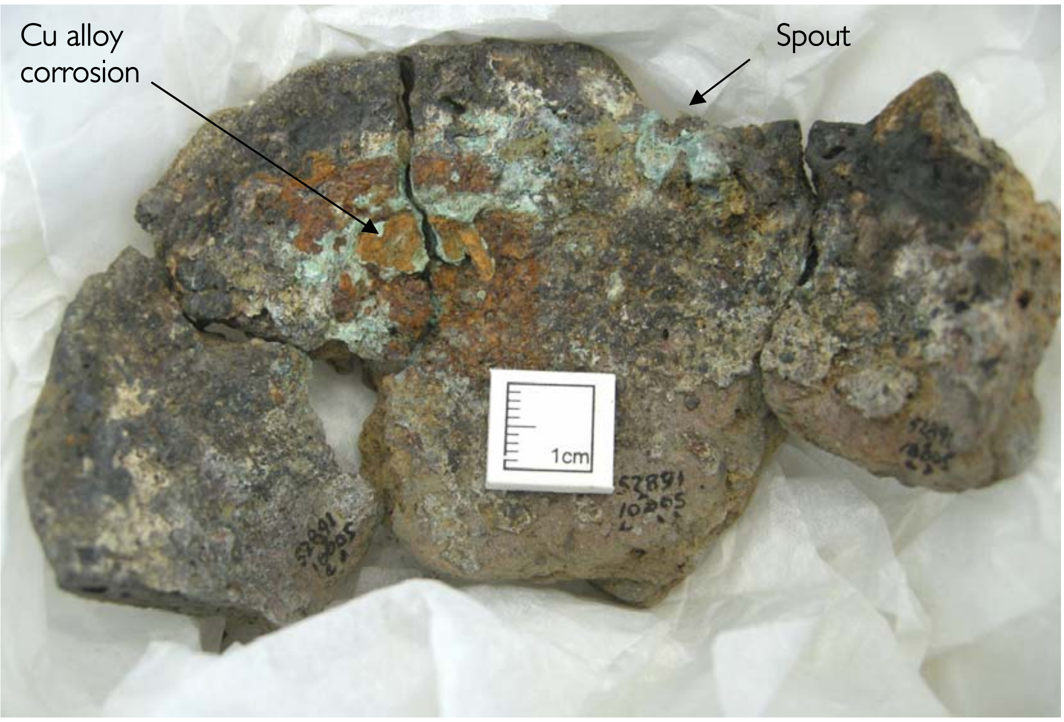

Figure 3 shows a plan view of four adjoining fragments from 10005 with a large amount of copper alloy corrosion adhering to the interior surface of the pouring spout

Figure 4 shows a side view of the reassembled partial crucible from 10007, showing the profile

The crucible rim fragment from 10032 appears similar to 10007 and 10005 in terms of fabric and form. The piece is small and so it is difficult to reconstruct the vessel's shape or size but it may be a fragment from the vertices of another triangular crucible. This crucible is only lightly fired relative to the other examples, suggesting that it has not seen the same amount of use (Figure 5). This may also explain the higher levels of sulphur consistently detected in fragments of 10032 relative to the other crucibles.

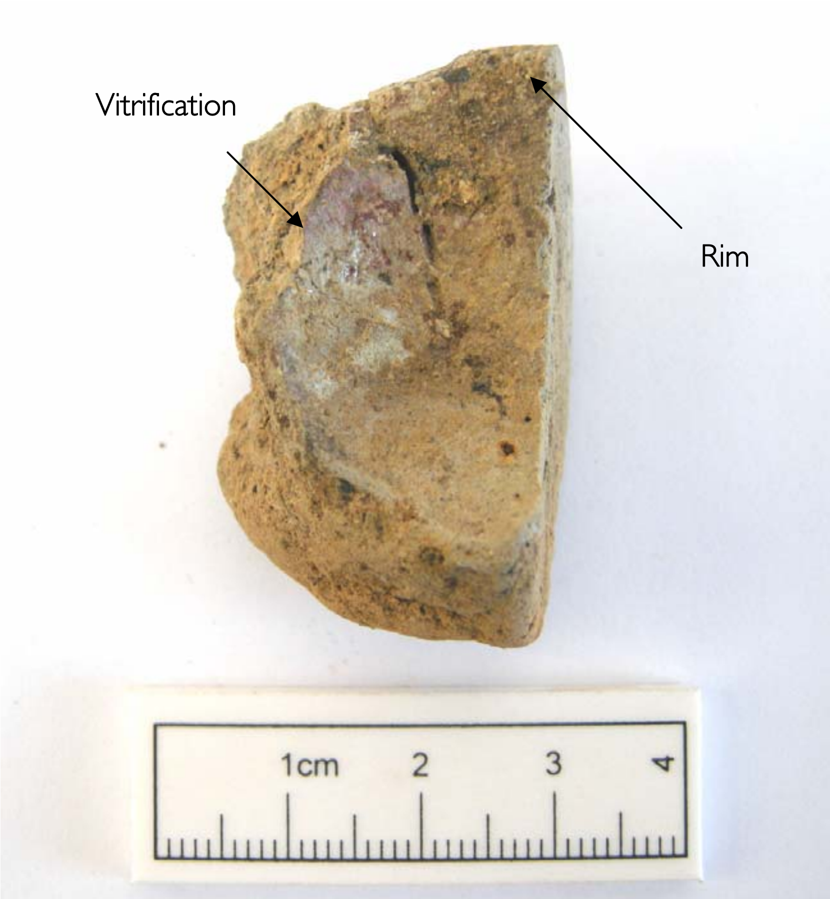

Figure 5 shows the interior surface of 10032

The thumb-pot (20010) is about 25mm in diameter and 15-20mm tall (Figure 6). It is tempered with yellow/white grog and some fine quartz comprising about 60-70% of the fabric. The vessel is red oxidised fired throughout and shows no signs of bloating or vitrification, there are no visible traces of copper alloy. Based on these attributes it was concluded that this vessel was unlikely to have been used as a crucible.

Figure 6 shows the small thumb-pot showing rim side on profile (left) and red oxidised fired interior surface (right)

Surface EDXRF Analysis

Surface EDXRF analysis detected zinc on crucible 10032, and also lead, copper and tin. Copper, tin, zinc and lead were detected on crucible 10007. Copper, tin, lead and a trace of zinc were detected on crucible 10005. This confirms that these crucibles were used to melt copper alloys.

The range of elements detected in thumb pot 20010 are all found in the clay used to make the pot; no evidence for the melting of metals or alloys was detected. This vessel also has a red oxidised fired fabric and no indications of exposure to particularly high temperatures. It seems unlikely therefore that this vessel was employed as a crucible.

Quantitative SEM-EDS Analysis

Quantitative SEM-EDS analysis was carried out on 10032, 10007 and 10005; 20010 was not analysed. Samples were taken from a single fragment of each crucible, avoiding joining edges between fragments. The samples were mounted in epoxy resin, polished to a 1 micron finish and carbon coated.

Crucible fabric

The compositions of the fabrics were determined. Bulk analysis of areas of fabric least altered by exposure to metals and fuel ashes was carried out at 150x magnification over an area approximately $4mm^2$. The results of these analyses are shown in Table 1.

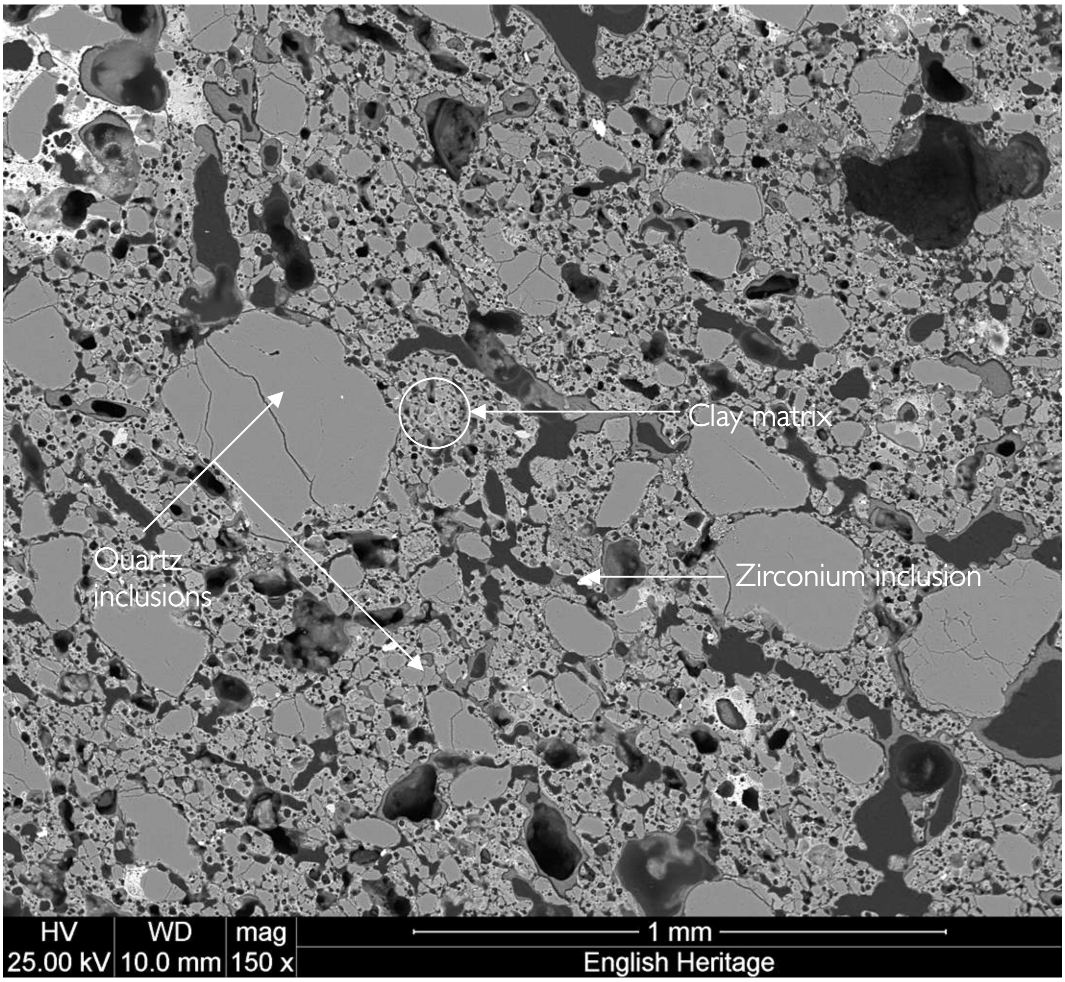

Figure 7 showing an SEM back-scatter image of crucible fabric 1007

Table 1: Average (21 analyses) bulk compositions of the fabric of crucibles 10005, 10007 and 10032 and associated standard deviations (SD). Arsenic was not present above the detection limits (for full data see Appendix Table 1)

		Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	SO_3	K_2O	CaO	TiO_2	MnO	Fe_2O_3
10007	Mean	0.3	0.5	9.8	79.9	0.4	0.0	1.3	0.2	0.8	0.1	6.6
	SD	0.1	0.1	1.6	3.1	0.1	0.0	0.2	0.1	0.1	0.1	1.3
10005	Mean	0.5	0.6	10.8	75.5	0.6	0.1	1.6	0.3	0.8	0.1	9.1
	SD	0.2	0.2	1.8	5.7	0.4	0.1	0.3	0.1	0.1	0.1	3.4
10032	Mean	0.7	0.5	9.0	82.1	0.3	1.1	1.4	0.3	0.8	0.0	3.7
	SD	0.2	0.1	1.0	1.4	0.1	0.3	0.2	0.0	0.1	0.0	0.5

In terms of chemical composition the fabrics of the three crucibles are very similar (Table 1) being roughly 79wt% silica with the remainder dominated alumina (Al_2O_3) and iron oxide (c10wt% and c6wt% respectively). Figure 7 is typical of the microstructure of the three crucibles. The majority of the fabric is poorly sorted quartz, explaining the high silica content obtained by the bulk analyses, in a matrix of clay with high proportions of alumina and iron oxide. The fabric displays considerable vitrification resulting from exposure to high temperatures and reactions with fuel ashes; little of the original clay matrix remains unreacted. 10032 shows marginally less bloating than the other two crucibles, indicating perhaps slightly lower temperatures or less prolonged exposure to high temperatures. The fabric of these crucibles is well suited to its purpose having relatively high proportions of silica, which would have ensured that it was suitably refractory (Dungworth 2001). All three crucibles also contained some zirconium inclusions, whilst 10032 contained a very small amount of rutile (TiO_2) and lathes of iron oxide (Fe_2O_3) as well as some monazite ([Ce,La,Nd]PO_4).

Vitrification

Many areas of the crucibles displayed vitrification, which fell into two categories: that found at the interior edges of the crucible and that found in the body of the crucible. The vitrification at the interior edges is generally the most extensive with the crucible fabric having become fully fused. The concentrations of K_2O are enhanced in these edge vitrified zones, probably due to reactions with the fuel ash, and increased levels of copper and tin were often detected. In the case of 10005 this is particularly evident (Figure 8) and the vitrified areas contain droplets of copper and droplets of tin bronze and large amounts of cassiterite (SnO_2) and copper oxide dendrites. 10032 also contained some droplets of what was originally tin bronze in the vitrified zones, however these were heavily corroded and only minute traces of copper remained.

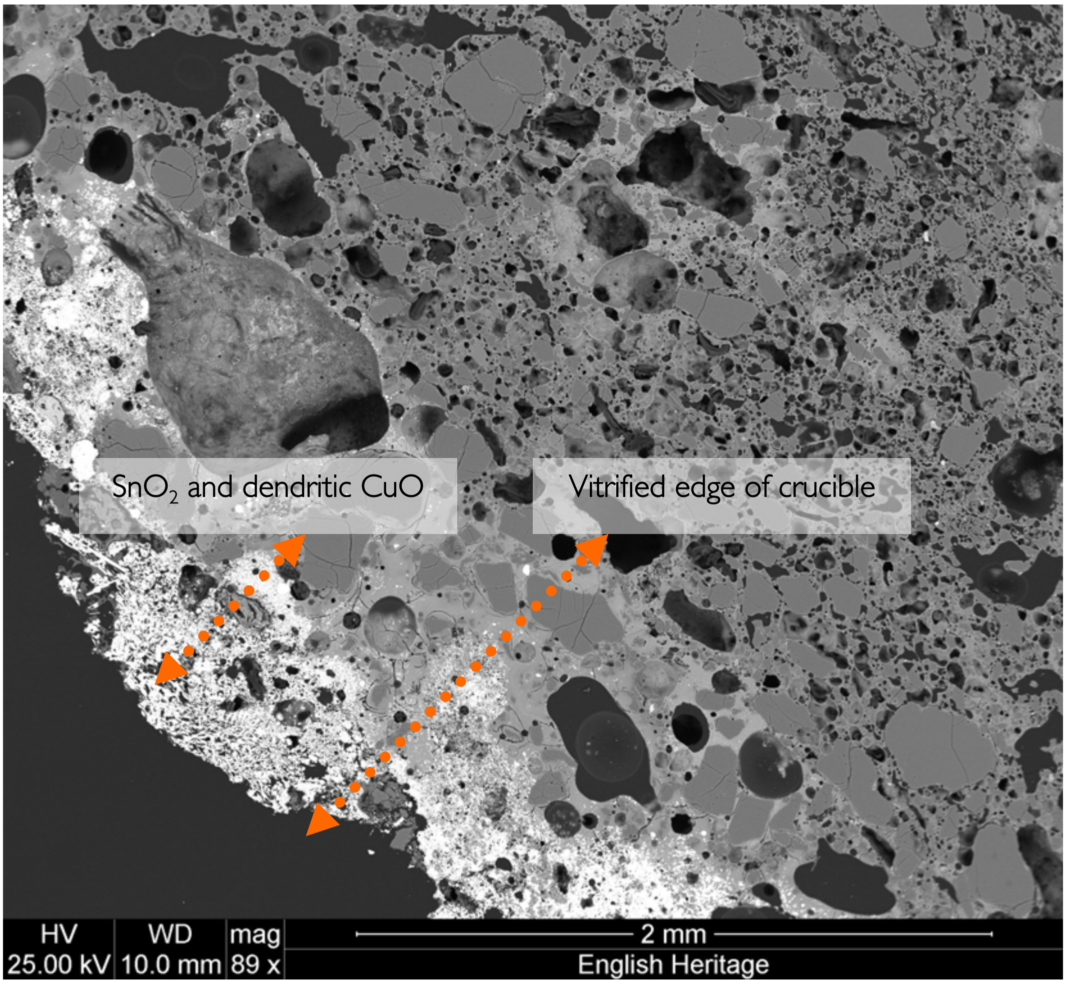

Figure 8 shows an SEM back-scatter image showing vitrification of interior edge of 10005 including cassiterite and copper oxide phases, lower image shows detail of fused ceramic

Vitrification of areas in the body of the crucible is localised and characterised by high proportions of Fe_2O_3 and TiO_2. These areas of vitrification result from the reaction of mineral inclusions, such as rutile, with the surrounding clay fabric (Figure 9).

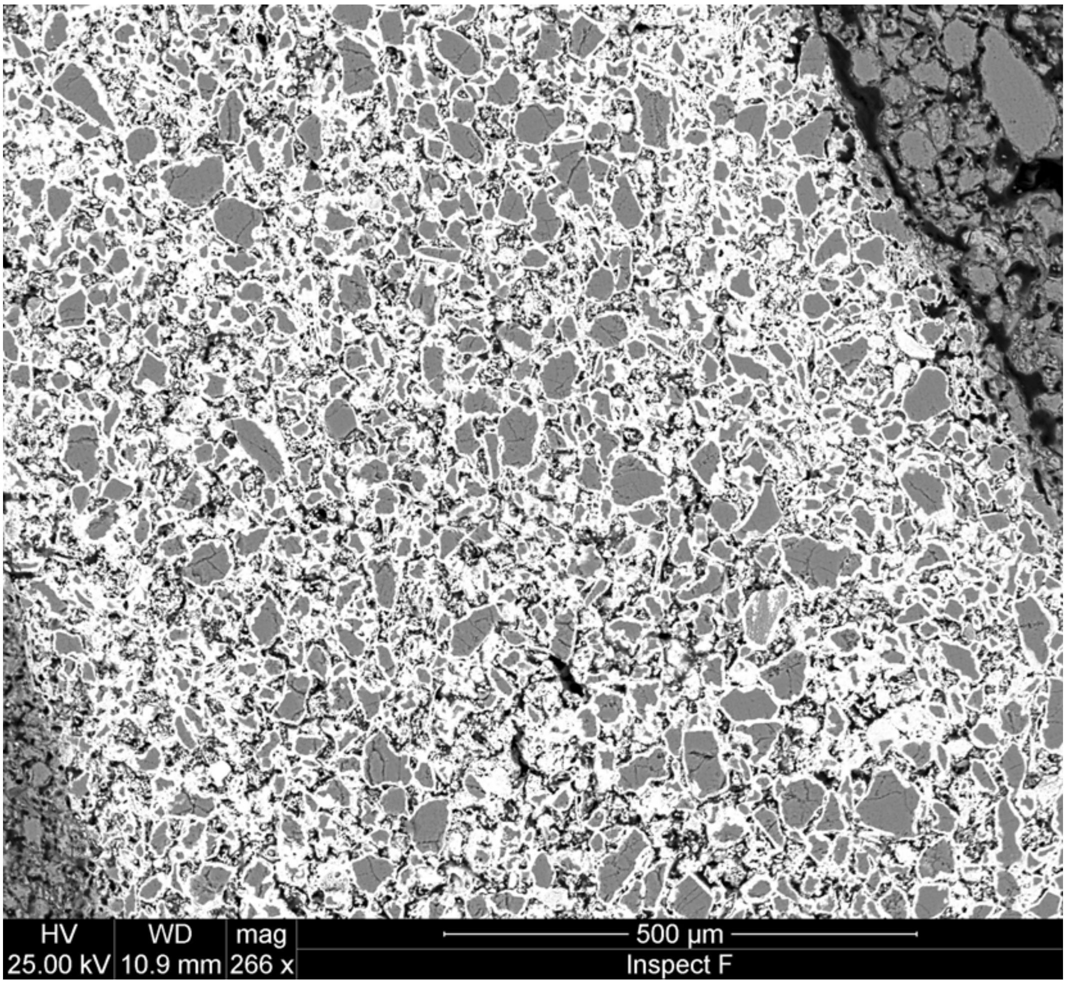

Figure 9 showing an SEM back-scatter image of vitrfication in the area of an iron- and titanium-rich inclusion (white matrix) incorporating quartz grains (grey particles) in crucible 10032

Copper Alloy Phases

Remnants of copper alloy were only found in significant quantities in the sample from crucible 10005 within the vitrified zone of the interior edge. Small amounts of cassiterite and corroded, tin-rich metal droplets were found in the sample from crucible 10007, but very little in the sample from crucible 10032.

The outer edges of the vitrified zones of 10005 contain large amounts of crystalline cassiterite (SnO_2) (Figure 10). There also appear to have been some copper oxide dendrites but the copper-rich phases have been preferentially dissolved post-burial, leaving mainly the insoluble cassiterite phase. The copper- and tin-rich phases are derived from slaggy oxidation products on the surface of the melt (Dungworth 2000b), and the molten metal itself, and have all been altered by the post-depositional environment.

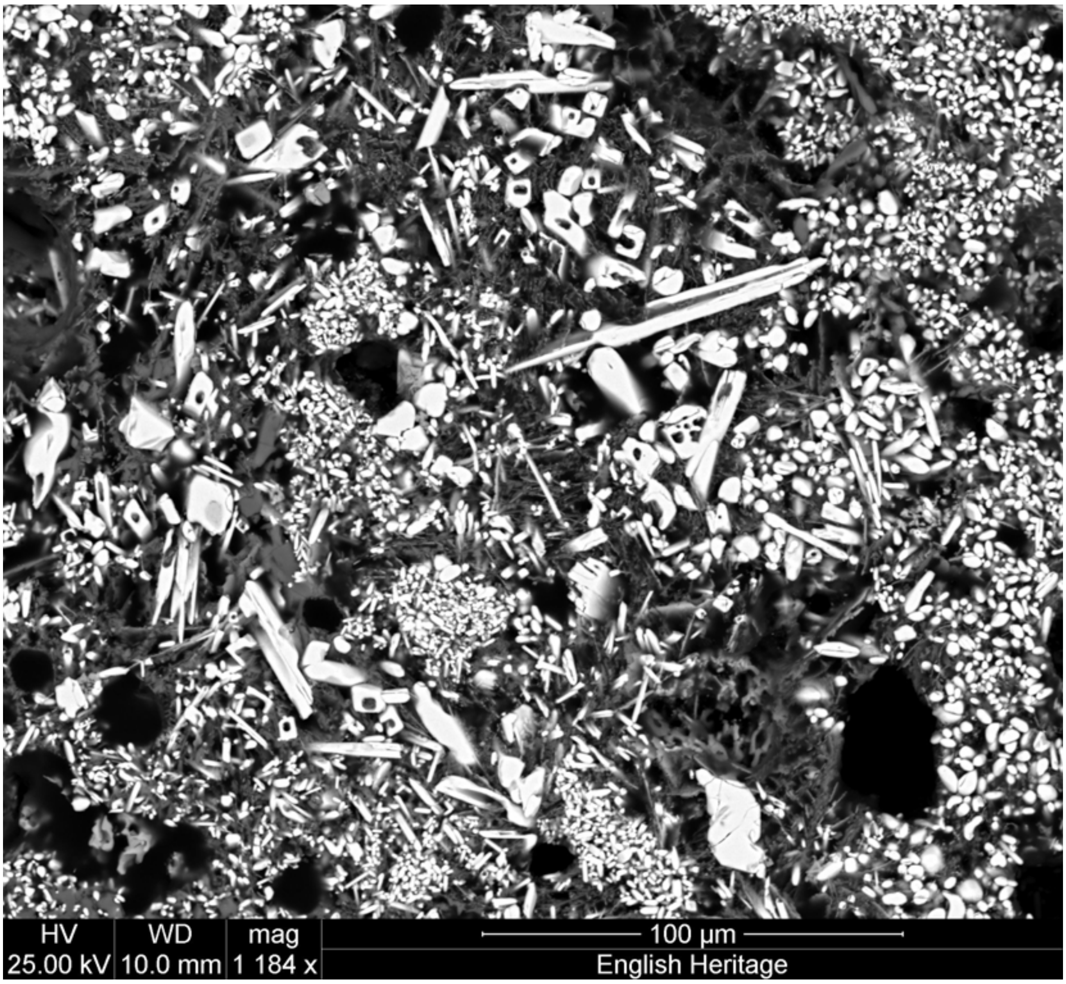

Figure 10 shows an SEM back-scatter image of cassiterite (bright white), there may originally have been some dendritic copper oxide (grey) present but most is likely to have leached out during burial

Metallic droplets were also found in the sample from 10005. The majority of these contained high concentrations of tin overall, largely ranging from 20 to 30wt%, but this is unlikely to be representative of the original melt. The values for tin detected in the droplets are enhanced relative to their original values due to corrosion, which has resulted in the copper being depleted (Dungworth 2001, Scott 1991). Traces of lead and sometimes arsenic were occasionally detected in the droplets; one copper droplet contained significant quantities of antimony. Small amounts of nickel were often present. Zinc was detected in bulk analyses of the crucible surface but rarely in the metallic droplets.

The alloy melted in the crucibles was probably bronze, typical of the Iron Age (Dungworth 2001). The detectable zinc and lead on the surfaces of the crucibles, however, may indicate small amounts of these metals were also present in the alloys (although both are volatile and so very small amounts in the metal melted may have given rise to disproportionately large amounts in the crucibles). If alloys containing zinc were in circulation, this would suggest that the crucibles were used in the Late Iron Age, perhaps the 1st century AD, when increasing amounts of zinc-containing alloys, like brass, were introduced from the Roman Empire. The presence of nickel may also suggest a later date (Dungworth 2001).

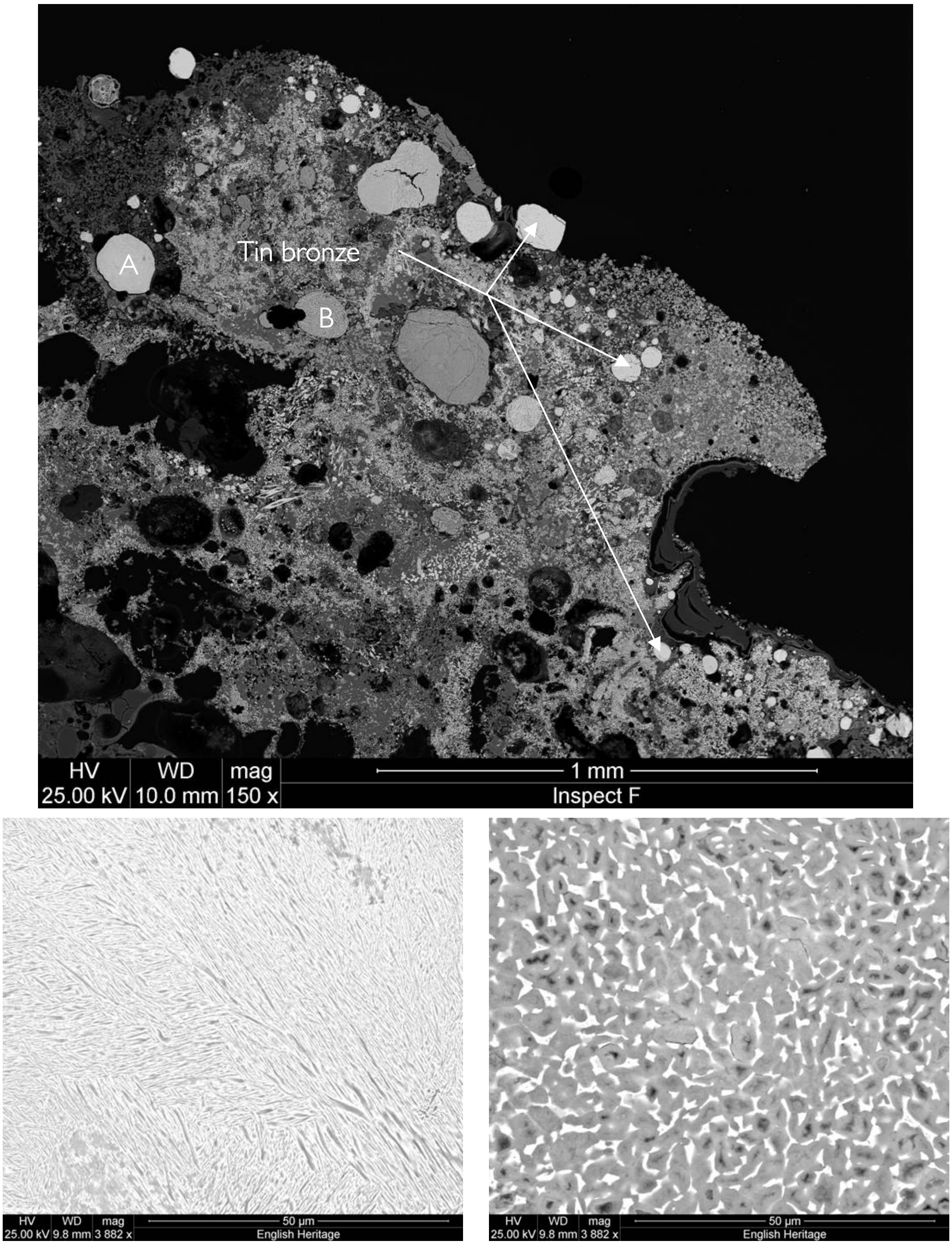

Figure 11 shows top: an SEM back-scattered electron image of the vitrified surface of 10005 with abundant metallic droplets (light grey). Below: an SEM back-scattered images of two droplets from 10005. A. (left) Tin-rich droplet with needle-like microstructure and B. (right) corroded dendritic structure showing tin-rich areas (light grey) and leached areas (dark grey) that were originally copper-rich

CONCLUSIONS

In conclusion, three out of the four vessels submitted for assessment were used as crucibles; the function of the thumb pot is unknown. Visual and compositional analysis have shown that the fabrics of these crucibles were similar to one another, being refractory and comprised largely of quartz in a matrix of iron-rich clay. The fabrics are quite similar to those of later Roman crucibles from Housesteads Fort, Northumberland (Dungworth 2001).

The Park Farm East crucibles were all employed for the melting of copper alloys, specifically tin bronzes, which contained small amounts of nickel and possible lead and zinc as well. This type of alloy composition suggests a Late Iron Age date (1st century AD). A more precise alloy composition cannot be determined from the evidence retained in the crucible fabric, due to differential volatilisation and oxidation of the alloying elements and subsequent corrosion.

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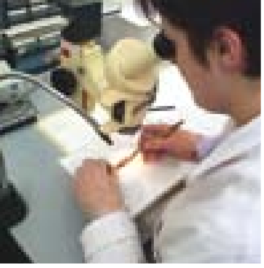
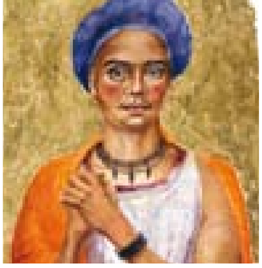
APPENDIX

Table 1: SEM EDS analyses of the crucible fabrics

Crucible	Analysis N°	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	SO_3	K_2O	CaO	TiO_2	MnO	Fe_2O_3
10005	1	0.36	0.41	8.33	82.65	0.44	<0.2	1.27	0.23	0.67	0.08	5.51
	2	0.51	0.77	12.10	75.18	0.45	<0.2	1.49	0.17	0.90	0.10	8.25
	3	0.35	0.57	11.02	79.24	0.34	<0.2	1.39	0.23	1.02	0.00	5.76
	4	0.32	0.50	9.87	78.77	0.41	<0.2	1.29	0.28	0.80	0.35	7.44
	5	0.27	0.60	9.86	79.42	0.34	<0.2	1.09	0.30	0.67	0.19	7.32
	6	0.26	0.42	7.74	83.91	0.63	<0.2	1.03	0.17	0.68	0.04	5.07
	7	0.35	0.55	9.82	79.86	0.44	<0.2	1.26	0.23	0.79	0.13	6.56
	Mean	0.36	0.41	8.33	82.65	0.44	<0.2	1.27	0.23	0.67	0.08	5.51
10007	8	0.33	0.51	9.40	81.48	0.26	<0.2	1.13	0.21	0.75	0.10	5.75
	9	0.46	0.50	10.18	78.46	0.47	<0.2	1.61	0.23	0.79	0.17	7.06
	10	0.78	0.80	13.92	64.58	0.64	0.2	1.99	0.47	1.05	0.20	15.43
	11	0.44	0.42	8.88	75.42	1.45	<0.2	1.43	0.29	0.73	0.08	10.75
	12	0.23	0.43	9.92	81.26	0.12	0.13	1.35	0.22	0.88	0.03	5.43
	13	0.75	0.68	11.01	74.47	0.77	<0.2	1.66	0.30	0.81	0.24	9.27
	Mean	0.64	0.76	12.18	72.58	0.70	<0.2	1.94	0.49	0.80	0.22	9.72
10032	14	0.84	0.42	8.82	82.06	0.32	1.30	1.69	0.28	0.81	0.11	3.35
	15	0.65	0.47	8.32	82.47	0.52	0.87	1.14	0.28	0.82	0.09	4.37
	16	0.45	0.53	9.44	82.06	0.19	1.36	1.36	0.24	0.91	0.00	3.46
	17	0.70	0.45	10.68	79.58	0.47	1.28	1.39	0.30	0.99	0.00	4.16
	18	0.55	0.49	9.84	80.81	0.23	1.4	1.4	0.3	0.9	0.1	4.0
	19	0.87	0.40	8.75	83.31	0.20	0.6	1.8	0.4	0.7	<0.1	2.9
	20	0.77	0.56	9.03	82.17	0.26	1.03	1.48	0.32	0.78	0.01	3.59
	21	0.40	0.35	7.45	84.31	0.28	1.34	1.19	0.20	0.75	0.07	3.65
	Mean	0.65	0.46	9.04	82.10	0.31	1.15	1.43	0.28	0.83	0.05	3.69



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