

GUNTER'S WOOD, HAMBLEDON, SURREY EXAMINATION OF GLASSWORKING DEBRIS

TECHNOLOGY REPORT

David Dungworth



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Gunter's Wood, Hambledon, Surrey

Examination of glassworking debris

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SUMMARY

The examination of six samples of glassworking waste from a Wealden glass production site has shown that the glass and the crucibles have compositions which suggest that the site was in operation before the arrival of French glassmakers in the late 16th century.

ACKNOWLEDGEMENTS

I would like to thank Mary Alexander, Curator Guildford Museum for providing access to the materials examined and permission to sample for scientific analysis.

ARCHIVE LOCATION

The glassworking debris is held by Guildford Museum, Castle Arch, Guildford, Surrey, GU1 3SX. The samples taken for scientific analysis are held by English Heritage, Fort Cumberland, Portsmouth, PO4 9LD

DATE OF RESEARCH

2006–2010

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INTRODUCTION

Gunter's Wood is the likely site of a medieval furnace used for the manufacture of glass (Kenyon 1967, 153). The site was discovered in 1959 and reported to Guildford Museum. No structural remains of a furnace had been discovered but quantities of glassworking debris, including crucibles, had been recovered which indicate that glass manufacture probably took place. The glass from the site has been described as 'unmistakably Early' (Kenyon 1967, 193). The analysis of the glassworking debris from this site contributes to the Wealden Glass Industry Project, funded by English Heritage (Historic Environment Enabling Programme Project Number 5299) and undertaken by the Surrey County Archaeological Unit.

THE GLASSWORKING DEBRIS

The only material held by Guildford Museum which can be positively identified as having been collected from Gunter's Wood in 1959 comprises a moil (the ring of glass between a blown artefact and the blowing iron, Figure 1), two amorphous lumps of glassy waste (Figure 2) and three crucible fragments (Figure 3) (Table 1).

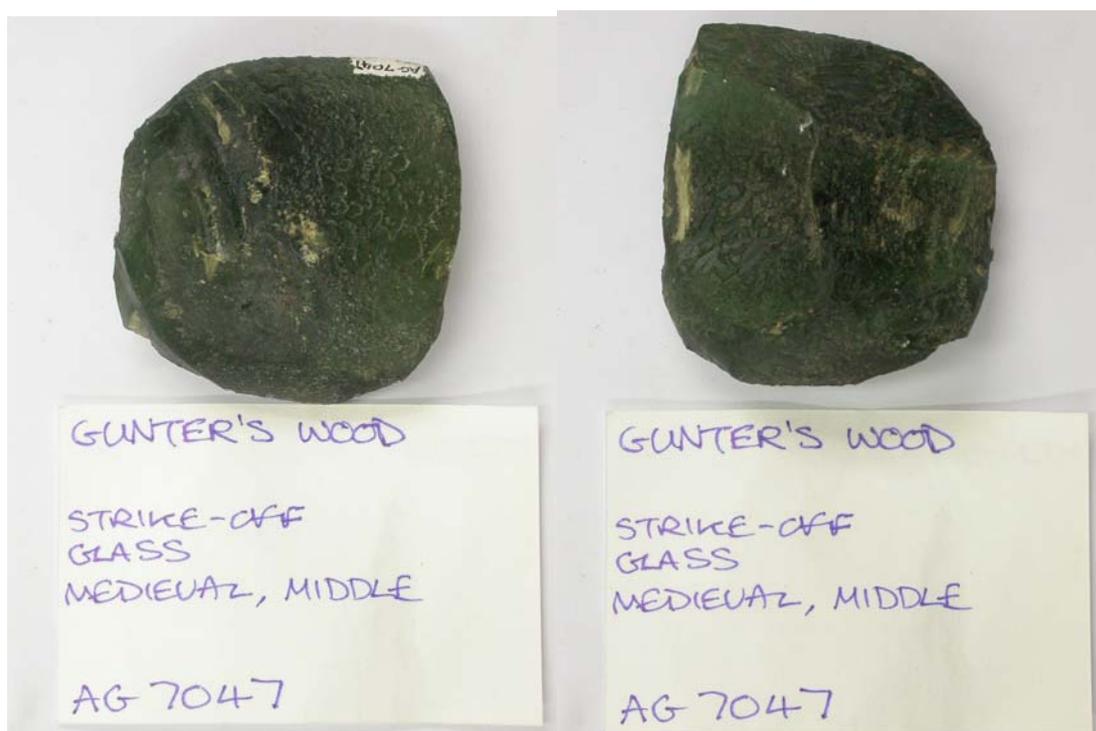


Figure 2. Exterior (left) and interior (right) views of the moil fragment (sample 2).



Figure 2. Amorphous glass waste samples 4 (bottom right) and 5 (top left)

The crucibles fragments are well fired with a cream-buff colour. The size and shape of the crucible fragments are comparable to the barrel-shaped crucibles from Blunden's Wood (Wood 1965, Fig 8). Two of the crucibles have areas of surface vitrification and/or adhering glass; however, these layers are extremely thin (generally less than 1mm) and survived only in patches (Figure 3).

Table 1. Details of materials sampled

#	Accession	Description
1	AG763	Crucible base, with vitrified surfaces and/or adhering glass (interior and exterior surfaces)
2	AG7047	Glassworking waste, a moil
3	AG7046	Crucible rim sherd with everted rim and vitrified surfaces and or adhering glass (interior and exterior surfaces)
4	AG7048	Amorphous glassworking waste
5	AG7048	Amorphous glassworking waste
6	AG764	Crucible rim sherd.



Figure 3. The exterior surface of one of the crucible fragments (sample 3; height 80mm)

METHODS

All of the fragments of glassworking debris were mounted in epoxy resin then ground and polished to a 1-micron finish to expose a cross-section. The samples were inspected using an optical microscope (brightfield and darkfield illumination) to identify corroded and uncorroded regions. All of the Gunter's Wood samples exhibited corroded surfaces. Where possible, the samples were analysed using two techniques to determine chemical composition: SEM-EDS and EDXRF. The energy dispersive X-ray spectrometer (EDS) attached to a scanning electron microscope (SEM) provided accurate analyses of a range of elements (especially where $Z < 23$) while the energy dispersive X-ray fluorescence spectrometer provided improved sensitivity (ie limits of detection) for many minor elements (especially where $Z > 23$) due to improved peak to background ratios.

The SEM used was a FEI Inspect F which was operated at 25kV with a beam current of approximately 1.2nA. The X-ray spectra generated by the electron beam were detected using an Oxford Instruments X-act SDD detector. The quantification of detected elements was achieved using the Oxford Instruments INCA software. The EDS spectra

were calibrated (optimised) using a cobalt standard. Deconvolution of the X-ray spectra and quantification of elements was improved by profile optimisation and element standardisation using pure elements and compounds (MAC standards). The chemical composition of the samples is presented in this report as stoichiometric oxides with oxide weight percent concentrations based on likely valence states (the exception being chlorine which is expressed as element wt%).

Table 2. Minimum Detection limits (MDL) and analytical errors for each oxide

	SEM-EDS			EDXRF	
	MDL	Error		MDL	Error
Na ₂ O	0.1	0.1	V ₂ O ₅	0.02	0.03
MgO	0.1	0.1	Cr ₂ O ₃	0.02	0.03
Al ₂ O ₃	0.1	0.1	MnO	0.02	0.03
SiO ₂	0.1	0.2	Fe ₂ O ₃	0.02	0.03
P ₂ O ₅	0.1	0.1	CoO	0.02	0.02
SO ₃	0.1	0.1	NiO	0.02	0.03
Cl	0.1	0.1	CuO	0.03	0.01
K ₂ O	0.1	0.1	ZnO	0.02	0.01
CaO	0.1	0.1	As ₂ O ₃	0.02	0.01
TiO ₂	0.1	0.1	SnO ₂	0.1	0.05
BaO	0.2	0.1	Sb ₂ O ₅	0.15	0.07
			Rb ₂ O	0.005	0.005
			SrO	0.005	0.005
			ZrO ₂	0.005	0.005
			PbO	0.05	0.02

The EDXRF used was an EDAX Eagle II which was operated at 40kV with a current of 1 mA. The Eagle II was fitted with a glass capillary to focus the X-Ray beam on an area approximately 0.3mm in diameter. This meant that it was possible to obtain EDXRF data for the bulk composition of the samples but not for the 'linescans' taken through the vitrified surfaces and/or adhering glass of the crucible samples.

The accuracy of the quantification of all oxides (both SEM-EDS and EDXRF) was checked by analysing a wide range reference materials (Corning, NIST, DGG and Newton/Pilkington). A number of elements were sought but not detected: vanadium, chromium, cobalt, nickel, copper, arsenic, tin, antimony and barium.

RESULTS

The bulk compositions of the samples given in Tables 3 and 4 represent a combination of SEM-EDS and EDXRF data. The samples displayed varying degrees of homogeneity and the data in Tables 3 and 4 represent the average obtained from analysing a number of different areas. The number of areas analysed depended on the degree of homogeneity/heterogeneity. Samples 2 and 4 were homogeneous with chemical composition of different areas varying by less than the analytical precision. Sample 5 and all the crucible samples (1, 3 and 6), however, were heterogeneous and required the analysis of at least 10 separate areas to obtain a reliable result.

The glassy samples (2, 4 and 5)

The glassy samples (2, 4 and 5) are all potassium-rich glasses which contain a wide range of other elements typical of medieval forest glasses (Barrera and Velde 1989; Dungworth and Clark 2004; Wedepohl 2003). The degree of variation between the three samples is considerable; it is greater than is usually seen among glassworking debris (Dungworth and Clark 2004). This variation could represent several different phases of glassworking, each with slightly different raw materials and/or recipes, or it could be the result of contamination of the glassworking waste by other materials (such as crucibles, furnace fabric, fuel vapour and/or fuel ash).

Table 3. Average chemical composition of the glassy samples

Sample:	2	4	5
Na ₂ O	2.57	2.36	1.97
MgO	6.42	7.79	5.45
Al ₂ O ₃	1.58	0.66	2.17
SiO ₂	56.6	54.2	60.2
P ₂ O ₅	2.92	3.67	2.62
SO ₃	0.31	0.32	0.14
Cl	0.56	0.49	0.26
K ₂ O	9.50	14.64	14.28
CaO	17.1	13.99	10.2
TiO ₂	0.19	<0.1	0.16
MnO	1.34	1.18	0.87
Fe ₂ O ₃	0.68	0.55	1.54
ZnO	0.05	0.05	0.04
Rb ₂ O	0.022	0.023	0.026
SrO	0.116	0.076	0.072
ZrO ₂	0.023	0.015	0.017
PbO	0.08	<0.05	<0.05

Sample 2 can be regarded as the sample least likely to have suffered from any contamination as it is a fragment of a moil. The two amorphous lumps, however, could have been formed in a variety of ways and contamination by other materials cannot be ruled out. The low concentration of aluminium in sample 2 suggests that it has not formed as a result of any reactions between glass and crucible fabric or elements of the furnace structure. Sample 5, however, shows the highest iron and aluminium concentrations of all three glassy samples. Sample 5 is also heterogeneous and contains some mineral phases, including unreacted silica as well as wollastonite (CaSiO_3) and apatite ($\text{Ca}_5\text{P}_3\text{O}_{12}\text{Cl}$) which had crystallised from the molten glass (Figure 4). The chemical composition and microstructure of this sample suggests that it formed as a result of reactions between glass and some other materials. It does not accurately represent the nature of any glass artefacts that might have been manufactured at Gunter's Wood.

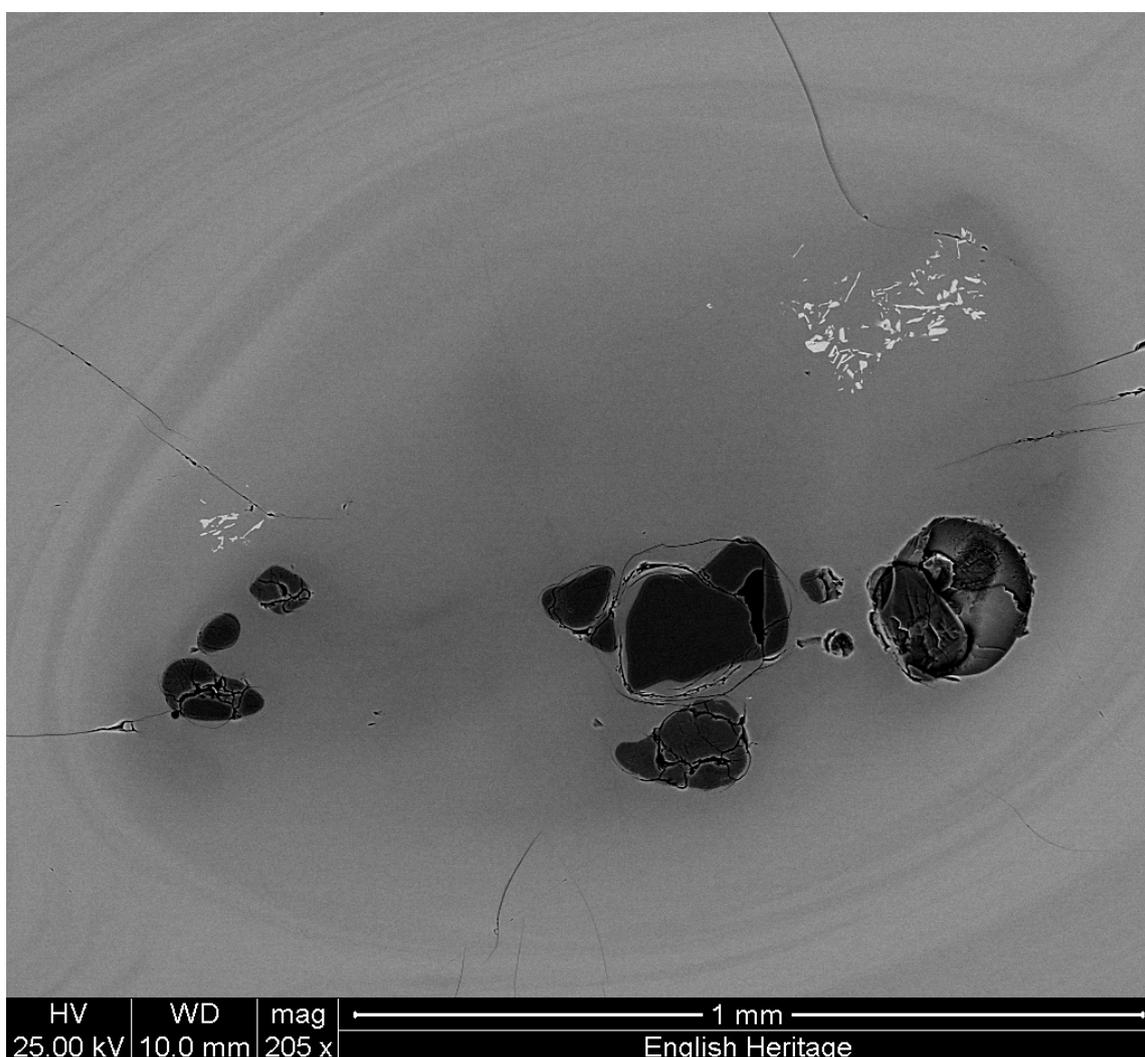


Figure 4. SEM image of sample 5 (back-scattered electron detector, brightness is proportional to average atomic number). The rounded dark patches in the centre of the images are silica while the small bright areas near the top right are crystals of wollastonite

The crucibles

The three crucible fragments share almost identical microstructures (Figure 5) and very similar chemical compositions (Table 4). The fabric of the crucibles comprises porosity, small silica grains (100–200 microns diameter) and a vitrified ceramic matrix (Figure 5). The vitrified matrix contains occasional recrystallised silica and mullite.

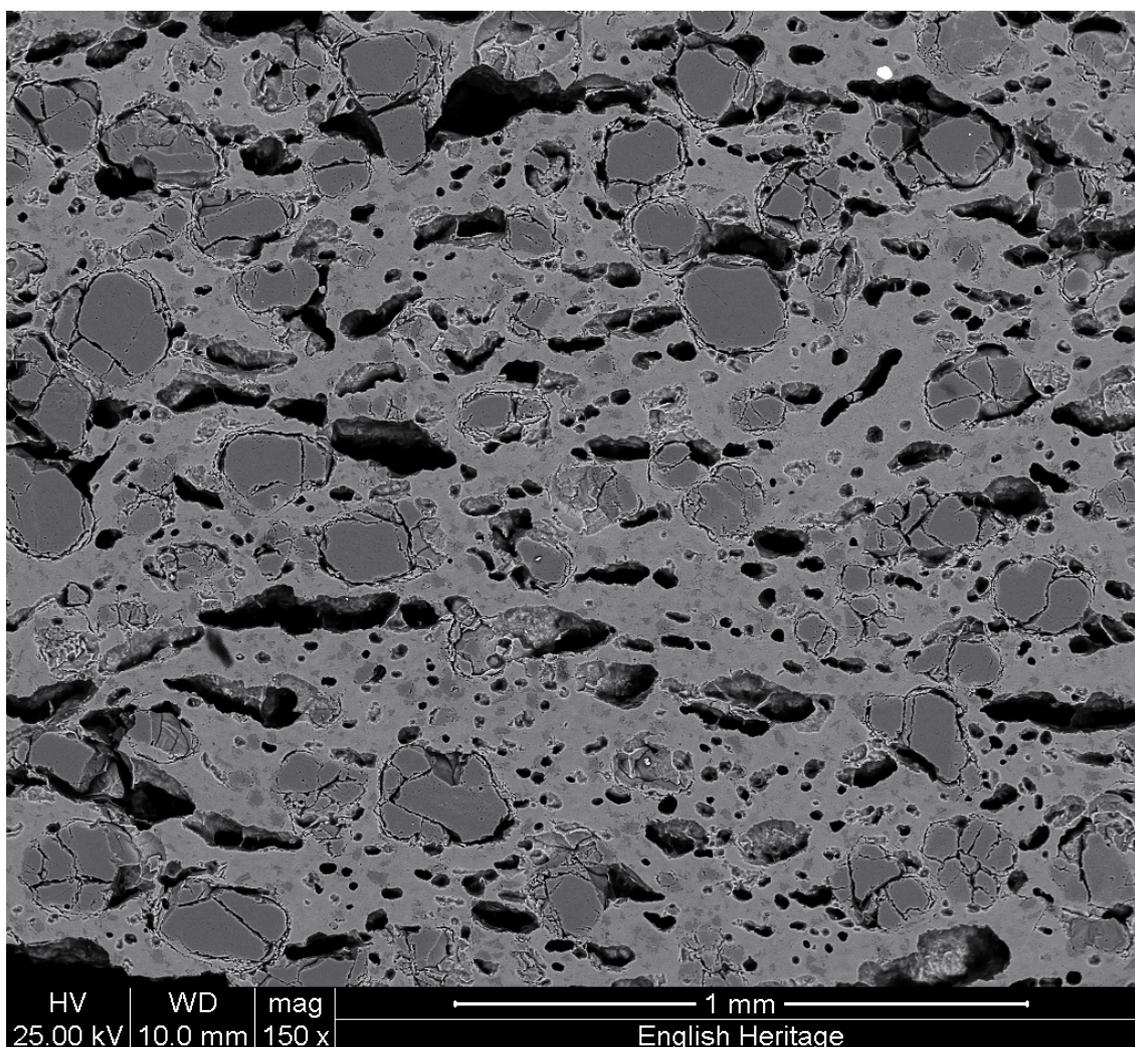


Figure 5. SEM image of sample 3 (back-scattered electron detector, brightness is proportional to average atomic number). The black areas are porosity, the dark grey areas are silica and the remaining light grey areas are the vitrified ceramic

Compared to somewhat later crucibles reported elsewhere (Dungworth 2008) the Gunter's Wood crucibles have rather thin (and patchy) layers of surface vitrification and/or adhering glass (Figure 3). In this respect, however, the Gunter's Wood crucibles resemble other medieval crucibles from the Weald (Dungworth and Paynter in preparation).

Table 4. Average chemical composition of the ceramic fabric of the crucibles

Sample:	1	3	6
Na ₂ O	0.25	0.15	0.12
MgO	0.72	0.74	0.61
Al ₂ O ₃	18.18	17.56	13.3
SiO ₂	75.4	75.4	81.2
P ₂ O ₅	0.15	0.16	0.15
SO ₃	<0.1	<0.1	<0.1
Cl	<0.1	<0.1	<0.1
K ₂ O	2.14	2.17	1.77
CaO	0.36	0.41	0.34
TiO ₂	0.78	0.84	0.7
MnO	<0.02	<0.02	<0.02
Fe ₂ O ₃	1.92	2.43	1.51
ZnO	<0.02	<0.02	<0.02
Rb ₂ O	0.016	0.022	0.02
SrO	0.064	0.104	0.088
ZrO ₂	0.054	0.042	0.039
PbO	<0.05	<0.05	<0.05

Crucible 3 had small patches of glass/vitrification adhering to both its interior and exterior surfaces as well as on the rim. The composition of these areas was investigated by undertaking a series of analyses through the glass/vitrification into the underlying ceramic fabric (Figures 6–7, see Appendices). The results confirm some of the observations made previously on post-medieval crucibles used to melt high-lime low-alkali (HLLA) glass (Dungworth 2008). In particular, elements which are normally found in high concentrations in the crucible fabric have tended to diffuse into the adhering glass/vitrification. Aluminium (Figure 6) and titanium concentrations in the adhering glass/vitrification are consistently higher (5–10wt% Al₂O₃) than the glassworking waste (0.7–1.6wt% Al₂O₃). In addition, potassium usually shows a maximum concentration within the adhering glass/vitrification but 0.1–0.3mm from the crucible-glass interface (Figure 7). This is comparable to the interaction zone observed in post-medieval crucibles used to melt HLLA glass (Dungworth 2008).

The interactions between glass and crucible (as well as possible contributions from fuel ash and vapour) have changed the composition of any adhering glass such that its analysis provides little direct information about the nature of the glass melted in these crucibles. The investigation of post-medieval crucibles used to melt HLLA glass (Dungworth 2003; 2008) showed that the chemical composition of glass adhering to interior surfaces shared many similarities with contemporary glassworking waste from the same site (notwithstanding the contamination from the crucible fabric). The exterior surfaces of the crucibles used to melt HLLA glass, however, usually had chemical compositions which were substantially different to the interior surfaces.

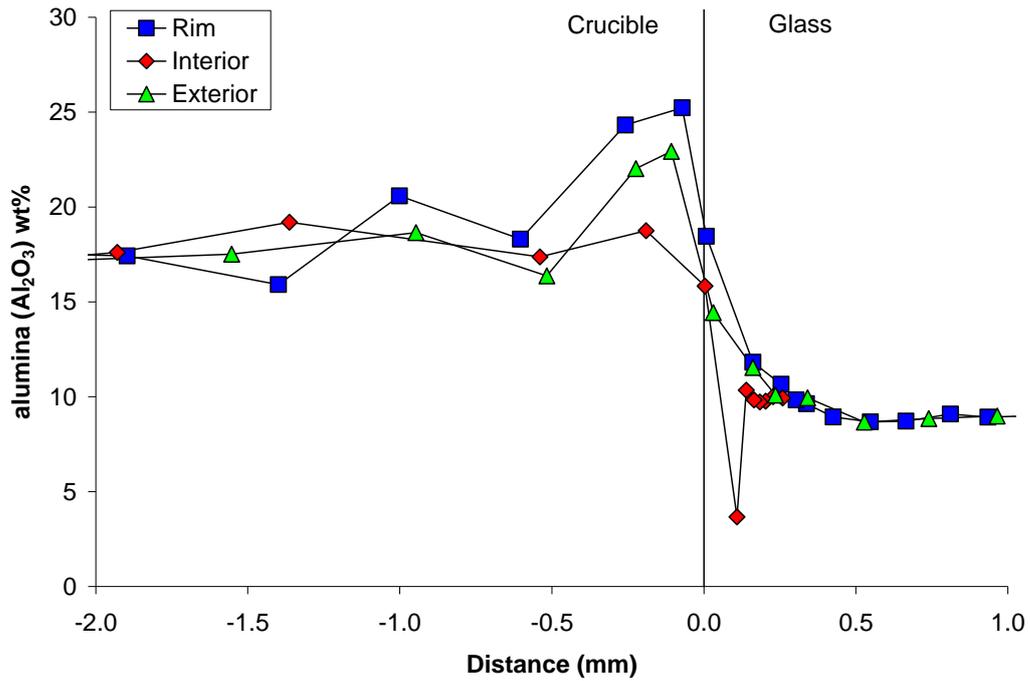


Figure 6. Linescan showing changes in aluminium concentration through the ceramic fabric and surface vitrification/adhering glass of crucible 3

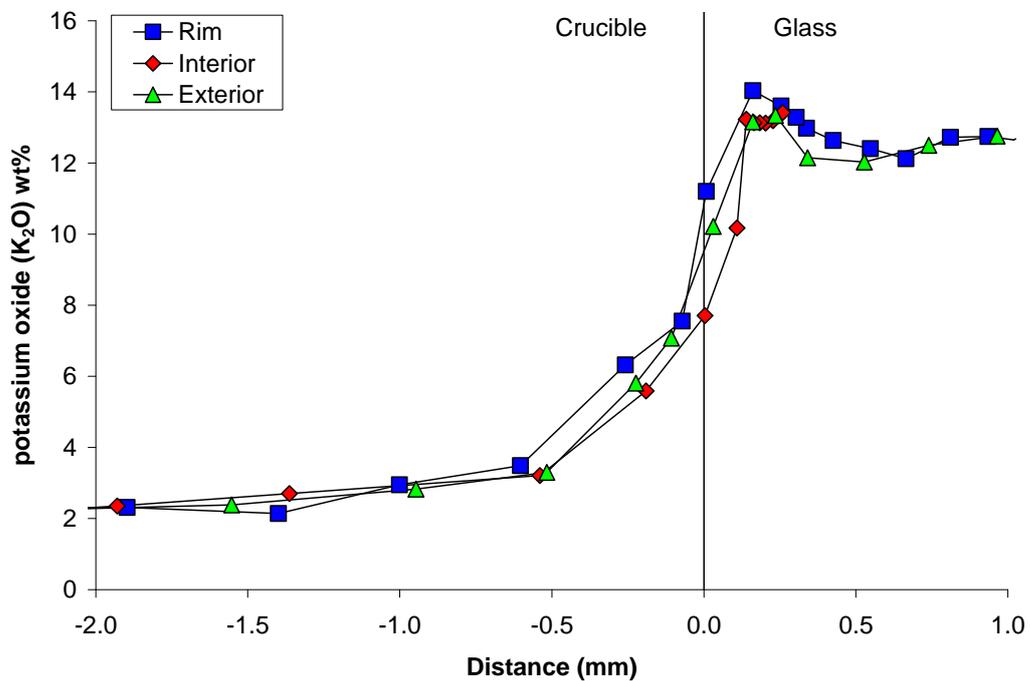


Figure 7. Linescan showing changes in potassium concentration through the ceramic fabric and surface vitrification/adhering glass of crucible 3

These exterior surfaces contained relatively low concentrations of many elements characteristic of the glass which was melted in them. The exterior surfaces were usually enriched in aluminium and iron; this was interpreted as deriving from the coal fuel used in the furnaces in which the crucibles were heated. The Gunter's Wood crucibles, however, have interior and exterior surfaces which are chemically similar. This similarity probably reflects the use of plant ashes as a source of alkalis in glassmaking and the use of organic fuel to heat the furnaces.

CONCLUSIONS

The analysis of the small assemblage of glassworking waste from Gunter's Wood has shown that the glass produced was a forest glass. This type of glass appears to have been manufactured in England from at least the 14th century until the arrival of French glassmakers in the late 16th century (Dungworth and Clark 2004). The French glassmakers brought with them a number of technological developments one of which was the production of HLLA glass instead of forest glass. The nature of the glass produced at Gunter's Wood supports Kenyon's opinion that the glasshouse at Gunter's Wood operated before the arrival of the French glassmakers (Kenyon 1967, 193). The early date for Gunter's Wood is also supported by the nature of the crucibles. A recent investigation of a range of Wealden glass-melting crucibles has shown that quartz-tempered crucibles are used on early sites while grog-tempered crucibles are used on late sites (Paynter forthcoming).

The similarities in the chemical composition of the glass/vitrification of the interior and exterior surfaces of the crucibles is noteworthy but should be interpreted carefully. It might be argued that the vitrification of the outer surface of a glass-melting crucible reveals something of the nature of the fuel used in the furnace. The similarities between the interior and exterior surfaces could then be cited as evidence that the same organic material was used as both fuel to heat the furnace and as a source of alkalis for the glass. The crucibles, however, would not have been in direct contact with the fuel and the vitrification of the exterior surfaces will have been due principally to reactions between the crucible and volatile components deriving from the fuel. Therefore, the concentration of elements in the exterior vitrified surfaces will not exactly correspond to the whole plant ash composition.

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