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FLINT LODGE, 2 VILLERS ROAD, SOUTHSEA
ANALYSIS OF WINDOW GLASS

TECHNOLOGY REPORT

David Dungworth and Roger Wilkes

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SUMMARY

The chemical analysis of twelve fragments of window glass from Flint Lodge was undertaken in an attempt to identify the composition of the glass originally installed during its construction (1851). Eleven fragments have compositions which are virtually identical to each other and are similar to previously analysed 19th-century glass. These samples probably represent the glass installed in 1851. The remaining fragment has a composition which suggests that it is a later replacement.

ACKNOWLEDGEMENTS

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ARCHIVE LOCATION

Fort Cumberland, Fort Cumberland Road, Eastney, Portsmouth, PO4 9LD

DATE OF RESEARCH

2009–10

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INTRODUCTION

The analysis of fragments of window glass from Flint Lodge, 2 Villiers Road, Southsea forms part of a much larger project undertaken to investigate the chemical composition of window glass produced and used in Britain during the past five centuries. Samples of window glass have been selected from archaeological excavations (including glass production sites) and from historic buildings. These have been analysed to determine their chemical composition. A comparison of the chemical composition with the available dating evidence shows that a series of changes in window glass manufacturing took place during this period. The aim of this research is to provide a technique to date the manufacture of individual panes of glass in historic buildings. This knowledge will allow architects and others to make more informed judgements about which glass to retain and which can be replaced (Clark 2001).

Almost all glass produced in Britain during the medieval period was produced using sand and terrestrial plant ashes (primarily bracken) and has a distinctive potassium-rich composition (Dungworth and Clark 2004). The arrival of French glassmakers in the late 16th century saw a change to a high-lime low-alkali (HLLA) glass. HLLA glass was probably made using sand and the ash of hardwoods (such as oak). This HLLA glass remained in use until the end of the 17th century when it was superseded by a glass made using sand and seaweed (kelp) ash (Dungworth *et al* 2009; Parkes 1823; Watson 1782). This kelp glass dominated the window glass industry until the early part of the 19th century when it was abandoned in favour of glass made using synthetic soda (Cooper 1835; Ure 1844; Muspratt 1860).

Nicholas Leblanc invented a process for the manufacture of synthetic soda at the end of the 18th century. Common salt was heated with sulphuric acid to produce sodium sulphate (soda saltcake), The sodium sulphate was then heated with lime and charcoal or coal to produce sodium carbonate. Initially, glass could only be made with sodium carbonate, but glassmakers soon discovered that the sulphate could be used directly if it was combined with charcoal or coal. Glass made for the century or so following the 1830s was a simple soda-lime-silica glass with low levels of impurities (Dungworth 2009).

The early decades of the 20th century saw the development of techniques for automatically drawing glass (Cable 2004; McGrath and Frost 1937; Turner 1926) which initially had problems with glass devitrifying. These problems were solved by substituting a small amount of magnesia for lime and virtually all window glass made in Britain since 1930 has contained 2–5% magnesia (Smrcek 2005).

THE GLASS

The construction of Flint Lodge in 1851 by the architect Thomas Ellis Owen (1805–1862) is commemorated by a plaque attached to one of the walls of the house (Figure 1). Recent refurbishment of the property provided the opportunity to examine window glass from two rooms (known as the Tack Room and the Laundry Room).

Figure 1. Flint Lodge, 2 Villers Road, Southsea showing the plaque commemorating the construction date

METHODS

All of the fragments of glass were mounted in epoxy resin then ground and polished to a 3-micron finish to expose a cross-section through the glass. The samples were inspected using an optical microscope (brightfield and darkfield illumination) to identify corroded and uncorroded regions. None of the Flint Lodge samples exhibited any substantial corroded surfaces. 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 while the energy dispersive X-ray fluorescence (EDXRF) spectrometer provided improved sensitivity and accuracy for some minor elements (in particular manganese, iron, arsenic, strontium and zirconium) 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%). The accuracy of the quantification of all oxides was checked by analysing a wide range reference materials (Corning, NIST, DGG and Newton/Pilkington). A number of elements were sought but not detected: phosphorus, chlorine, titanium, vanadium, chromium, cobalt, nickel, copper, zinc, arsenic, tin, antimony, rubidium, barium and lead.

Table 1. Minimum Detection limits (MDL) and analytical errors for each oxide

	SEM-EDS			EDXRF	
	MDL	Error		MDL	Error
Na_2O	0.1	0.1	V_2O_5	0.02	0.03
MgO	0.1	0.1	Cr_2O_3	0.02	0.03
Al_2O_3	0.1	0.1	MnO	0.02	0.03
SiO_2	0.1	0.2	Fe_2O_3	0.02	0.03
P_2O_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	0.02	0.01
K_2O	0.1	0.1	ZnO	0.02	0.01
CaO	0.1	0.1	As_2O_3	0.01	0.01
TiO_2	0.1	0.1	SnO_2	0.1	0.05
BaO	0.2	0.1	Sb_2O_5	0.15	0.07
			Rb_2O	0.005	0.005
			SrO	0.005	0.005
			ZrO_2	0.005	0.005
			PbO	0.02	0.02

RESULTS

Eleven samples of the window glass from Flint Lodge share virtually identical chemical compositions (Table 2) and were almost certainly made at the same time and place. One sample (LR05) has a chemical composition which suggests that it is a later replacement.

Table 2. Chemical composition of the Flint lodge window glass

	Na_2O	MgO	Al_2O_3	SiO_2	SO_3	CaO	MnO	Fe_2O_3	As_2O_3	SrO	ZrO_2
TR01	11.52	0.13	0.49	71.4	0.56	14.74	<0.02	0.160	0.021	0.018	<0.005
TR02	11.43	<0.1	0.57	71.5	0.56	14.82	<0.02	0.158	0.026	0.016	<0.005
TR03	11.43	0.14	0.56	71.5	0.58	14.89	<0.02	0.159	0.021	0.018	<0.005
TR04	11.49	0.12	0.53	71.7	0.58	14.81	<0.02	0.164	0.027	0.020	<0.005
TR05	11.53	0.13	0.55	71.5	0.58	14.93	<0.02	0.159	0.029	0.019	<0.005
TR06	11.42	0.11	0.53	71.3	0.62	14.95	<0.02	0.160	0.025	0.020	<0.005
TR07	11.43	0.14	0.49	71.8	0.58	15.01	<0.02	0.163	0.023	0.022	<0.005
LR01	11.83	0.14	0.56	71.6	0.56	14.74	<0.02	0.161	0.015	0.016	<0.005
LR02	11.73	0.14	0.54	71.3	0.56	14.81	<0.02	0.161	0.022	0.019	<0.005
LR03	11.56	0.10	0.57	71.3	0.61	14.99	<0.02	0.161	0.030	0.018	<0.005
LR04	11.70	0.14	0.51	71.3	0.56	14.90	<0.02	0.162	0.028	0.017	<0.005
LR05	15.04	2.93	0.39	71.5	0.35	9.44	0.05	0.100	<0.01	0.009	0.025

DISCUSSION

Eleven of the Flint Lodge glass samples (all except LR05) have compositions which are very similar to those of other 19th-century windows (Table 3) and probably represent the glass installed in 1851 during the building's construction. This soda-lime glass would have been made using synthetic soda obtained using the Leblanc process. The relatively high sulphur content of this glass suggests that sodium sulphate was used in preference to sodium carbonate (the former being substantially cheaper). The small proportion of arsenic detectable in the original Flint Lodge window glass is of considerable interest. Arsenic appears to have been deliberately added to glass manufactured 1837–40 by Chance Brothers for the Chatsworth Conservatory (Dungworth 2009). In addition arsenic has been found in glassworking debris from Nailsea (Hatton 2004), although this cannot be more precisely dated than 1830 to 1870. Arsenic has not been detected in any of the late 19th-century window glass analysed so far and so it appears to have largely gone out of use around the middle of the century. The original Flint Lodge window glass contains around 0.02wt% and suggests that its use was already declining by 1851.

Table 3. Chemical composition of some 19th-century glass (1 = Dungworth 2009; 2= Hatton 2004; 3 = this report; 4 = Dungworth and Wilkes 2010 a; 5 = Dungworth and Wilkes 2010b; 6 = Dungworth 2010)

	Source	Date	Na_2O	MgO	Al_2O_3	SiO_2	SO_3	K_2O	CaO	Fe_2O_3	As_2O_3	SrO
Chatsworth	1	1837–40	14.0	<0.1	0.7	70.3	0.34	<0.1	14.1	0.20	0.41	0.015
Nailsea	2	1830–70	13.1	0.2	0.8	68.9	0.60	0.1	13.5	0.33	0.22	0.022
Flint Lodge	3	1851	11.6	0.1	0.5	71.5	0.58	<0.1	14.9	0.16	0.02	0.019
Wentworth 1	4	1877	11.9	0.4	0.7	71.5	0.24	0.3	14.3	0.28	<0.02	0.026
Highland House	5	1880s	12.1	0.1	1.5	71.7	0.29	0.6	13.2	0.21	<0.02	0.010
Welch Road	6	1895	11.6	0.1	1.5	72.5	0.30	0.6	13.1	0.20	<0.02	0.019

Sample LR05 does not share the same composition as the other Flint Lodge samples and it is likely to be a later replacement. Its relatively high magnesium content indicates that it was probably made after flat glass drawing techniques were introduced c1930 (Cable 2004; Turner 1926). The low iron content of LR05 is also consistent with manufacture in the 20th century. LR05 also contains much more zirconium than most 19th- and 20th-century flat glass. While relatively high zirconium concentrations (0.02–0.07wt% ZrO_2) are encountered in many 17th- and 18th-century window glasses, it is present at very low concentrations (<0.01wt% ZrO_2) in most later window glass. Zirconium in early glass probably derives from the sand used, but in later glass it could derive in part from the use of zirconia refractories (in commercial use from the 1950 onwards, Michael Cable personal communication).

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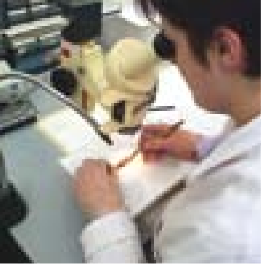
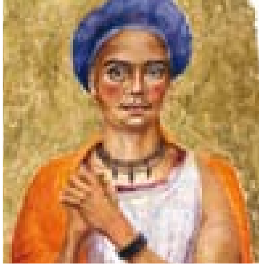



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