Compositional Imaging and Analysis of Late Iron Age Glass from the Broborg Vitrified Hillfort, Sweden

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Numerous Iron Age hillforts were constructed throughout Europe on high ground to serve ancient settlements [1]. The edifice walls of a small fraction of hillforts were vitrified as a result of high temperature activity, [2]. Swedish hillfort glasses from the Broborg Site near Uppsala, Sweden have recently been proposed as an analogue material to inform long term nuclear waste storage (Fig. 1A) [3]. As part of that effort, a fragment of the Broborg hillfort wall was embedded and polished prior to examination by x-ray methodologies to determine its composition and microstructure (Fig. 1B).

A Bruker M4 Tornado with dual Bruker XFlash 6|60 detectors was used to collect hyperspectral micro-XRF data with a Rh source and a 20 um (Mo K_{α}) polycapillary optic at two energies: 1) 25 keV/no filter, and 2) 40 keV with a 12.5 µm Al source filter [4]. An FEI Apreo scanning electron microscope (SEM) was used to collect an electron image montage spanning the specimen's polished surface, and electron beam-excited hyperspectral x-ray data was collected using dual Bruker XFlash 6|60 detectors at 15 keV.

Object-scale major element and electron imagery reveals the specimen is comprised of 3 principal chemical phases, an Fe-rich glass (dark), an Fe-poor glass rich in alkalis (milky), and quartz (Fig. 2A,E). Trace element imaging depicts unreacted zircon throughout the sample, Zr and V enrichment in the Fe-rich glass (Fig. 2B-C), and Rb enrichment in the Fe-poor glass (Fig. 2D). A more detailed view of the boundary between the 2 glass zones shows heavily embayed quartz, residual CaAl-silicate zones within the Fe-poor glass, FeMgAl spinel crystallization, and quench-crystallization in the Fe-rich glass (Fig. 3). Major element compositions of the glasses are broadly consistent with previous studies (Table 1) [4,5]. Importantly, the new trace element compositions of the glasses provide an opportunity to test a linkage with the bulk chemistry of lithologies found at Broborg to better understand the ancient melting processes via an elemental ratio comparison.

References:

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Figure 2. Micro-XRF elemental and electron imagery of vitreous wall fragment. A) Composite x-ray overlay (K K_{α}-red, Fe K_{α}-green, Si K_{α}-blue, Ca K_{α}-yellow). B-D) single element images in rainbow contrast scale Zr K_{α}, Rb K_{α}, and V K_{α}. E) Backscattered electron image (BSE) mosaic, highlighted area enlarge below. F) Reflected light color image.



Figure 3. Micro-XRF composite x-ray image superimposed on a BSE image showing the boundary between Fe-rich and -poor glasses [Fe K_{α}-red, Ca K_{α}-green, Si K_{α}-blue, and Zr K_{α}-majenta; quartz (qtz), anorthite (an), and spinel (spl)].

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Table 1 Major		glass	glass
and trace	SiO ₂	54.20	66.65
	AI_2O_3	14.69	20.12
element	CaO	6.42	0.83
analyses of	MnO	0.31	
Fe-rich and –	FeO*	11.33	
poor glasses.	TiO₂	2.26	
Major	MgO	3.73	
elements	Na ₂ O	3.84	5.18
determined	K ₂ O	2.15	7.18
by electron	P ₂ O ₅	0.71	
beam v_rav	Total	99.64	99.95
Dealin X-ray	v	279	
analysis (wt	Cr	27	
%), and trace	Mn		122
elements	Fe		1962
determined	Со	781	
hy micro-XRF	Ni		20
	Zn	362	
(ppm).	Ga	135	
	Rb	152	269
	Sr	972	449
	Y	203	
	Zr	1974	
	Ba	86	1517