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The technical characterisation of a single fragment of metallurgical waste from ……

Introduction
A sample of roughly plano-convex metallurgical slag (sample GB11/F61), probably broken off from a larger fragment, (see Figure 1) was presented to us for examination and analysis. The sample weighed 555gr and was 12.4cm along the long axis and 5.9cm along the short axis (width). It had a dark brown exterior with a black porous interior

Methodology

a. Sample preparation
The sample was sectioned and sub-sampled at two places (sub-samples GB11a and GB11b). Each of the sub-samples was mounted on a metallographic resin and ground and polished with a 6m and a 3 micron diamond paste and subsequently carbon coated for analysis with the scanning electron microscope attached to an energy dispersive analyser (SEM-EDAX).

b. Analysis
Metallurgical slags contain a number of distinct mineralogical phases which become apparent when the sample is examined with reflecting light. Analyses are undertaken first on the entire surface of the polished block, and subsequently on each of the different mineralogical phases. Each phase contains apart from the main constituents a suite of other minor and trace elements. Both sets of analyses are needed. The first type (taken over a mean of three) represents area or bulk chemical analysis and is considered to be representative of the composition of the artefact as a whole. As such it identifies the slag as a metallurgical slag of one kind or another ie ferrous versus non-ferrous. The second type is aimed at establishing the composition of each of the mineralogical phases within and so at identifying the process that generated it.

Bloomery slags whether smelting or smithing are characterised by a number of distinct mineralogical phases. These include dendrites of wustite (FeO), long or broken up needles of fayalite ($FeO.SiO_2$), angular grains of hercynite ($FeO.Al_2O_3$) and a glassy phase which grows interstitially within the other phases.

SEM-EDAX of sub-samples GB11a and GB11b

Area analysis of both sub-samples (see Table 1) revealed the artefact to be an iron bloomery slag (see Figures 2 and 3). They are both of the fayalitic type meaning an iron silicate, mildly magnetic, if at all. Dendrites of wustite, the brighter grey phase, are seen next to the darker grey long needles of fayalite. The darker grey interstitial phase actually consists of a eutectic of two phases (see Figure 3). Apart from the main constituents each phase contains a host of other elements in smaller quantities. Table 1 presents the mineralogical composition of each individual phase in this particular case wustite, fayalite and an interstitial glass. All three are formed while the slag cools from c 1050-1100 ^{0}C which is the temperature at which the fayalite is free running.

Closer examination of the composition of the mineralogical phases growing within the "glass" revealed other phases as well, which appear to have been formed on account of the localised high percentage of particular elements. Figure 3 shows a section of GB11b with a variety of "non-conventional" mineralogical phases like that represented by spot analysis X1, representing a potassium alumino-silicate (see Table 1) and X5 corresponding to an unusual barium/phosphorus rich phase appearing black on the SEM-BS image (see Figure 3b). The rest of the phases in Figure3b are X2, a fayalite, X3 the glassy component and X4 which corresponds to the iron rich wustite component of the interstitial material, a eutectic of the iron oxide and the glass;

Discussion

Iron slag in the context of a pre industrial bloomery process is broadly speaking the product of either the smelting or smithing cycle. The first encompasses all stages associated with the forming and shaping of the bloom to a workable piece of iron, the second with the shaping of the iron billet or bar to an artefact of a particular composition ie a composite of iron and/or steel. Both processes generate slag which, allowing for the composition of the ore, is either of the iron oxide or the fayalitic type.

Given that the majority of bloomery slags found in association with blomery mounds or "smithies" are amorphous spongy-looking, non or mildly magnetic materials of varying size, weight and density, the key question to address has been whether they derive from the smelting or the smithing process. Mineralogy is a means of differentiating between the two processes. The primary waste from the smithing cycle is hammer scale, magnetite or other iron oxides, formed in the process of forging or welding of two strips of iron/steel. In the course of welding, and in order to remove the iron oxide layer, sand is usually added; this creates an iron silicate or fayalite slag which is removed as the smith welds the surfaces together. Remnants of this slag can be seen clearly in the context of slag stringers on either side of the weld line. The large majority falls into the hearth and creates the fayalite-rich smithing heath bottoms.

The sample examined here is of the smelting type on account of the variety of the mineralogical phases present, their composition which reflects the ore, and the slow rate of cooling allowing for the formation of "hallos" or intermediary phases between the main phases and the interstitial glass. The presence of barium-derived from barite ($BaSO_4$)- within the glassy phase suggests the exploitation of a small hematite vein deposit, perhaps originating in the Ochils area. - Hematite deposits are relatively rare in Scotland although kidney ore of the Cumberland type is present in Muirkirk, Southern Uplands (Hall and Photos-Jones 1999) and at other localities in small veins in the South Highlands. The presence of small amounts of manganese and phosphorus may reflect mixing of this hematite with bog ore. Most Scottish iron ores are iron rich and self fluxing.

Morphologically and from the point of view of the analytical data, the GB11 sample resembles bloomery slags from Taras Farm by Forres, Invernesshire (Will 1999; Photos-Jones 2000). These slags derived from a bowl furnace dug into the soil, consisting of a stone wall at three sides. The slag was of the non-tapping variety and quite viscous, the bloom forming below the tuyere and both bloom and slag removed at the end of the smelting cycle. Within such a construction, the slag would be accumulating at the bottom of the furnace bowl. The Forres furnace dates to c. the middle of the 2nd century BC (cal BC 378-cal AD17, 2σ). This type of slag is in contrast to those derived from Highland bloomery mounds (Photos-Jones et al 1998) which are of the tapping type mainly on account of their low viscosity. The high levels of manganese in association with iron oxide " lost" in the slag contributes to them being molten and running freely out of the furnace at relatively lower temperatures. The Highland bloomery mounds furnace design rather than being bowl furnaces are actually low shafted furnaces, with only the furnace bowl lying below ground level. The Highland bloomery mounds furnaces are in their majority medieval to post-medieval in date.

References

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