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Identifying your local slag... the use of quantitative methods and microstructure analysis in determining the provenance of British bloomery slags from the late iron age to the end of the Roman occupation

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16.1 Introduction

This paper is dedicated to the work and memory of G. R. 'Reg' Morton, one of the leading industrial archæologists of the 1960s, founding Chairman of the Historical Metallurgy Group, 'saviour' of Ironbridge and co-founder of the Ironbridge Gorge Museum. I am much indebted to the Museum for its help in the research and preparation of this paper.

Among their wealth of publications, Reg Morton and his assistant, Joyce Wingrove, issued a number of interim reports on the constitution of early bloomery slags. Two such reports detailed the procedures and methods which they used in the chemical and microstructure analyses of slags relating to the Roman period (Morton & Wingrove 1968, 1969). A supplementary report describing a computer model which could be used to analyse and catalogue the results for comparative reference against future analysis of slags from other sites was published by Bullas and Morton in 1969.

The present paper summarises the technical concepts of slag analysis as pioneered by Morton and Wingrove and updates the original computer model in-line with current advances in technology.

16.2 The bloomery process, its efficiency and its products

Bloomery hearths represented the predominant smelting method used in Britain from the Iron Age through to the Late Medieval period. Figure 16.1 shows a section through a typical example, that of a Roman bloomery hearth excavated at Worcester.

Metal was extracted from iron-bearing rocks (iron ore) by firstly roasting them (to drive off superficial impurities) and then by heating them with either coal or charcoal in a furnace having a forced draft. The result of this process was that the ore was reduced to a bloom containing metallic iron and slag – waste iron residue together with fusible silicate, metallic impurities and charcoal ash. The bloom was removed from the furnace whilst still incandescent and forged (beaten with heavy hammers) to drive out the slag, at the same time welding the iron

together. Using this method, the best artisans at the most sophisticated sites could produce wrought iron containing as little as 3% of residual slag and 0.1% of other impurities.

The downside of such efficiency in the process was that there were relatively few examples of high-carbon steel – though recent discoveries at sites in Denmark and elsewhere have given rise to considerable conjecture on this point (Andresen *et al.* 1994).

On the other hand, the slag was usually high in waste iron residual. This could vary from less than 10% to over 60% by weight of original ore. Computer processing of successive Morton and Wingrove results shows the average value – but not the typical value (see below) – to be in the region of 24%. This is considerably better than the figure of around 50% that had been previously supposed and is in-part attributable to the additional use of lime as a flux.
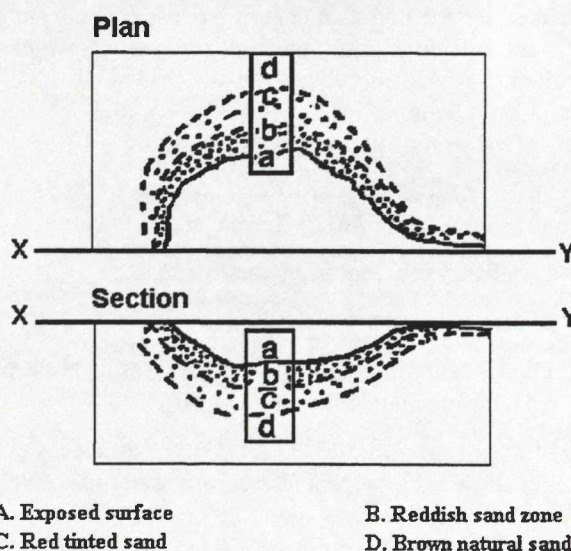

Plan

Section

A. Exposed surface   B. Reddish sand zone
C. Red tinted sand   D. Brown natural sand

Figure 16.1: Plan of Roman bloomery at Worcester (Bullas & Morton 1969).

Figure 16.2: Principal Romano-British iron smelting areas.

Figure 16.3: Metalling of Roman slag on the London-Lewes road at Holtye (Margary 1940).

16.3 Late Pre-Roman Iron Age and Romano-British usage

Figure 16.2 shows the principal areas of cross-era ore exploitation within the Province. From the map, it can be seen that certain high-grade ores from the eastern half of the Weald (but not from the western half) and their resulting products were transported along the south-east coast by the Roman fleet. This implies a high level of commercial and military integration in this area, but analysis has yet to show whether similar movements around the Bristol Channel (where very high-grade ore was in abundance for at least part of the period) were similarly integrated.

Analysed samples from diverse parts of the Province shows that the introduction by the Roman army and its artisans of more modern techniques into mainland Britain, (for example, the shaft furnace in place of the bowl furnace) saw the establishment of a two-tier quality system in metal manufacturing and in the use to which the resultant slag was subsequently put. It is remarkable that both tiers continued to function in parallel throughout most of the Roman period.

Once particular post-Romanisation foundries had become established, wastage levels tended to be:

- classifiably different from site-to-site;

- relatively constant at each individual site;

- independent of manufacturing era (mid 1st century A.D. through late 4th century A.D.).

There is, therefore, no absolute value of efficiency which typifies the process in general but rather a range of values, one of which particularises any given site. Consequently, the fact that the composition of a slag tends to characterise its place of manufacture may be used as a guide to establishing provenance.

On some sites, analysis is rendered more complex since iron-rich waste was re-used to supplement the supply of naturally occurring ores. At others, the slag was purposefully salvaged and transported to neighbouring areas for the literal metalling of the Roman road system, such as on the London to Lewes road at Holtye (Figure 16.3) as documented by Ivan Margary (1940). The resulting surface was considerably harder, particularly after weathering and oxidisation, than it would otherwise have been.

16.4 Assumptions and requirements of the analytical process

One of the major difficulties that can often be encountered when attempting to process the previously analysed data of other colleagues is the lack of necessary detail in the figures provided. This statement is not a criticism of the diligence with which the figures were prepared but rather is a reflection of the fact that the significance of some of the non-principal constituents may not have been realised. Indeed, Morton and Wingrove (1969) themselves point out that few workers give details of the mineralogical phases present, many leave out determinations of such important compounds as CaO and Al_2O_3, and frequently analyses fall short of 100%.

A second potential difficulty is that attached to the archæological record: out of any set of bloomery ore samples, how is the analyst to know whether a particular ore would have been or, indeed, was discarded by the operator as being of too poor a quality to use?

Modern methods support standard X-ray crystallography by a division of the analysis into two separate phases as outlined below.

16.4.1 Tap analysis

Tap analysis is a term which refers to the analysis of the slag as it would have been when first leaving the bloomery furnace. Oxidisation due to subsequent weathering, chemical effects due to burial in the ground and any other physical and chemical effects to which it may have been exposed will all have led to differences in its present constituency from that at the time of tapping.

Figure 16.4: Microstructures of two typical Worcestershire slags (Bullas & Morton 1969).

Figure 16.5: Isotherm diagram of bloomery operating temperatures (Morton & Wingrove 1969).

The following assumptions/requirements therefore need to be made in any mathematical model of the process:

- Any volatile materials which may be present in the excavated slag would not have been present in the tapped slag.

- Any remaining amounts of ferric oxide would have been insignificant immediately after the smelting process.

- Values of FeO, Fe_2O_3, Al_2O_3, CaO and SiO_2 must be available from the reported analysis of the excavated slag.

- Only results from those slags whose total analysed constituents are at least 95% complete should be used.

The steps for this part of the model can be summarised as follows:

A. Convert Fe_2O_3 in excavated slag resulting from subsequent oxidation processes to FeO.

B. Add to percentage of excavated FeO.

C. Round off to 100% (for future comparison).

D. Round off remaining constituents ($\times$ 100 / ΣCon where ΣCon represents the sum of all constituents including the iron as FeO from Step B).

16.4.2 Determination of mineralogical constituents

Figure 16.4 shows the microstructures of two typical slags from sites in Worcestershire (Worcester and Sharpley Pool). Whilst both slags were from Roman bloomeries, the Worcester slag had been buried for many centuries whereas that from Sharpley Pool had not (Yarranton 1680, quoted in Morton & Hallett 1966). Both slides show features consistent with Yarranton's report of three hundred years ago in that their principal constituents are glass, Fayalite ($2FeO.SiO_2$) and Wüstite (FeO) but blow holes (due to gas escaping during the cooling process) are
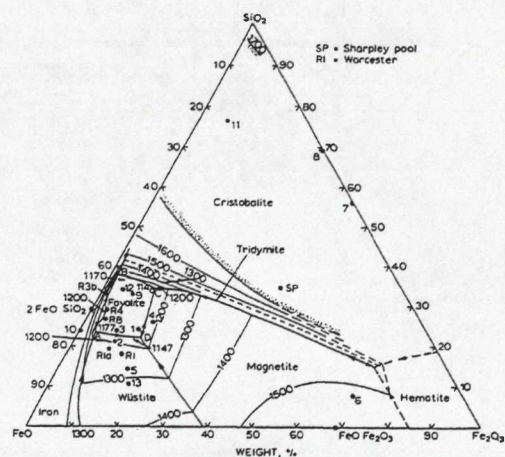
only readily discernible in the samples from Sharpley Pool, to where Roman bloomery slag had been transported from the original Worcester site for subsequent reuse in late medieval times.

The glass is represented as Anorthite ($CaO.Al_2O_3.2SiO_2$), the extreme calcite end of the plagioclase feldspar series; the amount present is dependent upon the CaO or Al_2O_3 content of the original ore, SiO_2 always being present to an excess of that required to form Anorthite.

The amount of Fayalite is controlled by the amount of SiO_2 used in the Anorthite and the presence of both of these minerals is directly proportional to the impurity of the original ore.

The Wüstite itself acts as a reduction agent in the bloomery process and the amount remaining after smelting will vary according to the purity of the ore, the skill of the operator and the efficiency of the process.

The steps for this part of the model can be summarised as follows:

E. Calculate percentage Anorthite ($CaO.Al_2O_3.2SiO_2$) after solidification

F. Calculate its SiO_2 content

G. Subtract from SiO_2 in the tapped slag

H. Round off FeO, $CaO.Al_2O_3.2SiO_2$ and SiO_2 to 100% ($\times$ 100 / (B + E + G)) where B, E, and G refer to the results of the above steps.

I. Calculate amount of FeO needed to combine with this SiO_2 in order to form Fayalite

J. Subtract from previous value (Step H) to obtain percentage of iron lost to the Slag from the ore

K. Add the SiO_2 values from Stages H and I to give percentage of Fayalite.

District:	N. Lancs	Forest of Dean	Northants	Lincs	Durham	Yorks	Derbys	S. Staffs	N. Staffs
Fe_2O_3	94.23	90.05	52.86	55.46	49.57	2.39	3.49	3.75	3.11
FeO			Trace		10.77	41.77	39.55	49.04	46.35
SiO_2	4.90	0.92	13.16	19.65	6.64	8.93	10.22	7.94	5.78
CaO	0.05	0.06	7.46	0.70	5.69	2.55	3.38	0.79	1.93
MgO	Trace	0.20	0.68	1.15	1.21	3.85	2.88	0.66	2.24
MnO	0.23	0.08	0.51	0.95	3.06	1.13	1.50	0.79	1.61
Al_2O_3	0.63	0.14	7.39	7.70	0.84	4.79	5.65	3.76	1.52
P_2O_5	Trace	0.09	1.26	0.42	0.01	0.75	1.12	0.18	0.67
S	0.05	Trace	0.03	0.16	0.03	Trace	0.05	0.11	0.15
TOTAL %	100.09	91.54	83.35	86.19	77.82	66.16	67.84	67.02	63.36
CO_2					14.49	31.39	28.63	30.80	32.46
C						0.86	1.14	0.60	2.95
H_2O	0.56	9.22	11.37	31.11	8.44	1.70	1.75	1.03	1.43
Alkali								0.42	0.18

Table 16.1: Typical analysis of principal constituents of bloomery ores

District:	N. Lancs	Forest of Dean	Northants	Lincs	Durham	Yorks	Derbys	S. Staffs	N. Staffs
Total Fe before roasting:	65.98	63.04	37.00	38.65	43.02	34.16	33.20	40.81	38.29
Fe in 100lbs roasted ore:	65.98	68.87	44.39	44.84	55.29	51.64	48.94	60.89	60.43
Slag in 100lbs roasted ore:	20.40	4.02	54.25	100.0	37.37	45.15	49.52	44.60	34.27
Fe lost to slag in 100lbs roasted ore:	11.51	2.28	18.82	63.96	25.10	21.52	21.51	32.93	24.67
Fe available for reduction from 100lbs roasted ore:	54.47	66.59	29.57	0.00	20.19	31.12	27.43	27.96	35.76

Table 16.2: Efficiency Statistics.

16.5 Calculation and classification

In their paper, Bullas and Morton (1969) described a computer program written in FORTRAN IV and running on an ICL System 4–50 mainframe computer. The program calculated the results of the above formulae and classified them in a flat-file format commensurate with the computer technology of the time. The calculation part of this FORTRAN program is currently available in the form of an executable file, a run-time FORTRAN library and a set of sample input data for use on virtually any personal computer running under DOS. It operates on an input file of F12.7 floating point FORTRAN-format ASCII values, two records per slag, representing the following absolute constituent values from the chemical analysis of each excavated slag to be processed:

FE_2O_3, FeO, SiO_2, CaO, MgO, MnO, Al_2O_3, P_2O_5, S CO_2, C, H_2O, Alkali

i.e. 13 fields in all, 9 of which are in the first record and 4 in the second.

Example output from this program is shown in Tables 16.1 and 16.2.

One interesting by-product of this modelling process is the ability to quantify the operating temperature of the original bloomery furnace from which the sample was taken. This has important connotations in being able to classify the on-site capabilities, the metallurgical knowledge and the efficiency of the operation.

In order to ascertain the operating temperature, the model produces three values which, when inserted into a suitable isotherm diagram, yield a very good correlation (better than 0.9%) with the known estimates. Such a diagram (after Levin 1956) is shown in Figure 16.5. Although inspection against a properly calibrated diagram is presently a manual process, it is hoped that an extension project will be shortly undertaken so as to fully automate the procedure.

The FORTRAN program has since been rewritten as a spreadsheet template with embedded formulae for Quattro Pro for Windows version 5. By using either the Database Desktop provided with this product or Paradox for Windows (both Borland products), the results may be automatically classified and cross-referenced with results from other sites.

Acknowledgements

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