Contextualising Metal-Detected Discoveries: Staffordshire Anglo-Saxon Hoard

(Project 5892)

Stage 2 Project Design

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Appendix 3: Materials Analysis Programme. Assessment report

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1 Pilot Projects

There were five pilot projects in the first stage of the materials analysis programme in the Staffordshire Hoard project design[62].

1.1 Gold enrichment study

A pilot study of 16 gold items was carried out to determine whether there was any surface enrichment/depletion and whether this related to find location. This was carried out using the equipment stated in the project design[63]; small areas on these pieces were prepared using a tool to reveal the sub-surface for SEM-EDX and XRF analysis.

The results from the pilot study of surface enrichment of 16 gold sheet objects, mostly hilt plates, clearly showed that in many cases there is significant but not consistent surface enrichment of the gold at the surface owing to the depletion of both copper and silver. The analysis of deep scrapes, probably made when dismantling the swords before burial, indicated the expected loss of copper from the surface during burial, and little loss of silver. However, the results from undamaged surfaces of the same objects suggest that some form of deliberately induced depletion gilding was carried out by the goldsmiths to remove both silver and copper from the surface of sheet gold components, perhaps to enhance their golden colour.

At this stage it was difficult to say whether more sophisticated surface treatment was a widespread Anglo-Saxon metalworking practice. This was a small sample of similar artefacts and bias may have been introduced to the results because of the selection of such a specific type of object, as they are almost all of similar alloy composition. Therefore, more work was necessary on a larger range of object types, and particularly objects of composite construction, to see whether the components vary in composition and surface treatments. This was carried out in the second stage (see Section 2).

The results also suggest that XRF analysis on unprepared object surfaces, even with its deeper penetration into the body of the object can provide only semi-quantitative information at best about the original alloy composition. The accurate determination of the original alloy or 'core' composition of objects can only be achieved by sub-surface analysis using techniques such as SEM-EDX and/or XRF, or other invasive or destructive analytical techniques. The unpredictable variability within the surface and sub-surface results means that there is no feasible method to recalculate the sub-surface results using surface XRF or SEM-EDX data.

[62] Cool 2013: PD 2013 Section 15.8iii.
[63] PD 2013 Section 15.8vi.

A full report of the results and a detailed discussion of the methodology has been circulated[64]. In addition to this, a methodological paper is being prepared to be published in the journal *Archaeometry*.

1.2 Component study

The second pilot study focused on the comparison of the alloy compositions of individual components in objects of composite construction. This research was carried out in conjunction with the second stage of the analysis project, and is therefore reported below in Section 2.

1.3 Comparison between institutions

The third pilot study was originally envisaged as a comparison of the surface XRF and PIXE analyses conducted at the various institutions who have undertaken analytical work on the hoard since its initial discovery. In light of the surface enrichment noted during the first of the pilot studies, the original set of PIXE surface data is no longer of such direct relevance to the subsequent analytical work undertaken during the hoard project. The hoard owners have copies of all of the raw PIXE data acquired in Paris and scientists at the BM still intend to prepare a basic report to accompany the deposit of this data in the hoard project database. There is also a stand-alone publication planned in the near future to honour agreements with the CHARISMA project, under which the PIXE work was undertaken, and with colleagues at the C2RMF who assisted with the work, but this will be easier to do once the enrichment work has been published and can be cited and the typological work is complete.

A new study focusing on the comparison between SEM-EDX (surface and sub-surface) analysis results and the results from the different XRF systems (surface and sub-surface analyses) at BM and BMT is being carried out. The aim of this is to establish the degree of inter-comparability of the data.

On 21 March 2014, Eleanor Blakelock from the British Museum visited BMT to discuss and share information about the procedures employed at the British Museum to undertake the sub-surface alloy compositional analysis of the hoard objects using XRF. During this visit, the ‘MAC’ standards commissioned for the hoard project and a number of British Museum alloys of known composition were analysed.

A report on the inter-comparability of the data from the British Museum SEM-EDX and XRF and the BMT XRF is being prepared[65].

1.4 Die-impressed sheet analysis

To support the work of the BM conservators[66] (15.7), a selection of the silver die-impressed sheet fragments were chosen for analysis. In total 18 foil fragments from six different friezes were examined by surface XRF[67].

[64] Blakelock 2013
[65] Blakelock forthcoming a.
[66] PD 2013 Section 15.7.
[67] PD 2013 Section15.8vi

With the exception of the interlace sheets (frieze 10), the XRF analysis of the fronts of all the foils revealed the presence of mercury gilding, which prevents direct access to the silver alloy below. Surface XRF of the fronts of the foils therefore cannot be used to group them. The analysis of the backs of the foils showed a range of compositions that overlap between friezes and therefore do not help in the identification of compositional groups linked to specific friezes. This study overall shows that a rapid surface XRF survey is not the appropriate technique to identify frieze groups.

The non-gilded interlace foil fragments from frieze 10 are analytically different but they are easily distinguished from other fragments owing to the lack of surface gilding, so XRF analysis is unnecessary. If more types of non-gilded friezes are present within the hoard assemblage, XRF analysis may be able to help distinguish between them.

A report of the results has been circulated[68].

1.5 Coin study

A recent study by Gareth Williams and Duncan Hook at the British Museum of a large group of Anglo-Saxon coins, some originally studied by Oddy, has been published[69] . This study showed a good correlation between Oddy's original specific gravity (SG) analysis carried out in the 1970s[70] and the SG and XRF analysis of the same coins undertaken in the Williams and Hook study.

With the involvement of Gareth Williams and Duncan Hook, eight additional Merovingian coins were selected for comparison with the previously examined coin group and the gold alloy compositions analysed using sub-surface XRF[71] and SG (see Section 3 below). In addition to the eight new coins analysed, six Anglo-Saxon coins from the study by Williams and Hook (2013) were also re-examined using SEM-EDX in light of the results from pilot 1.1 here. This examination has revealed that surface treatments resulting in surface enrichment were also being applied to contemporary Anglo-Saxon coinage.

A full report of the results is being prepared[72].

2 Second Stage Analysis

In the second stage of the materials analysis programme, the group of gold objects from the hoard selected for analysis was increased. The study broadened in scope with the inclusion of a number of objects from the BM's collection and a pendant from PMAG as *comparanda*, allowing extension of the study to functional categories not included in the hoard.

[68] Blakelock 2014.
[69] Williams and Hook 2013.
[70] Oddy 1972.
[71] PD 2013 Section 15.8vi.
[72] Blakelock and Hook forthcoming.

In total, 114 individual objects from the Staffordshire Hoard were analysed, along with the pendant from PMAG and a further 36 individual Anglo-Saxon objects from the BM (excluding the coins included in the surface enrichment study mentioned in Section **1.5** here). This created a data-set of gold alloy compositions for 157 Anglo-Saxon objects and coins.

Analysis of individual components from six hoard objects selected from the pilot study (K88, K301, K352, K660, K673 and K689) showed that there were clear differences between them. Therefore, when it was possible to do so, individual components were also examined from a number of the objects selected for this second stage analysis. In total, 288 individual object components were examined including sheets, cast bodies, cell walls, caps, beaded wires, twisted wires and pins.

A full report of the results is being prepared[73].

Individual reports are also being produced for specific sets of objects including the seax set (K376, K354, K690, K370 and K449), the great cross (K655, K656, K657, K658, K659 and K1314) and the 'mystery' object (K130, K545, K1055)[74]. In addition to these reports, two multi-author articles are planned and will be produced based on the second stage analysis work, one for the *Journal of Archaeological Science* and the other for *Antiquity*.

3 Techniques Used

Specific gravity (SG) allows for the estimation of the percentage of gold in an alloy, and in this case is used as a check on the XRF analyses, and for identifying plated or otherwise unusual coins. This non-destructive method has the advantage of taking into account the whole object. The specific gravity is determined by weighing the object first in air and then suspended in a suitable liquid. The coins were suspended in Flutec (perfluoro-1-methyl-decalin) for 10 minutes and the temperature was recorded to take into account the coefficient of thermal expansion of the liquid, which influences the result of the measurement. Further details on the method and its accuracy are described in Hughes and Oddy (1970) and Oddy and Blackshaw (1974).

4 Analysis of Technology, Construction and Inlays

To support the conservation work[75] and to gain a better understanding of the technology, construction techniques and materials used in the objects in the hoard, identification of materials such as inlays, textile fragments and other macro-organic residues, adhesives and backings, etc. have been carried out. A range of techniques were used, including PIXE-PIGE, XRD, FTIR and Raman spectroscopy, GC-MS, SEM-EDX and XRF, in addition to X-radiography, and a number of reports have

[73] Blakelock forthcoming b
[74] Blakelock forthcoming c; Blakelock and Ambers forthcoming; Blakelock forthcoming d.
[75] PD 2013 Section 15.8v.

been circulated or are in preparation[76] . A stand-alone publication is also in preparation that will bring the preliminary analytical work (PIXE analysis undertaken in Paris) on the garnets from a group of objects from the hoard into the public domain.

5 Future Scope for Scientific Analysis

5.1 Metal analysis of the hoard

The second stage analysis project has allowed a large range of gold objects and components to be analysed, creating an extensive database of the core and surface metal alloy of the gold objects in the hoard. This work could be extended to include a wider range of object types, but the results obtained to date do not suggest that this would reveal a significantly different picture to that already obtained in stage 2 of the gold analysis, but any future work should be carried out on prepared sub-surface areas.

To assess whether analysis of silver objects from the hoard would be of value, a pilot study should first be carried out on a limited number of items to determine whether a rapid surface XRF survey will reveal any distinguishable alloy groups. This may assist conservators grouping the objects and may reveal changes in composition through time. Silver alloys are even less stable than gold alloys and the changes caused by corrosion are likely to penetrate deeper into the metal, factors that may affect the value of such analysis.

5.2 Metal analysis of contemporary artefacts in other collections

Analysis of further male- and female-associated items of the period would be required to understand more about the differences in surface enrichment seen in this study.

More research on the surface enrichment seen in the contemporary coinage is required to understand the use of this technique in coin manufacture. Enrichment analysis of coins from a wider time period might help to determine whether the later coins are generally more enriched than the earlier ones, for example. It may also be possible to identify different coin groups or mints, if there are differences in the amount of enrichment present.

5.3 Metallographic studies of the hoard

Previous studies by other researchers have shown that it is possible to understand more about metalworking technologies, such as the apparent deliberate surface treatment to give a gold-enriched surface, through microstructural analysis, especially when combined with chemical analysis[77]. Depletion techniques affect the surface of the object, and although burnishing often removes this evidence at the very surface of an object, below this burnished surface a porous zone created by the loss in silver and copper can often be seen[78]. The technologies employed in the deliberate silver depletion seen in the Staffordshire Hoard objects would therefore

[76] Cartwright 2013a; 2013b; La Niece 2013; Meek 2012; 2013a; 2013b; Mongiatti 2014; Mongiatti and Ambers 2013; Mongiatti *et al*. 2013; Stacey 2014; Steele and Hacke 2013.
[77] La Niece 1995; Lehrberger and Raub 1995, 347–50; Voute 1995, 336
[78] Lehrberger and Raub 1995, 347.

most likely be better understood through the microscopic examination and analysis of cross-sections.

5.4 Inlays and decorative techniques

Further scientific study of inlays and wire and filigree, as well as construction techniques, could contribute to an understanding of the workshop practices to be seen among the hoard items. Further work on the non-metallic components of the hoard may also be helpful. For example, several items have what appears to be a green inlay in cells that has yet to be satisfactorily identified. This might be attempted by a combination of X-ray diffraction, SEM-EDX analysis and various spectroscopic techniques. Further work to identify and characterise other inlay materials or organic materials may also be promising avenues for further research.