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**From the Fabricae of Augustus and the Workshops of Charlemagne: A compositional study of corroded copper-alloy artifacts using hand-held portable XRF**

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## Chapter 9

### Conclusion

The purpose of the research presented in this thesis was firstly to explore the limits of HHPXRF use on corroded copper alloys, then secondly to develop a methodological approach using research questions that are relevant to archaeology. This was addressed in the methodology section presented in chapter 2. This methodology was then tested on a large number of Roman and early medieval personal objects dating between the 1st century BC and the 12th century AD. Subsequently a number of published case studies are presented (chapters 3 to 8) as examples of its effectiveness.

The aim of the project was to contribute to our understanding of Roman and early medieval craft organisation, through gaining a better understanding of the choices made in the production of copper-alloy personal items. More specifically the aim was to investigate variability in the control of copper-alloy composition, in particular the variation between bronze, brass or gunmetal, and to identify regional or chronological differences between groups of artefacts. Subsequently the intention was to gain new insights into the way production was organised, including the supply of raw materials, and to identify differing cultural associations through the choice of alloy.

This was achieved through the application of HHPXRF, deployed in the reconnaissance method described in chapter 2. The method was found to be able to sort objects into basic copper-alloy groups (bronzes, brasses, gunmetals, then also identifying high or low levels of lead). It also had some success in identifying objects that had been surface coated such as the disc brooches presented in chapter 4. This qualitative, semiquantitative method, enabled a better understanding of the choices being made in the production of copper-alloy personal items and therefore contributes to our understanding of Roman and early medieval craft organisation. Now that objects could be sorted by both typology and composition, over a given region and time, the level of variation, both within and across typological groups could be assessed. This assessment was central to the case studies presented in chapters 3 to 8. The results of these case studies demonstrated that a level of compositional control could be established for typological groups, allowing for an exploration of regional or chronological differences. Subsequently new insights into the way production was organised were uncovered. The investigation of the Roman period bow brooches presented in chapter 3 demonstrated that the transition from bronze to brass during the 1st century BC could be identified using the method, but importantly after this transition a number of brooch types were found to continue in bronze, in parallel with the new brass working tradition. This allowed new hypotheses to be formulated, exploring for example, differences between Roman military and local civilian production. Because brass and bronze have differing raw material sources, the direction in which fresh copper-alloy travelled allows discussion about contact

between the people of different regions. It is particularly useful in establishing what is common for an area in terms of alloy use at a given time. When this is established any variation in objects with a differing alloy choice can be explored. If both the typology and the alloy of an uncommonly found object is seen to match those more commonly found in a distant region, then it adds something to discussions regarding mobility. In this case the evidence supports the notion that the objects were made in that distant region and had travelled. Conversely if the alloy choice for this uncommon object was found to match that of locally produced objects, then local copying could be considered. This is kind of difference is particularly evident in the composition of the disc brooches discussed in chapters 5 and 7. The standard for the region was established as being zinc based, and it was only on the small island of Walcheren that deviation in this alloy choice was observed. This suggested that if disc brooches were made on the island, and not imported from elsewhere (possibly from a region outside the current research area), the production output, in particular the raw material exchange and technological knowledge, was different to that for the rest of the region. This difference could then be compared with other data (such as historical sources) to provide new hypotheses for why the difference is present. This included the formulation of different cultural associations, such as those presented for the Roman period disc brooches in chapter 4 and the Viking age mounts in chapter 6.

The answers to the research questions:

a) Was it possible to construct an interpretative framework for the use of HHPXRF on copper-alloy, particularly regarding the issues concerning its use on corroded artefacts?

The scientific objection to HHPXRF centred around the inability to produce highly accurate quantitative data on corroded copper-alloy surfaces. Furthermore objections were raised about the potential for misuse, by archaeologists lacking appropriate scientific training. Supporters of HHPXRF countered that it could be effectively used in a qualitative or semi quantitative way. That it was capable of basic alloy classification and therefore useful in answering certain archaeological questions. To address the objections of inappropriate use, a scientific methodology was called for that established reliability and validity. Importantly the nature of the analysis needed to be understood; be it qualitative or quantitative.

The method presented in chapter 2 addresses this problem. Its departure point argues for a qualitative and semi quantitative approach, but in doing so investigates the extent to which a non-destructive analysis of the corrosion layer can be used to classify copper-alloy artefacts. A reliable level of accuracy and precision for XRF had already been established elsewhere, but the degradation of copper-alloys and the subsequent formation of a corrosion layer, needed better understanding. To achieve this a group of early Roman brooch fragments were measured in a corroded then cleaned state. This tested the deviation in results between the two states, which was

necessary if interpretations regarding alloy choice were to be made on large numbers of corroded objects. This test was critical if the aims of the project were to be achieved. Steps were also taken to address the objections surrounding inappropriate use.

The methodology addresses the issues regarding lack of 'scientific' approach, by demonstrating an understanding of the instruments parameters and settings. It also provides a basis for experimental reproduction by other machines and also allows comparison with other published datasets. To achieve this the method recommends the inclusion of a detailed account of the HHpXRF device being used when publishing results. Including details of its internal calibration standards and specific operating geometry. Furthermore the performance of the device would ideally be tested against the CHARM reference set and the results also reported, to further aid comparison to other machines and datasets. The tests on the Roman brooch fragments showed satisfactory levels of accuracy and precision albeit under more ideal analytical conditions. The lack of sample preparation and non-ideal measurement conditions on corroded brooches did not prevent a broad recognition of different alloy groups. An intermachine comparison was also conducted in chapter 4. A Bruker machine was used for the analysis and deployed in a 'off the shelf' capacity. Comparison between the Bruker and Niton devices was undertaken using a set of locally made standards. This linked the two machines together even though only one had been linked to the CHARM standard. In addition, measurements took place on objects from three quite different regions (The Netherlands, the east coast of England and the cremation cemeteries of Estonia and Northern Latvia) to investigate if different preservation conditions would significantly alter the measurements. It was established that the measurements taken by the Bruker device could be compared to data published elsewhere, as well as that gathered in The Netherlands and England using the Niton device. It was found that the overall error for the elements of interest, for both machines, was well within the limits needed to achieve the projects research aims.

b) What compositional variation is there in large numbers of similar copper-alloy objects?

Variation in composition could be seen when analysing large numbers of corroded items. But the data produced this way requires an interpretive step to be of use, due to the bias introduced by corrosion. The normalised data, especially when visualised in ternary diagrams frequently showed a strong correlation to artefact type. This allowed for classifications to be made along the lines of brass, bronze, gunmetal, with high or low levels of additional lead. To attempt to interpret the data coming straight from a corroded surface is highly misleading. This is because the bias introduced by the leeching of zinc from corroded outer surfaces has the effect of artificially enriching the tin values. Specifically, the tin levels are magnified in the resulting data. This means for example that a group of very similar objects may have all been made in brass, but the results show a more randomised pattern due to the variability caused by

zinc loss. This means that some of the results could inadvertently be misinterpreted as gunmetal because of the tin enrichment. An amount of correction needs to be applied therefore, in line with the results of the corrosion tests presented in chapter 2. Generally the measurements from these objects can still be interpreted as being produced from a bronze, brass or gunmetal alloy. There are some object types however that do not demonstrate controlled composition, such as the disc brooches found in Estonia and Northern Latvia. One interpretation is that these objects were surface treated, e.g. coated in tin to change the colour. These thin surface treatments would be the first to decay during the corrosion process, possibly creating a patchy, inhomogeneous result. The presence of a decayed surface treatment in this case prevents the deeper exploration of the main alloy composition. That said, identifying deliberate surface treatments, is as worthwhile as identifying controlled alloy groups in terms of production choices, especially when considering the highly visual nature of personal costume accessories.

c) Is there evidence for deliberate control of copper-alloy composition (perhaps between different types of objects)?

The assumption from the outset was that groups of closely related objects might have been made in the same or very similar alloy. That is to say that a choice was made to make an object either in brass, bronze, or gunmetal. Deliberate control was observed from the outset, starting with the introduction of brass in the production of early Roman period brooches. Certain brooches, which have been traditionally associated with the Roman military, were consistently produced in brass. Other brooch types conversely were made in bronze. Both brass and bronze existed as a readily available choice in the early Roman world, at least until the widespread change to production in bronze and gunmetal towards the end of the 1st century AD. For some brooch types however, such as the Gallic bow on Spring-tube type presented in chapter 3 and the later Roman period disc brooches presented in chapter 4, the opposite appeared to be true. The highly variable results suggested that the craftsmen did not care about the alloy choice for these items, or that they were solely produced using highly variable recycled scrap, which if the case was a different form of deliberate control, avoiding fresh raw material. The other hypothesis was that a tin based surface treatment had broken down within the corrosion layer masking the underlying copper-alloy. The identification of a surface treatment however could also arguably be evidence for deliberate control as it is a deliberate technical choice, reserved for these brooch types, possibly intended to change or enhance their colour.

The reduced use of gunmetal (probably made by recycling scrap objects) in the pins found at Sedgeford and Domburg, can also be seen as evidence for deliberate compositional control. This is because the craftsmen would have needed regular access to fresh bronze and fresh brass to avoid widespread use of recycled material. This fresh material probably took the form of trade ingots, and as zinc (for brass) was not mined in England, yet tin (for bronze) was, ideas could be formulated regarding

their movement within a North sea trade network and the organisation behind their production.

d) Is there evidence for chronological change in alloy use from the early Roman period to the beginning of the Middle Ages (1st century BC - 12th century AD)?

A relationship between alloy choice and typology was established using the methodology presented in chapter 2. Typology forms an important dating tool in archaeology and subsequently allowed the assemblages to be studied for chronological variation. The clearest variation was the widespread introduction and subsequent control of brass in the early Roman period. But brass use was seen to exist in parallel with bronze use for a number of centuries, until a general change to gunmetal took place during the later Roman period. There is a widespread revival in brass use during the Carolingian period, at a time when gunmetal and bronze was also available. These long-term chronological changes were identified by the HHPXRF methodology.

e) Can regional differences in composition be seen (perhaps eluding to the availability or otherwise of raw materials)?

Typology is an equally useful tool for studying regional differences as well, especially when linked to alloy choice. This study identified different alloy choices between the brooches found on the island of Walcheren, Frisia and the rest of The Netherlands. The Carolingian/Ottonian period disc brooches found on Walcheren showed a significantly wider range of alloy choice than that for those found in the rest of The Netherlands. A difference was also apparent when the equal-arm brooches were analysed. Those found in Frisia are typically of a leaded brass, but those found on Walcheren were typically produced in gunmetal and bronze. This last example could have had a chronological aspect as well as regional one, but the current dates for the typologies suggest that some of the equal-arm forms existed alongside each other, but were consistently produced in differering alloys. HHPXRF was also involved in revealing regional differences in Estonian and Northern Latvian disc brooches, supporting the identification of different surface treatments.

f) What are the potential supply lines for the sources of raw materials?

The results for the early Roman brooches (chapter 3) identified two parallel production traditions existing at the same time. Some brooch types were produced in bronze and other types were produced in brass. This allowed an hypothesis to be formed regarding two different supply lines. One being a local civilian one, using bronze, perhaps following pre-Roman alloy traditions, where the tin (possibly in the form of bronze ingots) was imported from England. The second was a new tradition using brass (with the zinc possibly coming from La Calamine in the Ardennes, Belgium), possibly being reserved initially to Roman military centres.

For the early medieval period, the regional differences between the equal-arm brooches found at Walcheren and the northern Frisian areas, also allows discussion regarding raw material supply. The equal-arm types found on Walcheren have a find distribution pattern that places them well within the Frankish realm. But the research identified that some were made in bronze, not in brass, which presumably would have been available locally at the same time. In contrast, the equal-arm brooches from the Frisian north are made in brass. As in Roman times, the closest source of zinc at this time is thought to be the Ardennes region of Belgium. But if correct this means that Frankish production of the Walcheren brooches (if they were indeed made locally) ignored the local brass, raw material supply, in favour of bronze (using tin presumably imported from England). Conversely the production of the brass made brooches found in the Frisian north, suggests a different raw material source, perhaps one more closely connected to the Viking world. Alternatively, Frankish brass production could have solely been reserved for export to the north. Brass was also linked to the Viking north through the analysis of the zoomorphic mounts found on Walcheren (chapter 6). Apart from the zoomorphic decoration having strong cultural associations with the Viking age North Sea world, they were made in brass. The same goes for the equestrian stirrup-strap mounts from the same period (chapter 7), also bearing similar decoration. Two supply lines are visible therefore. Tin, possibly in the form of bronze ingots would have come from England and zinc in the form of brass ingots could either have come from the Ardennes region, or much further afield through the Viking sphere of influence. The discussion regarding the raw material supply for the pins found in Britain (chapter 8) demonstrate the usefulness of knowing which alloy an object is made of. No zinc is mined in Britain, therefore all brass in the form of fresh raw material would have to have been imported. These small personal costume items, when made in brass, must have been made from imported raw material, or possibly imported as ready-made objects in their own right.

g) To what extent can compositional variation, across geographical and temporal distributions, allow new insights into the social organisation of production, or the identity of those wearing the finished objects?

Many hundreds of costume accessories analysed in this project may have frequently travelled on the clothes of their owners. When an individual object, or a type of accessory is found some distance from its believed place of origin, then discussions surrounding mobility or migration frequently take place. The prime purpose of the method proposed here is not to identify unusual objects from a typological perspective. It identifies them from an alloy perspective. This was the case for a number of early medieval disc brooches found on the island of Walcheren (chapter 7), where the typology of the objects matched the rest of the region, but the alloy did not. But costume accessories could well have been involved in more complicated expressions of identity as well. Pilgrims or other travellers, when returning to their homelands, might have brought souvenirs back with them, perhaps as "returning gifts" for friends and family, which when worn could change a persons status. This



idea is expanded upon in chapter 5, where these early medieval disc brooches are discussed against the hypothesis of monastic production and ecclesiastic distribution. This discussion was made possible because of the consistent use of the same alloy (as identified by the HHPXRF), for several centuries, could well have been made possible through control based on long term monastic tradition. Linking these brooches to monastic production subsequently allowed for discussions regarding exchange between the church and the peasantry, one where these brooches may have been imbued with 'cosmological authentication' in a similar way that pilgrim badges did a few centuries later.

The method also enabled identity discussions surrounding local versus regional production. The high concentration of unusual zoomorphic mounts found on the island of Walcheren (chapter 6), suggested local production. The decoration on the mounts and the alloy used, allowed an expression of identity to be formulated, for a cultural group, based on intensive relations between Franks, Frisians, Vikings and Anglo-Saxons, in an interconnected North Sea world.

It was possible therefore to construct an interpretative framework for HHPXRF use on copper-alloy, taking into account surface interaction and corrosion. The relationship between form and bulk alloy composition could be explored and the results applied to the archaeological questions formulated for the project. The conclusion is that this interpretive framework enables basic alloy classifications to be formulated for typological groups and is therefore useful in answering certain archaeological questions.

#### Limitations of the study

From the outset there were two key objections to this line of investigation. Firstly that the approach was not quantitative therefore should not be undertaken in the first place. Secondly that the ease of access that HHPXRF provided to the archaeological community would lead to misinterpretation and misuse by poorly trained and or inexperienced users. Addressing these issues formed an important part of this thesis. Firstly if we accept the argument regarding quantitative analysis, that it is the only acceptable scientific route for data gathering, then the methodology presented above fails. The critical stumbling block to fully quantitative analysis (excluding differences in costs) is the need to take destructive samples, which is usually only possible from a very limited number of archaeological objects. The departure point for the qualitative, semi quantitative method presented above is that non-destructive analysis is preferred if we are to conserve this stock of objects for future generations. The key issue is how or indeed if we should infer any useful archaeological meaning from the corroded outer surfaces of these objects. The methodology presented here limits itself to data recovered from these corroded outer surfaces and will always be substantially

different to data recovered by sampling an objects core. Therefore the corroded data as it stands is not suitable for answering traditional archaeological questions. The study found no meaningful correlations for trace elements for example (other than the presence or absence of mercury, used with gold as part of a deliberate surface treatment) and has to limit itself to the study of the bulk metals used in production. The methodology only works because an interpretive jump is required to classify the alloys from corroded data to original choice. This interpretation is possible thanks to the deviation study conducted between clean and corroded surfaces, identifying a systematic trend in copper and zinc loss during the corrosion process. It is therefore at risk of subjective observer error and therefore as it stands cannot satisfy the scientific objections posed earlier. This issue also applies to the second objection, that operators can easily misuse or misinterpret the data. The biggest risk in the application of HHpXRF by untrained or inexperienced archaeologists is the mistaken belief that the measurements obtained are quantitative, i.e. that they are a 'true' measurement of the items original composition. This is particularly true if a measurement is taken on one single object with the notion of gaining a "correct" value of its composition. The methodology has been shown to work in identifying trends for groups of objects, not for gaining reliable single alloy measurements in isolation. Therefore a misinterpretation of the alloy composition is a very real risk in this circumstance. The risk is doubled in this scenario by the mistaken belief that whole objects, or patches on the objects (lacking a corroded outer patina, perhaps where it has been chemically removed in the conservation process), can yield a similar result to a measurement taken from a clean core sample. This is not the case as this "shiny" outer surface deviates further from this 'true' measurement than the corroded patina. This is because the outer surface has also been significantly altered, by the leeching of copper and zinc. A related objection regarding misinterpretation was that inexperienced operators would take the internal data being produced by a single machine and consider it to be comparable (and therefore replicable) to other studies, without the need for referencing against external standards. The methodology developed in this thesis and published in the article presented in chapter 2 goes some way to address these issues. The methodology provides a basic guide, or protocol for other HHpXRF users. It warns of the issues just mentioned and also provides a useful calibration reference through its link to the CHARM standards. Even though this methodology is published however, there is no guarantee that all users of HHpXRF will adopt it in their research. Ultimately therefore Speakman and Shackley's call for the peer reviewers of all HHpXRF based manuscripts, to seriously scrutinise them for invalid and unreliable data, holds true.

The range of artefact types presented in this thesis is quite limited, in comparison to the much larger number of copper-alloy object types known from these periods. This was primarily due to the need to gather large number of measurements on objects from the same typological groups. It was not intended to provide an exhaustive typological/chronological survey of the Roman and Medieval periods, so brooches in particular were good candidates to study. This is because they are found in large quantities and are supported by pre-published typological and chronological

reference data.

The limited case studies, presented in this thesis serve to highlight the effectiveness of the methodology, but in doing so leave large typological and chronological gaps. Some of these gaps have been addressed in publications elsewhere, (See Roxburgh *et al.* 2014, Roxburgh *et al.* 2017). Brooches have been extensively published for the Roman period, but other important artefact types are missing and also need analysing (e.g. coins, bracelets, finger rings, neck rings, items of military gear). It is not enough to use the alloy choices identified in Roman brooches as a proxy for raw material availability for other personal dress items. For example it is not yet clear if all objects relating to the military or military centres were made in brass, or conversely those from outlying civilian centres relied more heavily on older bronze making traditions or the recycling of mixed scrap items into gunmetal. Coins in particular have been left out. This was primarily due to the frequent use of surface enrichment or coating employed in their production. Also coins are not often used in identity construction, therefore they are a less useful than brooches for example. A useful HHpXRF survey of Carolingian coins using die chain analysis has recently been undertaken elsewhere however (Buis 2016). The subject of surface enrichment or treatment is quite an important one and is touched on briefly in the disc brooch article in chapter 4. The surfaces of corroded objects that have had a treatment, such as tinning, silvering or gilding, are much more variable in their composition, due to the fragmentary remains of these external treatments being trapped so to speak in the corrosion layer. The presence of tin from a coating for example is especially problematic because it would cause the results of an HHpXRF measurement to be tin rich, suggesting that the alloy of the object itself was a bronze or a gunmetal, when in fact the underlying alloy could easily be a brass. The presence of silver or gold could equally lead to misleading surface measurements. This becomes problematical when attempting to analyse some important object types from the Merovingian period for example (The main reason why Merovingian period artefacts, and especially brooches are under-represented in this thesis). Visual inspection revealed that from the late Roman period onwards many brooch types showed traces of surface treatment. Several Merovingian types in particular appear to have been consistently surface treated, including radiate head, square head, bird, garnet inlaid disc and S-shaped brooches (see Heeren and van der Feijst 2017, types 81, 82, 83, 84, 85 respectively). These types frequently occur in furnished burials from this period. Whilst these brooch types are available in large enough numbers to be included in the project, the variation introduced by corroded surface treatments put them beyond the methodological limit for establishing alloy choice and therefore engaging with the debate surrounding workshop organisation, changes in raw material supply and cultural association. That said, establishing regional or chronological patterns in external colouring, as discovered for the disc brooches in chapter 4, is also of archaeological interest but is a line of enquiry that is not continued here.

If the quantitative objection is firstly ignored in favour of the development of a non-destructive qualitative method, the limits outlined above still highlight the risks posed by the misuse or misunderstanding of HHpXRF by inexperienced

archaeologists. That said the method is effective in answering some very relevant archaeological questions.

#### A course of action and useful areas of further research

All of these limitations would initially seem to support the objections posed earlier. However like any emerging technology further steps are necessary to improve its effectiveness. These steps include the development of better machine performance in relation to more readily available external calibration standards. This study would not have been possible without the destructive testing of the fragments of early Roman brooches to establish the deviation between corroded and clean measurements. Knowing how the results deviate is critical in making the interpretive step that assigns an alloy classification to a group of objects. A larger data set, including objects taken from a wider timespan (late Roman for example), especially including items with higher lead content, would further strengthen the interpretive step. But what would be much more effective would be to develop a statistical regression model or similar that removed the interpretive step completely. A return to the Nijmegen assemblage from which the original fragments were selected would be the first course of action, to establish if more objects could be released for destructive testing. Then other large assemblages might be sought from which fragments could be gathered. But whichever the source, the destructive cleaning of artefacts, even fragments cannot be casually undertaken, the end use of the data would have to be clear and permission would need to be granted from the owners and/or curators.

A further avenue of investigation would be to gain a better understanding of the corroded remains of surface treatments. A visual inspection of an item, especially of an artefact type known to have been tinned, silvered or gilded, can frequently identify patches of this outer colouring amongst the corroded patina. But many artefacts do not offer up this secret so readily. But there are most likely chemical clues left behind in the patina that HHpXRF can unlock. Unusually high levels of tin, silver, gold and mercury for example, measured on what would usually be considered a featureless (corroded) surface, may indicate a surface treatment that had worn off, or that has become lost in the corrosion phenomena. The development of HHpXRF in this direction would allow further exploration of metallic colour choices, used in past identity constructions.

The systematic "fingerprinting" of assemblages from both settlement and cemetery sites can now also be considered. That is the amount of objects produced in varying alloy types, perhaps expressed as a percentage of the overall assemblage, may be of great archaeological use. This additional way of exploring the material culture would allow for inter site comparisons to be made, similar perhaps to that made between the assemblage on Walcheren and the rest of The Netherlands, but on a much more intensive scale. Cemetery sites could be "fingerprinted" in the same way, so that geographical and chronological variation in alloy choice can be explored, adding a

useful tool in understanding how cemeteries were organised if distinctly different alloy groups are found to be present amongst the grave goods. Furthermore, the organisation of the alloys in a cemetery, are closely related to the deceased individuals buried there. As we have seen in the previous chapters, a link between typology, alloy choice and surface colour can infer something about the lives of the people buried there, especially if used in conjunction with other avenues of investigation such as those within the field of Osteology. Does HHPXRF have a future then? Yes it does. It is very much an emerging tool for the study of a range of artefacts, including corroded copper-alloys. The methodology needs refining and developing further but the potential for answering important archaeological questions is high, as has hopefully been demonstrated in this thesis.

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