

Ancient Monuments Laboratory
Report 66/96

ANALYSIS OF NON-FERROUS METAL
ARTEFACTS FROM BARRINGTON
(EDIX HILL) ANGLO-SAXON
CEMETERY, CAMBRIDGESHIRE

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Summary

Quantitative analysis of 26 copper alloy artefacts showed a range of copper alloys were being used. Brasses with small amounts of tin and gunmetals with about 5.4% zinc and 5.5% tin are the most common alloy types. There is little difference between the alloys used for casting and those used for wrought work, nor is there any clear evidence of alloy selection for objects to be gilded. Pairs or sets of artefacts are often of the same alloy type.

The gilding on one cast saucer brooch and one applied saucer brooch was shown to be amalgam gilding (fire gilding), with a relatively high level of mercury (up to 20% Hg in one case). This suggests that the gilding was carried out at relatively low temperatures which were just sufficient to give the surface a golden colour. Tinning was also carried out at quite low temperatures which preserved a good tinned surface.

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Analysis of non-ferrous metal artefacts
from Barrington (Edix Hill) Anglo-Saxon Cemetery, Cambridgeshire

Catherine Mortimer and Kilian Anheuser

Assessment of the non-ferrous metalwork at this site (Mortimer 1993) suggested that compositional and structural analysis could provide interesting information about metal supply and use by the metalworkers who made the artefacts found at the site. It was also noted that a detailed study of the punchmarks would establish the range used at the site and might indicate any instances of a single tool being used on more than one artefact; the results of this work are described in a separate report (Mortimer and Stoney 1996).

Composition (Catherine Mortimer)

Analysis: Compositional analysis was carried out using a scanning electron microscope with an energy-dispersive X-ray analyser attachment (SEM-EDX). Samples were cut or drilled from discrete areas of the artefacts, mounted and polished to 1 μ . Each sample was analysed three times, where possible (some samples were very small) and the average values calculated. The results of analysis on four copper alloy standards are presented in table 2. The default parameters on the analyser gave slightly high tin values and low lead values on these standards, compared with the given values; the results of the Barrington samples were accordingly corrected. Normalised compositions are presented in Table 1.

Results: A plot of the tin and zinc contents (Figure 1) shows the majority of the alloys have significant amounts of both zinc and tin, with few pure brasses or bronzes and the majority of the datapoints lying in a continuum between the zinc-rich and tin-rich zones. Thus far, the samples are comparable with samples from other sites, or from particular artefact types (Blades 1995; Mortimer 1990). However, at Barrington a relatively large proportion of the samples are brasses with a few percent tin; 9 out of 26 (35%) have more than 10% zinc and less than 5% tin and seven of these points lie in a cluster, averaging 16.1% zinc and 3.4% tin. One sample is a good-quality brass, with a very high zinc content (424 Δ 19; 23.9% zinc). A second distinct grouping can be seen in the dataset, where zinc and tin contents are about equal - eight samples have average values of 5.4% zinc and 5.5% tin. There is no convenient, agreed descriptive term for these types of mixed alloys, but 'gunmetal' will be used here. Another significant grouping in the data is those with low zinc contents (seven samples with less than 2.5% Zn). These alloys have a variety of tin contents. In three cases, the zinc and tin contents are so low (below 4% Zn and 4% Sn) as to suggest any alloying was accidental, and these alloys can be termed 'coppers', since true coppers (with less than 1% zinc, lead or tin) rarely occur in this period. In another two cases, the tin contents are high enough (above 8%) to indicate a relatively-pure bronze, which is fairly common at this time. The final two points in this group are low-tin bronzes. Only three of the

ANCIENT MONUMENTS LABORATORY REPORT

26 samples (11%) lie outside these groupings and these are low-zinc brasses with significant tin contents ('tin brasses'). Lead contents are generally low throughout, with only four samples having 2% or more.

Several of the samples are from pairs or sets of finds which might reasonably be expected to have been made from the same alloy. Representing these points by average values effectively reduces the number of datapoints which have to be considered, as indicated on Figure 1, although it does not significantly change the overall proportions of alloy types used. For example, four of the 'copper' and low-tin bronze samples are from pairs of saucer brooches, 354 Δ 28 and Δ 29 and 428 Δ 99 and Δ 100. There is reasonable similarity between the alloys used for the latter pair. Although the tin contents of the saucer brooches from 354 are less close to each other (at 3.9% and 6.2%), they have similar low lead contents and a distinctive high iron content, which makes it possible that they were cast from the same melt, perhaps with some tin segregation. Dickinson (1993) found that some pairs of saucer brooches with very similar designs were cast with the same alloy, whereas others seemed to be cast with different alloys. The alloys of the brooches from 354 can be seen as comparable, if not identical. Similarly, six out of the eight gunmetal samples come from sheet metal artefacts which are parts of larger assemblages - Δ 8, Δ 26, Δ 40 and Δ 41 from 428 (of which Δ 8 and Δ 26 can be paired, as can Δ 40 and Δ 41) and Δ 58 and Δ 59 from 44b. Amongst the brasses, two of them were from a pair of wrist clasps from 626a (Δ 148 and Δ 149) and four from objects from 44b, two of which are sheet (Δ 36 and Δ 89) and two are cast artefacts (Δ 48a and Δ 49). The tin brass alloys were used to cast a pair of saucer brooches from 626a (Δ 145 and Δ 155) as well as an item from 44b (Δ 80a). Table 3 shows the number of individual samples and pairs/groups of samples of each alloy type.

Belonging to the same alloy type does not necessarily imply that two artefacts were made from the same melt. This study has only quantified a small range of elements, and the alloy type here is dependant on the zinc and tin contents, because there is relatively little variation in the lead content. A wider range of elements might suggest more or less similarity between pairs or sets of artefacts. But, even where a wide range of elements has been analysed for, it is difficult to state how similar compositions must be to be classed as 'significantly similar'. Multi-variate statistical analysis on a large dataset with a wide range of elements (*eg* a combination of Mortimer 1990 with Blades 1995) could be used to explore this problem, but this is too large a project for the current research. In addition to this lack of comparative studies, it should be remembered that analysis of two objects made from a single alloy could give slightly different compositions, when drilled and analysed, because copper alloys segregate during casting (tin-rich phases cooling last, leaving a relatively tin-rich surface and lead lying in pools, rather than dissolved throughout). In this project, porous areas and lead-rich pools were avoided during analysis (some porosity may be the result of lead-rich droplets being lost during sample preparation). This procedure which tends to give lower lead concentrations than would, for instance, be produced by a solution analysis method, but it gives analytical totals closer to 100%. Multiple analyses of comparable samples from Buttermarket, another Anglo-Saxon cemetery (Mortimer forthcoming) showed considerable variety in lead contents may be achieved if this precaution is not observed. Hence alloy heterogeneity could be quite significant when compared to solution analysis methods, which provide an average value for a much larger sample, often taken from deeper within artefacts (*eg* the 1cm penetration quoted in Craddock 1976). Similarly, although individual crystals within the metal cannot be seen in the SEM, it is likely that many of the areas analysed represent single crystals, because lead tends to pool at the edges of crystals. There is also the question of

ANCIENT MONUMENTS LABORATORY REPORT

analytical errors, although multiple analyses of the copper alloy standards (which should be homogeneous) suggests these are small (see standard deviations in Table 2).

Hence a certain amount of leeway must be allowed when suggesting identity on a compositional basis. For the moment, it is safe to conclude that if artefacts in a pair or set of artefacts are compositionally within $\pm 1\%$ tin and $\pm 2\%$ zinc of each other, they are 'similar', but it is not clear whether clusters of samples from several contexts, such as those in the gunmetal group, can be **proved** to be from the same melt, using the current data.

Discussion: There are several possible reasons for the range of alloy types used to make the artefacts at Barrington. For example, it could be suggested that different alloy types were used for different artefact types since these would have different constructional demands. It is certainly true that all but one of the low zinc alloys were found amongst cast artefacts, the exception being the rather pure bronze used for the sheet metal of 626a $\Delta 151$. However, the other alloy types seem to be spread fairly equally between cast and sheet samples, suggesting there was little deliberate alloy selection or design. Interestingly, the sheet metals are slightly more tin-rich than the cast metals, in each of the two alloy types where there are sufficient samples to give a pattern (brasses and gunmetals), but the differences and the numbers of samples are not enough for this to be significant.

Consideration of the manufacturing techniques used gives some explanation of this feature. Casting is a particularly robust method of manufacture, and there are few types of copper alloy which are not satisfactory for the purpose. Hence it is not surprising to see a range of alloys used here, as among cruciform brooches (Mortimer 1990). Similarly, all of the alloys used during the early Anglo-Saxon period were reasonably well suited to the type of sheet metalworking seen at Barrington, where the metals were not worked very heavily. Notably the Barrington artefacts have low levels of lead which were not high enough to significantly impair working properties (although the analytical problems discussed above should be noted).

Analysis of samples from seven cemetery sites, including the East Anglian ones at West Garth Gardens, Bergh Apton, Spong Hill and Morning Thorpe (Blades 1995), indicates that the balance of alloy types seen is very similar to that at Barrington. Blades also concluded that there was very little alloy selection for casting or working sheet metal at this time. Although there were relatively few wrought samples in his study, he did detect a slight bias towards zinc-rich alloys amongst the 26 sheet/wrought metal artefacts, due to six high-zinc brasses used for three tweezers at Spong Hill and three wrist clasps (including a pair) at Empingham (*op cit*, 151). This could partly be a chronological effect (see below), but it is interesting that zinc-rich alloys were preferred for wrought artefacts at Lechlade (Mortimer 1988).

Alloy selection might have been more important for gilded cast objects as it is said that certain alloys, such as heavily-leaded alloys, are difficult to gild properly. Only two pairs of gilded saucer brooches were sampled here; two other samples came from saucer brooches which had gilded appliqués, rather than being gilded cast artefacts. There does not seem to have been alloy selection for these items; certainly the levels of lead are so low at Barrington that it is difficult to discern a difference. This agrees with the results of an earlier project which showed there was very little difference between the lead contents of alloys used for gilded and ungilded cruciform brooches in the sixth century AD (Mortimer 1990, 378). Interestingly, Blades (1995, 136) noted that two pairs of gilded wrist clasps from Bergh Acton were 'coppers', in the terminology of this report, although he does not comment on the composition of the other gilded artefacts in his

ANCIENT MONUMENTS LABORATORY REPORT

study. Theophilus, writing in the twelfth century, specifies that a pure copper is best for gilding, but that copper alloys could be pickled before gilding, to remove lead from the surface (Hawthorne and Smith 1963, 145-6). Hence alloy selection need not have been critical for gilded artefacts if the metalworker prepared the surface suitably.

Another possibility might be that the different alloy types reflect changes in alloy use over time. Again, an earlier study showed that the earliest cruciform brooches are more often pure bronzes or brasses than later forms, which are more frequently mixed alloys (Mortimer 1990). This patterning was attributed to increasing pressure on metal resources, leading to increased recycling and resulting in many more mixed alloys. Patterning observed for saucer brooches is less clear (Dickinson 1993), admittedly using data from two different, and not entirely compatible analytical methods. Blades' 1995 data was not sub-divided chronologically within the early Anglo-Saxon period, although a brief inspection of the cruciform brooch analyses within his dataset indicates that earlier brooch forms were more frequently tin-rich than later and gilded forms, thus conforming to the already observed patterning.

It has proved impossible to provide any finer chronological detail at Barrington than division into Migration Period and Final phases. All the sampled artefacts from the site come from graves dating to the main Migration period phase (c.500-c.570AD), with only one undated sample (372 Δ1), so it is not possible to investigate changes in alloy use/preference over time at this site.

Conclusions: The data gained from a quantitative analysis of the copper alloy artefacts shows that there was minimal alloy selection or design, and that alloys from a broad range of compositional types were used for both cast and wrought artefacts. This is similar to the patterning seen at other sites of this period. Material from only one phase was analysed, so information about changes in alloy usage over time was not available.

Structure (Kilian Anheuser)

Analysis and results Five objects were sampled for an investigation of their tin and gold 'plating' - cast saucer brooch 354 Δ28, and applied saucer brooches 530 Δ53a and Δ56 primarily for their gilding and wrist clasps 626a Δ148 and Δ149 for their tinning. In this case, the samples were scraped from a small area of the plated surface (approx. 1 mm square) with a scalpel. The samples consisted of copper-alloy corrosion products mixed with small particles (a few tenths of a mm) of the plating. Samples were hot-mounted in carbon-filled Bakelite, ground and polished to a mirror finish to display a cross section of the plating. The sections were examined using optical microscopy, scanning electron microscopy with qualitative energy-dispersive X-ray analysis (EDX), and quantitative wavelength-dispersive electron microprobe (EMP) analysis.

Two samples included areas of gilding which could be examined; these were from the cast saucer brooch 354 Δ28 and the appliqué from the applied saucer brooch 530 Δ53a. Both were identified as amalgam gilding (fire-gilding) by their mercury content. The gilding on 354 Δ28 had a thickness of 4μ. Analysis of a drilled sample (Table 1) showed that the substrate was a copper alloy with low alloying levels. The gold-rich phase of the gilding contained 22-27% Hg, 8% Ag, and 2-3% Cu (results of three EMP analyses). Two further EMP analyses of a gold flake found within a drilled sample of the same brooch substrate gave similar compositions. A cross-section of the copper-alloy sheet metal appliqué from brooch 530 Δ53a included a small piece of

ANCIENT MONUMENTS LABORATORY REPORT

gilding on its front. The gilding layer had a thickness of 2.5-3 μm and contained 16-19% Hg and 1.2-1.5% Ag (three EMP analyses). The base metal used for the appliqué had a high copper content and contained only 1.2 % Sn and a small amount of Pb. Traces of iron, silver, antimony, bismuth, nickel and zinc were also detected, at levels comparable with those found in other early Anglo-Saxon copper alloys. No gilding was visible in the sample from 530 Δ 56.

The tinned samples showed a layer of tin/copper alloy (η -phase, Cu_6Sn_5) beneath a tin-rich corrosion layer. No layers of ϵ -phase (Cu_3Sn) were observed. On the back of both appliqué samples, 530 Δ 53a and 530 Δ 56, Cu/Sn intermetallics were found, which are probably remains of a tin solder which was originally used to attach the appliqué to the body of the brooches (Figure 2).

Discussion In fire-gilding, a paste of gold amalgam was made up by grinding together gold leaf and mercury, which was applied to the base metal surface. Subsequent heating to 250-350°C evaporated most of the mercury, but typically 8-25% Hg remained in the gold. A colour change from grey to dull yellow indicated sufficient heating (Anheuser 1996). Fire-gilding layers usually had a thickness of 2-10 μm . The relatively high mercury content (more than 20% Hg) in the gilding of saucer brooch 354 Δ 28 indicated that the object was heated to just about the required temperature but not much higher. Overheating could have caused the gilding to flake off the surface. Amalgam gilding was the standard gilding technique for Anglo-Saxon metalwork.

Tinned copper alloy surfaces and copper alloys which were soldered with a tin-rich solder show a layered structure of tin/copper intermetallics. The thickness ratio between the individual layers indicates the temperature to which the object was heated in the process (Meeks 1986 and 1993). The objects were heated above the melting point of tin, and tin metal was then wiped over the surface. Pure tin melts at 232°C, but if it was alloyed with lead, which was often the case with solder, its melting point could be as low as 183°C. The presence of a layer of η -phase (Cu_6Sn_5 , Figure 2) and the absence of an ϵ -phase layer (Cu_3Sn) between the copper and η -phase are characteristic for a low temperature process. The objects were heated to just above the melting point of tin or tin solder, but not any higher. This made sense both for tin plating and tin-rich solder, because overheating would have caused diffusion of the tin into the base metal and oxidation of the copper and would therefore have led to inferior results (such as low tin levels at the surface).

In a normal fabrication sequence of an appliqué brooch, the appliqué was first fire-gilded, then decorated (repoussé, chasing of patterns) and finally soldered to the body of the brooch.

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ANCIENT MONUMENTS LABORATORY REPORT

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Table 1: EDX analyses of copper alloy samples from Barrington

Context/SF	Sample		Weight % normalised						
	Manuf.	Artefact type	Fe	Ni	Cu	Zn	Ag	Sn	Pb
44b Δ36	s	pendant?	0.2	0.1	77.4	17.2	0.1	4.5	0.5
44b Δ48a	c	swastika disc brooch	0.2	tr	81.7	15.6	nd	2.3	nd
44b Δ49	c	disc brooch	0.2	tr	82.2	14.8	nd	2.3	0.1
44b Δ58	s	strapend?	0.2	0.1	86.4	6.4	tr	5.2	1.3
44b Δ59	s	strapend	0.2	tr?	86.6	5.8	0.2	5.2	1.8
44b Δ80a	s	plate	0.2	tr?	81.6	11.4	0.4	5.0	1.3
44b Δ89	s	strip	0.2	tr?	77.6	15.9	0.2	4.0	2.0
128 Δ11	c	small-long brooch	0.1	tr	92.7	1.5	0.5	5.0	0.1
354 Δ28	c g	saucer brooch	0.6	nd	94.2	1.2	nd	3.9	nd
354 Δ29	c g	saucer brooch	0.6	tr?	91.8	1.0	tr	6.2	0.1
372 Δ1	c a	applied disc brooch	0.2	nd	84.5	4.8	tr?	4.4	5.8
428 Δ8	s	sheet	0.2	tr	85.0	6.5	0.1	5.4	2.7
424 Δ19	s	sheet	0.1	tr?	74.9	23.9	0.1	0.8	0.2
428 Δ26	s	sheet	0.2	tr?	85.6	6.0	tr?	6.2	1.6
428 Δ40	s	plate with rivets	0.3	0.1	86.9	4.6	0.1	6.6	1.3
428 Δ41	s	plate with rivets	0.2	tr?	88.8	4.4	tr	5.8	0.4
428 Δ99	c	saucer brooch, pair to 100	0.1	tr	94.4	2.2	nd	2.9	0.1
428 Δ100	c	saucer brooch, pair to 99	0.3	tr	94.7	2.4	tr	1.9	0.4
530 Δ53a	c a	applied saucer brooch	0.5	nd	89.4	4.8	tr	4.8	0.2
530 Δ55	s	sheet	0.2	nd	80.3	14.1	0.1	4.0	1.3
530 Δ56	c a	applied saucer brooch	0.6	tr	89.6	1.1	tr	8.2	0.3
626a Δ145	c g	saucer brooch	0.3	nd	88.2	7.6	tr	3.0	0.7
626a Δ148	s t	wrist clasp, pair to 149	0.1	tr	79.1	17.4	tr	3.0	nd
626a Δ149	s	wrist clasp, pair to 148	0.1	0.1	78.3	17.7	nd	3.6	0.1
626b Δ151	s	sheet	0.3	nd	85.6	1.8	tr	8.9	3.0
626a Δ155	c g	saucer brooch	0.9	0.1	86.7	8.9	nd	3.3	0.1

Notes

All artefacts come from graves dated to within the Migration Period, except 372 Δ1 which is unstratified.

s = sheet metal, c = cast, t = tinned/soldered, g = gilded, a = applique.

nd = not detected (less than 0.1%) and tr = trace

It seems likely that the EDX method, as used here, will give lead contents which are slightly lower than those which would have resulted from a solution-based analysis method; this is because large lead-rich areas and porous areas (likely to be lead-rich) were avoided during analysis.

Table 2: EDX analyses of copper alloy standards.

		Weight %								
		Mn	Fe	Ni	Cu	Zn	As	Ag	Sn	Pb
BCS207	EDX	nd	nd	nd	83.8	2.3±0.05	0.4	0.1	10.8±0.1	0.4
	given				86.8	2.5	0.1		9.8	0.4
AC23	EDX	nd	tr	nd	70.2	29.8±0.05	nd	nd	nd	nd
	given				70	30				
C30*09	EDX	nd	tr	nd	85.2	14.7±0.2	tr	nd	nd	tr
	given				85	15				
C71*08	EDX	nd	nd	1.1	85.3	4.5±0.05	tr	tr	5.7±0.1	3.4±0.05
	given			1	84.5	4.5			5	5

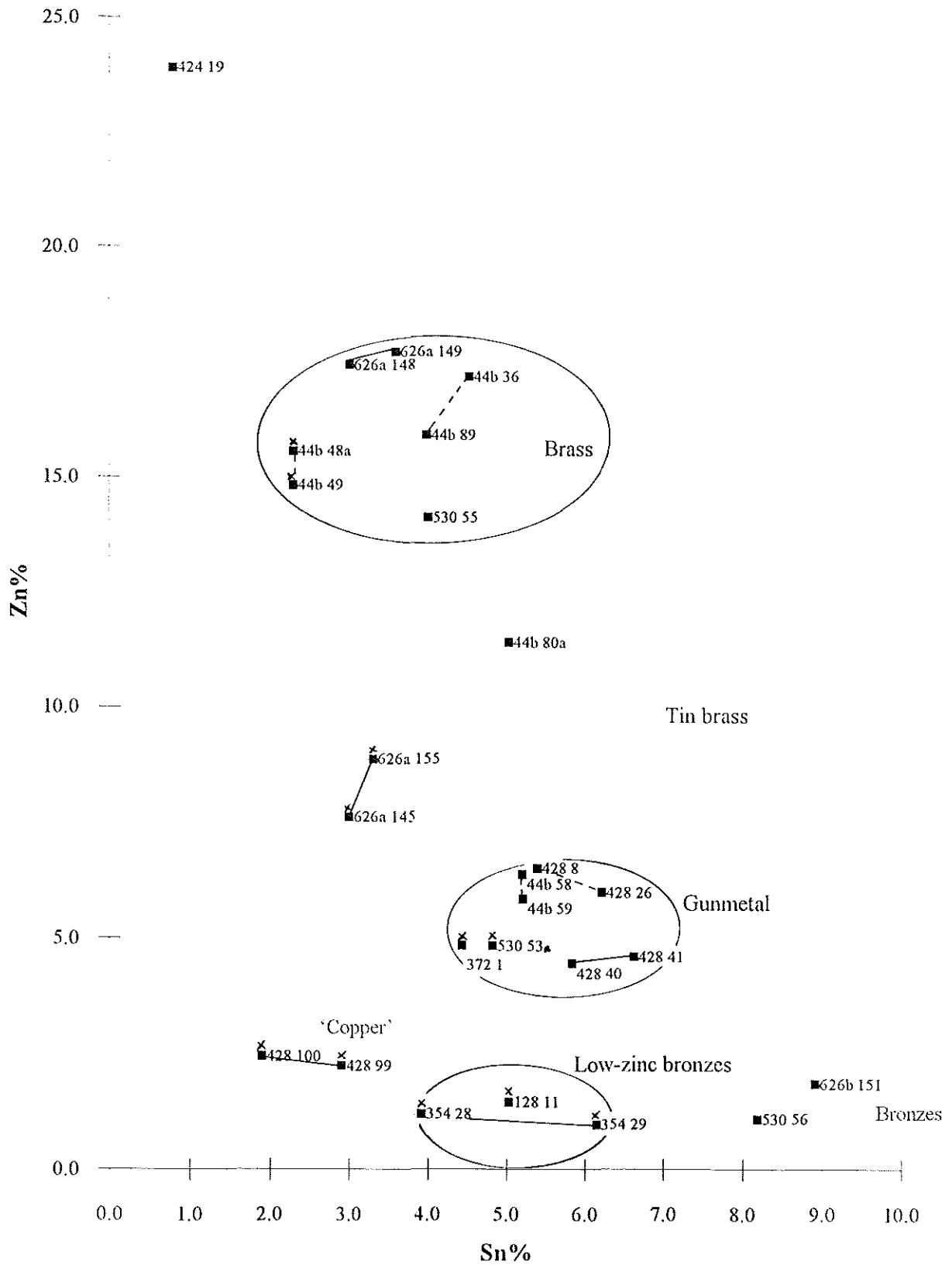
Codes as for table 1. Mean values (and standard deviations, where appropriate) of two or three EDX analyses are given in each case.

Table 3: Frequency of alloy types

Alloy type	Number of individual samples	Number, with pairs or groups of samples counting as one
Brass	8	5
Tin brass	3	2
Gunmetal	8	5
Low-zinc bronze	2	2
Bronze	2	2
'Copper'	3	1

Alloy types as shown on Figure 1. Brass includes one very high zinc brass. 'Copper' includes copper alloys with small amounts of zinc and tin (<4% of either), as purer alloys are rarely found at this time.

Figure 1: Zinc% vs tin% for all copper alloys



Code: Solid lines join samples from stylistically-comparable pairs or sets from single graves. Dashed lines join samples from single graves, where they are within $\pm 1\%$ tin and $\pm 2\%$ zinc of each other. X marks cast artefacts.

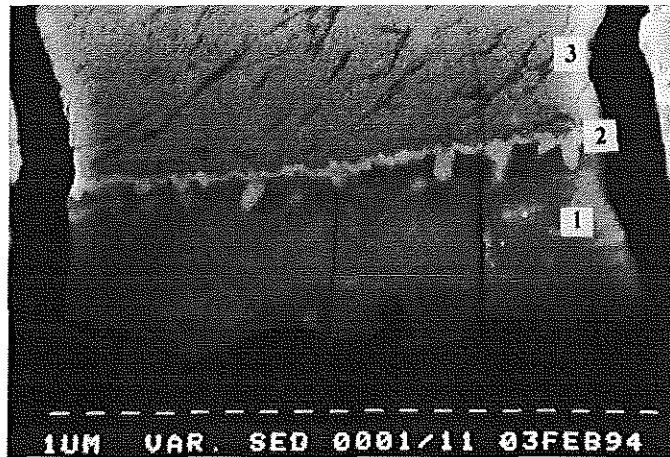


Figure 2: Cross-section through surface of tinned wrist clasp 626a Δ148. 1 = Tin-rich corrosion products on the surface of the artefact, 2 = layer of η -Cu/Sn intermetallic and 3 = corroded copper alloy base metal.