Analysis of Anglo-Saxon Solder Deposited on a Copper-Alloy Disc from the Cemetery at Marina Drive, Bedfordshire

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SUMMARY

Among the objects displayed at Luton Museum, Wardon Park is an artefact described in the Accession Register (52/120/58) as "Bronze Disc with punched decoration"; it was found, with other grave-goods in Burial E3, that of a young girl at the Anglo-Saxon Cemetery, Marina Drive, Bedfordshire. Its position in the grave indicates that it had been attached to her waist-belt or châtelaine, presumably to serve as a decorative ornament, possibly having some amuletic significance. The purpose of this article is to record the discovery and resulting scientific examination of solder found adhering to the underside of the disc and to offer an explanation as to its original function and purpose.

DESCRIPTION OF MARINA DRIVE DISC

This near circular flat polished copper-alloy disc (Fig 1.1, Plate 1) is approximately 50 mm \emptyset x 0.55 mm in thickness and is repoussé decorated with a border consisting of a single row of punch dots concentrically placed around its edge, inside this circle is an interrupted cross saltire meeting at a second smaller circle enclosing a central boss; a pierced suspension hole can also be observed. This pattern is a prominent Anglo-Saxon motif and can be seen decorating a number of seventh century silver-gilt scutiform pendants (Evison, 1987: Fig 37-3a) and (Hines, 1984: Fig 4-10).

On the underneath side (Fig 1.2, Plate 2), located in the void between the decorative border and the disc edge are a number of irregular shaped silver coloured metallic deposits. This substance has the appearance and touch of soft-solder, a material used in the process of joining metal together; usually consisting of lead or tin, or an alloy of the two, which has a lower melting range than that of the metal to be joined.

At the time of re-discovery the writer was engaged in a research programme relating to copper-alloy Anglo-Saxon relic boxes (Gibson, 1993) and attention was drawn to this disc because in material specification, size and decoration it has the appearance of a re-used lid top or base from such a box; furthermore, two other relic-boxes are known to have been recovered from this cemetery (Matthews, 1962), both from double burials Grave B3 and B4 and the adjacent burial to Grave E3 - Grave E1 and E2.

RELIC BOXES

Forty-five provenanced relic-boxes are recorded Table 1. These small often naïvely decorated containers are occasionally found in seventh and early eighth century female burials in cemeteries and barrow graves widely dispersed throughout Anglo-Saxon England. For over two hundred years since their earliest discovery relic-boxes have been considered unusual and enigmatic objects, their origin and function has been the subject of calculated guesswork and academic research and an aura of mystery has evolved around these small containers. Recent reports and studies Ager (1989), Crowfoot (1987), Evison (1987), Hawkes (1973, 1982) and Meaney (1981) have failed to reach a consensus as to their function and purpose and they have been variously referred to as work, thread or sewing-boxes, amulet capsules or Christian reliquaries; the latter description based mainly on the cruciform ornamentation sometimes displayed together with their appearances in what are often termed "Final Phase or Proto-Christian" cemeteries. These views and interpretations have been challenged by the author who argues that the decorations on the boxes are indicators of Pagan not Christian symbolism and they evidence a pagan survival rather than representing a "new" Christian accessory and they should be considered as pagan relic-boxes.

Two distinctive types can be recognized; Type I, Fig 2, the largest group with forty recorded can be defined as follows. They are two piece cylindrical copper-alloy boxes, between 40 - 70 mm in diameter and 40 - 72 mm in shut length. The top or lid assembly, partly slide-fits over the bottom or base assembly and is retained in its closed position by metal friction between the inside diameter of the lid assembly, and the outside diameter of the base assembly. Without exception each box originally consisted of four principal elements, lid-top,



Figure [1] Copper-alloy disc Grave E3 Marina Drive. 1.1 Top view. 1.2 Under side showing solder.



Plate 1. Top view. Scale 1:1.



Plate 2. Under-side showing solder. Scale 1:1.

lid-ring, body and body-base, all manufactured from copper-alloy sheet between 0.45 and 0.65 mm thickness. Individual components were assembled together by bronze rivets, solder or were mechanically retained by metal crimping, or any combination of these processes. Lid-tops are one piece circular or near circular convex or flat discs, often decorated with internally applied punch dot impressions in various styles and motifs (Fig 3). Lid rings are made from a single piece of strip formed into shallow open ended parallel tubes, jointed or seamed along their length, these are often bell-mouthed or flared at one end to make a platform to which the lid-top is fitted. The circumference of these rings is usually stamped from the inside with row(s) of raised punch dots applied to the flat sheet-metal prior to forming and final assembly. Attached and externally positioned on the joint is a single bronze split eyelet or bronze loop. This acts as an attachment point for a linked bronze or iron

DESIGN PRINCIPLES AND GENERAL ARRANGEMENT IN WORN POSITION



Figure [2] Design principals Type 1 relic-box.

chain complete with a central ring and serves the dual purpose of retaining the lid to the body, and also suspending the relic-box from the wearer's châtelaine or waist-belt. Relic-boxes' bodies are longer versions of the corresponding lid-ring formed and constructed in the same manner with an identical method of chain attachment; however the greater material area allows space for a more elaborate degree of artistic impression, an example of which can be seen on the Hawnby North Yorkshire box (Fig 4). Body-bases can be identified with lid-top in respect of material, shape and the method adopted in their attachment to the body. When decorated they often reflect the aesthetic quality of the tops, although not all carry the same motif and some are left plain.

Type II boxes, five in number, unlike the Type I classification are noticeably different in appearance from one another. They do however have enough common characteristics to enable them to be placed within a specific group. They share some features with Type I boxes in that their lid-tops, lid-rings,

body and body-bases are constructed in a similar fashion from the same material. Additionally, however, all have either a rigid or pivoting suspension flange, plate or arm projecting from one side of their body like the North Leigh Oxfordshire box (Fig 5); their lid assemblies were so designed that they were secured to the body assembly by either a wire ring, hinge or pivot pin; these allow the lid assembly to open or close onto the body without becoming totally detached.

NOT TO SCALE.

As the lid tops and bases of both types are identical in design it is not possible to determine from which classification the Marina Drive disc originated, although the two other boxes found in the cemetery are both Type I. Nevertheless, the position of the solder like substance leaves little doubt that it had at sometime been attached to a circular component, most probably a lid-ring from a relic-box.

During the course of my research a number of relic-boxes were reconstructed using only tools and manufacturing techniques employed by craftsmen



Figure [3] Examples of repoussé patterning on relic-boxes. [a] Polhill. [b] Kingston Grave 96. [c] Marina Drive Grave E3. [d] Uncleby Grave 1. [e] Sibertswold. [f] Ashmolean Museum. [g] Garton II. [h] Uncleby Grave 3. and metal-smiths of the early and middle-Saxon period so the author was confident that the Marina Drive disc was a re-used component from such a box. What I was unable to determine was whether the original excavator, Les Matthews or the conservation staff at the museum at the time of discovery, (1957) had attempted to re-construct individual components from the other two boxes found at the cemetery with the Grave E3 disc using modern solder. Additionally, I needed to know if the disc had originally been silver-gilded, a decorative finish that features on a number of relic-boxes including the Marina Drive Grave B3 and B4 and another from Kempston Bedfordshire (Fitch 1864); could the metallic substance be all that remained of these plating processes?

SOLDER ANALYSIS: COPPER-ALLOY LID-TOP, MARINA-DRIVE

GRAVE E3

Solders have been in use in the British Isles certainly since the late Bronze Age. A range of solder alloy compositions have been utilised, with the majority apparently based on the tin-lead system (Tylecote, 1986). This situation has not changed until the last decade or so. During 1957 when the artefact was discovered, there were three major uses for solders, plumbing, electronics, and the canning industry. Plumbers generally used alloys containing 50 (weight) % lead and 50 % tin (50 Sn 50 Pb). This alloy does not have a melting point as such, but on cooling from the melt first becomes pasty (during which time it can be conveniently worked) before solidifying, The requirements of the electronics industry are different and they typically, even today, use 60 Sn 40 Pb, or 63 Sn 37 Pb alloys, which have a sharp melting point. The canning industry on the other hand used a high lead content solder with a composition of 98 Pb 2Sn. The tin is essentially the active component in all of the alloys and is largely responsible for their good wetting and bonding properties. The addition of lead does confer a number of advantages, reducing the cost of the alloy while at the same time improving mechanical properties. In the case of plumbing and electronics solders it reduces the working temperature of the alloy, which is of particular importance in the latter case where excessive temperature excursions can damage both electrical components and the printed circuit boards on which they are mounted. Inclusion of lead also negates the possibility of both tin-whisker formation, and the occurrence of 'tin pest' (allotropic conversion of white tin into grey), which are important practical considerations to industry. As a result of the these advantages tin-lead solders have dominated the soldering market for most of the current century and the use of pure tin solder has been quite rare.

The specimen was examined using a Jeol 5400 scanning electron microscope (SEM), equipped with a conventional Everhard-Thornley secondary electronic detector and a four quadrant solid state back scattered electron detector. Within the SEM a finely focused beam of electrons is repeatedly scanned over a rectangular area of the specimen. The electron bombardment causes the material immediately under the beam to emit low energy secondary electrons, back scattered primary electrons and characteristic X-rays. Monitoring the intensity of the secondary and back scattered electron signals as a function of the position of the beam on the sample permits the formation of high resolution images of the specimen. Secondary electron images reflect the topography of the surface. The contrast in back scattered images, on the other hand, is primarily due to differences in the mean atomic number of the materials present and hence effectively provides a high resolution map of chemical composition.

A Link QX2000 energy dispersive X-ray analysis system was used to carry out microanalyses, and was used in both ultrathin window and windowless modes. The technique is capable of detecting all elements in the periodic table with atomic number greater than 4, with detection limits of the order of 0.1-1 atomic %. The depth of analysis and spatial resolution of the technique depend on a number of factors but are typically 0.5 - 2 micrometres.

The artefact had a generally bright, bronze appearance. EDX analysis indicated that the artefact was made of a copper-tin bronze. There was evidence of widespread pitting, undoubtedly associated with corrosion. There were, however, no major deposits of corrosion products present suggesting that they must have been removed at some stage. Solder was present on the outer periphery of the structure, consistent with its use to attach the artefact to another object. The outermost areas of the solder were roughened, presumably due to the mechanical disruption of the joint. Inner areas of the solder had a much flatter, mirror-like appearance where the molten metal had apparently flowed away from the joint during soldering.

A combination of scanning electron microscopy (SEM) and energy dispersive X-ray analysis (EDX) were used to study the morphology and composition









Figure [4] Hawnby North Yorkshire relic-box.







Figure [6] EDX spectrum of solder, as received.

of the solder. Fig 6 shows a typical EDX spectrum taken from the sample, indicating the presence of tin, copper, phosphorous, chlorine, oxygen and carbon. To distinguish between bulk constituents of the alloy and surface contaminants, the solder was gently scratched using a scalpel blade. Subsequent examination in an SEM revealed that a film of material was present on the surface. EDX analysis in the scratched areas detected only tin and copper, Fig 7. In the light of the general appearance of the specimen, and in particular the absence of corrosion products, the most plausible explanation of the EDX findings is that after its discovery the specimen had been cleaned by pickling in a hydrochloric acid solution (to remove oxide scale), followed by passivation in a phosphating solution. Pickling/passivation treatments of this type have been used for many years, for cleaning and preserving metallic surfaces. The fact that the chloride/phosphate layer was on top of the solder suggests that the solder was already present when the cleaning/passivation treatments were applied. and hence that it is indeed Anglo-Saxon.

The absence of lead in the solder is of great interest. At the time of discovery of the specimen virtually all solders contained high levels of lead, for the reasons outlined above. More recently, in the last decade or so, lead-free formulations have been introduced because of toxicological concerns over the use of lead. But at the time in question, such matters were not appreciated and lead-containing solders were the norm.

The surface of the solder was generally flat and apparently single phase, with no signs of either the presence of any second phase or cavitation caused by the chemical removal of one. The observed copper signals were almost certainly derived not from the solder itself but from the copper-tin substrate immediately below it, i.e. the depth of analysis of the EDX technique was greater than the thickness of the free solder. If obtained from a homogeneous sample the copper signals detected from the solder would correspond a Sn-Cu alloy containing 30-35 weight % copper. Such an alloy would be contained several phases, including a copper-in-tin solid solution phase and copper-tin intermetallic compounds (primarily Cu6Sn5), (Hansen, 1958). The different phases should be readily visible in the SEM, none, however, were observed. Moreover it would have a liquidous temperature of 600-650° C and thus be useless as a solder. To test this theory analyses were carried at a



Figure [7] EDX spectrum of scratched area on solder.

range of accelerating voltages (15, 20 and 30 KeV) - effectively changing the sampling depth of the analysis technique, from approximately 0.5 to 1.7 micrometres. Increasing the depth of analysis was observed to result in an increase in the copper signals relative to those of tin, to a much greater extent than would be expected from a homogeneous material, supporting the theory that we are looking at a thin layer of tin on a copper-rich substrate.

As mentioned above tin reacts strongly with copper, forming intermetallic compounds at the copper-tin interface. Cu₆Sn₅ is usually the first compound to form, and this may be followed by a second layer of Cu₃Sn which forms at the Cu₆Sn₅ - substrate interface. On recently soldered joints, the thickness of free solder is normally always larger than the sampling depth of the EDX technique. It is most unusual to observe substrate signals since this implies that the free solder layer is less than 0.5 micrometres deep. A possible explanation of this unusual feature is that a thick intermetallic layer has formed at the interface, by progressively consuming the overlying free tin layer.

Intermetallic compounds form at copper-tin interfaces, even at ambient temperatures. Typically of the order of 1 micrometre of intermetallic compounds form during the soldering process itself. Studies of intermetallic growth thereafter indicate that the growth rate can be estimated with reasonable accuracy using a parabolic relationship (Klein-Wassink, 1989);

thickness = $k\sqrt{t}$

where $k = k_0 exp(-Q/RT)$

and t = time, Q = the activation energy for growth, R = the gas constant and T = absolute temperature.

On (pure) copper at 20° C the growth constant k has been measured to be $0.26 \text{ nm.s}^{-1/2}$, (Klein-Wassink, 1989). Thus over the lifetime of an Anglo-Saxon tin soldered joint one might expect a substantial quantity of intermetallic compounds to be formed, and this provides a useful method for checking the antiquity of soldered joints. Unfortunately, this could only be verified in the present case by destructive means, for example by cross sectioning a portion of the soldered area, and this was considered to be unacceptable.

DISCUSSION

In metal specification, size, thickness and shape the Marina Drive Disc can be paralleled to many of the circular lid-tops and bases incorporated into extant relic-boxes; additionally, the method of decorating by repoussé is a prominent feature on 35 (77.8%) of known boxes. Metallurgical evidence allied to the position of the solder indicates the disc had at sometime been an integral component of a circular box-like object, most probably a relic-box. Furthermore and most unusually, un-alloyed tin had been used as an agent in its original construction; had this process been carried out at, or after the time of discovery it is more likely that a modern alloyed solder would have been used. This observation combined with evidence of tin solder underlying 1957 conservation material indicates further that it is indeed of Anglo-Saxon origin and can with some confidence be assigned on scientific, excavation and typological evidence to the mid-seventh century AD.

Unlike others, the writer is unconvinced that the decorative pattern displayed on the disc has ever had Christian significance, instead it reflects only an unsophisticated, popular art-style of its day. Elsewhere (Gibson 1993) the view has been expressed that relic-boxes form the signifier of a conventional pagan rite, they were worn to indicate the completion of that passage; in the circumstance of conversion, some may have abandoned them, others by contrast, continued to wear them as attractive dress accessories. The hypothesis in respect of the Marina Drive disc is that it represents all that remained of an old dilapidated box, which, as an important pagan talisman was utilised as a pendant, and like the earlier relic-box, was worn, suspended from the young lady's waist.

That the pendant accompanied other grave-goods including a necklace comprising amethysts, glass, amber, fish-bones and ceramic beads, together with a cowrie shell, (most probably a fertility amulet) suggests that under the increasing influence of Christianity, now was the time to retreat from the outward trappings of pagan custom and practice and the disc was finally consigned to the grave at Marina Drive.

TABLE 1. Relic-boxes by County (pre-1974 re-organisation)

	County	Site	Total from Site	Total for County
	Bedfordshire	Kempston	2	
		Marina Drive	3	5
	Cambridgeshire	Barrington 'A'	1	
	0	Barrington'B'	1	
		Burwell	2	
		Haslingfield	3	7
	Derbyshire	Hurdlow	1	
	100 10 10 10 10 10 10 10 10 10 10 10 10	Standlow	1	2
	Gloucestershire	Lechlade	1	1
	Hertfordshire	Verulamium		
		King Harry Lane	2	2
	Kent	Dover-Buckland	1	-
		Market Square	1	
		Finglesham	1	
		Kingston Down	2	
		Polhill	1	
		Sibertswold	1	
		Updown	1	8
	Lincolnshire	Barton-on-Humber	3	3
	Norfolk	Caister St. Edmunds	1	1
	Northamptonshire	Cransley	1	1
	Oxfordshire	North Leigh	1	
		Standlake	1	2
	Warwickshire	Bidford-on-Avon	1	1
	Wiltshire	Yatesbury	1	1
	Yorkshire	Aldborough	1	
		Garton II	1	
		Hambleton Moor	1	
		Hawnby	1	
		Painsthorpe Wold	1	
		Uncleby	5	10
	Unprovenced	Ashmolean	1	

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