



***Staffordshire Hoard
Research Report 4***

**The Scientific Analysis of Niello Inlays
from the
Staffordshire Hoard**

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Information about this report

This report was produced in 2013 as part of Stage 1 of the project, i.e. before fragments were joined and catalogued. The concordance of the K numbers given in the report to the catalogue numbers as they appear in the final publication is as given below. The list also includes the names of the objects as used in the final publication.

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K number	Catalogue number	Name in publication
82	569	Mount with fantail in silver with geometric niello inlay and gilded borders (part).
137	409	Pair of hilt-guards in cast silver, with panels of gilded interlace, and gold mounts with filigree decoration and a gem-setting (part).
160	187	Hilt-collar in cast silver, of narrow form, with gilded low relief decoration and black niello inlay (part).
182	569	Mount with fantail in silver with geometric niello inlay and gilded borders (part).
242	76	Pommel in cast silver, of cocked-hat form with double sword-rings, with cast interlace and niello inlay and mounts with cloisonné and filigree decoration (part).
291	76	Pommel in cast silver, of cocked-hat form with double sword-rings, with cast interlace and niello inlay and mounts with cloisonné and filigree decoration (part).
304	186	Hilt-collar in cast silver, of narrow form, with gilded low relief decoration and black niello inlay.
310	567	Eye-shaped mount in silver with geometric niello inlay and filigree trim.
347	56	Pommel in gold, of round-back form, with incised decoration inlaid with niello and filigree ornament.
550		Inscribed strip
660	166	Hilt-collar in gold, of high form, with cloisonné decoration.
713	570	Strip-mount in silver with geometric niello inlay and gilded borders (part).
1005	569	Mount with fantail in silver with geometric niello inlay and gilded borders (part).
1021	568	Mount of eye-shaped form in silver with geometric niello inlay (part).
1069	570	Strip-mount in silver with geometric niello inlay and gilded borders (part).
1235	571	Strip-mount in silver with pointed turned ends, geometric niello inlay and a gilded border (part).
1364	187	Hilt-collar in cast silver, of narrow form, with gilded low relief decoration and black niello inlay (part).
1630	569	Mount with fantail in silver with geometric niello inlay and gilded borders (part).

DEPARTMENT OF CONSERVATION AND SCIENTIFIC RESEARCH

The scientific analysis of niello inlays from the Staffordshire Hoard

Science Report PR07444-3

Susan La Niece

Abstract: A number of silver objects and fragments and also some gold items from the Staffordshire Hoard have black inlays which are likely to be niello. Samples of some of these were collected during conservation work by Cymbeline Storey, Hoard Conservator at Birmingham Museums Trust. Other examples have come to light amongst the Hoard pieces being studied and conserved at the British Museum. The main analytical technique employed to characterise the types of niello in the Hoard was X-ray diffraction (XRD) with some energy dispersive X-ray analysis in a scanning electron microscope (SEM-EDX).

The surprising and notable finding at this stage of the study is that all the inlay materials identified optically and confirmed analytically as niello, whether on the silver, silver gilt or gold items from the Hoard, have proved to be of the silver sulphide type (Ag_2S). No examples have yet been found of the silver-copper sulphide niello which has previously been reported to be the more common type amongst niello inlays in analytical surveys of Anglo-Saxon silver and silver gilt items (La Niece 1983 and 1988). By no means all the inlaid items in the Hoard have been analysed and widening the survey to include all the niello inlays would complete the picture emerging from these analytical findings, increasing the understanding of the relationship of the inlaid items in the Staffordshire Hoard with Anglo-Saxon inlaid metalwork more generally.

CSR Project no. PR07444

Staffordshire Hoard numbers: K82, K137, K160, K182, K242, K291, K304, K310, K347, K550, K660, K713, K1005, K1021, K1069, K1235, K1364, K1630

22 March 2013

Introduction

Niello is a black decorative inlay material manufactured from metal sulphides and applied to gold, silver and copper alloy objects. It has a long history of use (La Niece 1983 and 1998) and was a popular decorative technique in the Anglo-Saxon period for silver and gold military fittings, female dress ornaments and objects with Christian iconography although, surprisingly, there seems to be no Old English term directly referring to niello (Coatsworth and Pinder 2002; 116-118, 248-249). Two compositions of niello have been analytically identified on Anglo-Saxon metalwork – silver sulphide (acanthite - Ag_2S) and silver-copper sulphide (stromeyerite – CuAgS) (La Niece 1988). Lead sulphide niello is unknown in Anglo-Saxon metalwork and does not appear in England until the late Medieval period (La Niece 1983).

A number of silver objects and fragments as well as some gold items from the Hoard have black inlays which are likely to be niello. Samples of some of these have been collected by Cymbeline Storey, Hoard Conservator at Birmingham Museums Trust (BMT), during the conservation work. The items sampled are illustrated in Storey (2013). Other examples have come to light amongst the Hoard pieces being studied and conserved at the British Museum. The main analytical technique employed to characterise the types of niello in the Hoard was X-ray diffraction (XRD) with some additional use of energy dispersive X-ray analysis in a scanning electron microscope (SEM-EDX). XRD is the preferred method for identification of the metal sulphides because it identifies the compound present, not just the individual elements present. For example, silver artefacts are made of an alloy of silver with copper (and other elements often present as impurities). Surface X-ray fluorescence (XRF) or SEM-EDX analysis of an inlay in a corroded silver artefact will detect silver and copper, together with chlorine, bromine etc. from the metal corrosion as well as the elements present in the inlay and it can be difficult to be certain whether the copper is an ingredient of the niello, or if it is simply a carbonate or chloride corrosion product on the metal. The identification by XRD analysis of the specific sulphide compounds leaves no such uncertainty.

Methodology

All samples were first examined under low magnification for their suitability for analysis, in terms of size and homogeneity.

Suitable samples were analysed by Debye Scherrer powder camera X-ray diffraction using a Philips X-ray generator with an X-ray tube with copper target, operated at 40 kV. The resulting diffraction pattern was captured on radiographic film and identified by comparison with the database of the JCPDS International Centre of Diffraction Data.

Some samples were large enough to be mounted on an adhesive carbon stub for examination in a Hitachi S-3700N variable pressure scanning electron microscope, and analysed semi-quantitatively using energy dispersive X-ray analysis.

Staffordshire Hoard objects with inlays that were being worked on at the British Museum were sampled under low magnification with a clean scalpel and the resulting powder (about the size of a pin head) was collected directly onto a gelatine filament which was mounted in the Debye Scherrer powder camera for analysis (Figures 1 and 2).



Figure 1: K242 with sample position marked. Scale bar = 5 mm

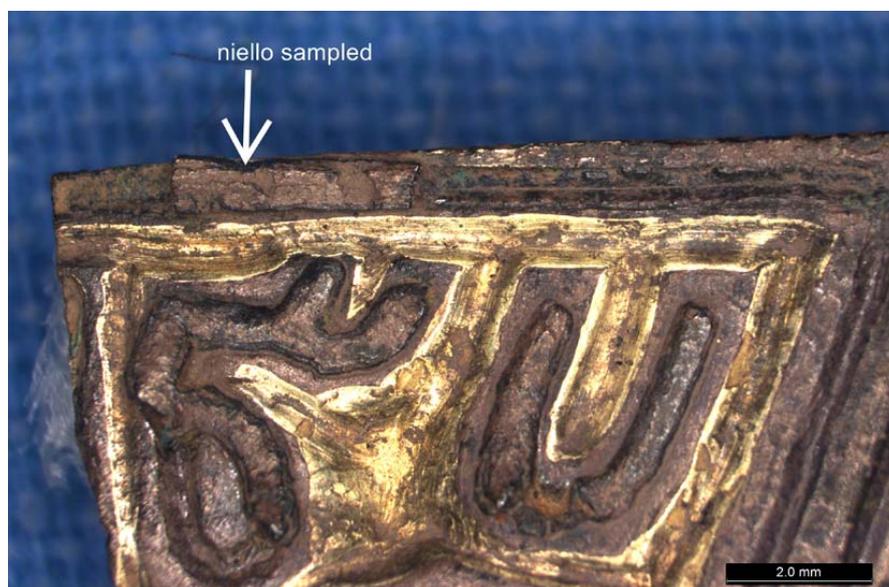


Figure 2: K160 silver-gilt with niello. Detail showing sampled area (small depression on the straight edge at the top). Scale bar = 2 mm.

Results and discussion

Table 1 below lists all the objects in which silver sulphide niello (Ag_2S) has been analytically identified. No examples of silver-copper sulphide niello (AgCuS) have been identified so far. The K numbers listed in groups in table 1 are those identified by Storey (2013) as fragments from the same silver or silver gilt object or same set of objects, on the basis of their design and, in some cases, of matching break edges.

Gold hilt collar K660 (Figure 3) is noteworthy as it has gold cloisons which would normally be expected to contain garnet inlays; the tops of the cell walls are even rubbed over to hold the inlaid stone or glass in place. However no garnets can be seen and most of the cells are filled with green or black material. The black material has been identified as silver sulphide and fills the cells which are approximately 3 mm deep. This thickness of sulphide would be most unlikely to form due to burial conditions, especially as sulphides are not the predominant corrosion compounds found amongst the Hoard, so it would appear that the sulphide somehow found its way onto the piece during its destruction, or it was applied deliberately during manufacture, i.e. as a niello inlay, albeit an unusual one.

The green material which has copper as the main component is as yet unidentified. It is not clear if the green fill is a decayed inlay material, backing paste or metal corrosion, though the latter seems unlikely as the hilt collar has no copper components. K 660 is being further investigated by SEM-EDX in conjunction with conservation cleaning, to clarify the questions of its decoration.



Figure 3: Gold cloisonné hilt collar with no garnet inlays. Some of the cells are filled with a black material identified as silver sulphide.

Table 1: Objects in which silver sulphide niello (Ag₂S) has been analytically identified. [# indicates those provided by BMT as samples, nd =not detected].

Silver Group	Object description	Object K no.	XRD film no.	SEM-EDX semi-quantitative analysis approx. weight %		
				Ag	Cu	S
A	Silver lentoid and fragments of its pair	K310 #	17674	85	nd	15
		K1021 #	17698			
B	Silver strip with gilt border. Triangular inlay	K1235 #		87	1	12
C	Silver strip with gilt border. Triangular inlay	K713 #	17694			
		K1069 #	17683	86	nd 9% Fe + Au	4
D	Silver fitting, gilt border, pins. Stepped inlay	K1630 #	17675			
		K82 #		88	1	11
E	Silver strip with gilt border. Mushroom inlay	K1005 #	17678	85	0.5	14
		K182 #	17685	85	nd	15
F	Silver gilt hilt collar	K1364 #	17705			
	Silver gilt sword fitting	K304	17699			
	Fragment, silver gilt hilt collar	K160	17700			
I	Fragmentary silver pommel	K291	17702			
K	Silver gilt with greenish glass inlay	K137 #	17695			
N	Silver filigree bead	K242	17701			
Gold						
	Gold chape cap. Style II zoomorphic design	K347 #	17687			
	Gold hilt collar	K660	17624			

Fourteen of the samples supplied were found to be either too small for analysis, or were identified as consisting largely of silver and copper corrosion products rather than niello. These samples came from K94, K241, K538, K592, K831, K953, K975, K1098, K1112, K1168, K1192, K1334, K1384 and K1631. This does not indicate that these items do not have niello inlays, only that they have not yet been analytically confirmed as niello.

The black inlay material in the inscription on the folded gold strip K550 is almost certainly niello but the identification has not yet been confirmed. It was analysed using proton induced X-ray emission (PIXE) at the Accélérateur Grand Louvre d'Analyse Élémentaire (AGLAE), Paris, in November 2010.¹ PIXE is an analytical technique which quantifies the elements

¹ AGLAE beam time was made possible under the FIXLAB transnational access programme of the CHARISMA project. Financial support by the Access to Research Infrastructures activity in the 7th Framework Programme of the EU (Grant Agreement n. 228330) and the help of the AGLAE team is gratefully acknowledged.

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present (Calligaro 1997), but unlike XRD, does not identify the compounds. The PIXE found silver and iron to be the main components; with copper and sulphur also present (other elements detected include zinc, gold, lead and bromine). This analysis could indicate that the inlay is niello, but does not prove it, and does not indicate whether the copper is a component of the niello or contamination from corrosion. This illustrates the point made above about the importance of the use of XRD analysis for identification of niello.

Conclusions

The surprising and notable finding at this stage of the study is that all the inlay materials identified optically and confirmed analytically as niello whether on the silver, silver gilt or gold items from the Hoard have been proved to be of the silver sulphide type of niello (Ag₂S). No examples have yet been found of stromeyerite (AgCuS) niello which was found to be the more common type amongst Anglo-Saxon niello inlays from previous analytical surveys of silver and silver gilt items. These previous surveys focused mainly on brooches from burials in Kent and the Isle of Wight, and also some military equipment such as the late 8th century silver gilt sword hilt from Fetter Lane, London (BM1893,0715.1) (La Niece 1983 and 1988).

Niello inlays in gold items of all periods are almost always of the silver sulphide type, and the large gold buckle from Sutton Hoo mound 1 (BM1939,1010.1) is no exception, so it is not surprising to find that all the examples analysed here from gold objects are silver sulphide. Gold cloisonné hilt collar K660 is unusual in apparently having some cells filled with silver sulphide. This piece is being further investigated using SEM-EDX in conjunction with conservation cleaning, to clarify the questions of its decoration.

By no means all the inlaid items in the Hoard have been analysed and widening the survey to include all the niello inlays would complete the picture emerging from these analytical findings and increase the understanding of the relationship of the inlaid items in the Hoard with Anglo-Saxon inlaid metalwork more generally.

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