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Danish niello inlays from the Iron Age
A technological investigation

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INTRODUCTION

Niello is the collective term applied to silver, copper and/or lead sulphides, which are inlaid as dark embellishments on bright metal objects. Fire gilding is often included as a further decorative element. The word niello comes from medieval Latin nigellum which means dark or black.

In earlier research, considerable attention has been focused on the techniques by which niello inlays were produced, but only the medieval, easily-meltable, lead-containing sulphides, which are described in several primary historical sources, have been fully investigated.

Many of Denmark's niello-inlaid artefacts from the Iron Age are to be found in the collections of the National Museum's Department of Prehistory and Early History and the investigations described here are confined to these.

In the following account, earlier research in the field is reviewed, the technological investigations of this project are described, and the results of these are compared with previously published niello analyses. Finally, the micro-topographical characteristics of niello inlays are described along with the possible reasons for the breakdown of the sulphides.

Finds from the Roman Empire from the 1st century AD bear witness to the use of niello decoration and the technique was spread to the rest of Europe in the course of subsequent centuries. The use of the niello technique is thought to have ceased in Scandinavia at the end of the Viking Age, after which it was largely confined to religious metalwork produced further south in Europe. In Italy in the 15th century, niello was widely used for many different purposes only to be completely replaced in the course of the 16th century by patterns in enamel (La Niece 1983; Oldeberg 1966). Benvenuto Cellini (Ashbee 1967) wrote in 1530 that the niello technique had been forgotten when he became an apprentice goldsmith in Florence in 1515, but elsewhere, for example in Russia, the tradition continued into the 18th and 19th centuries.

PRIMARY SOURCES AND EARLIER RESEARCH

Schweizer (1993) presents the previously published niello analyses of archaeological artefacts chronologically (fig. 1). Byzantine niello, which was of particular interest for his investigations, is shown separately. Pure silver sulphide was termed type I, silver-copper sulphide type II and niello of silver-copper and lead sulphide type III. Also included in the table are niellos of pure copper sulphide which have only been found on some bronze or brass objects from Roman times.

In conjunction with the information from La Niece (1983), Schweizer's table shows that niello of pure silver sulphide (type I) occurs with varying intensity from the 1st century AD, through to around 1000 AD. Silver-copper sulphide (type II), with a significant copper content was most prominent at the end of the 5th century AD and in the 6th century AD, but its use continued up until the 13th century.

According to La Niece (1983), the proportion of copper in the niello of type II was clearly greater in

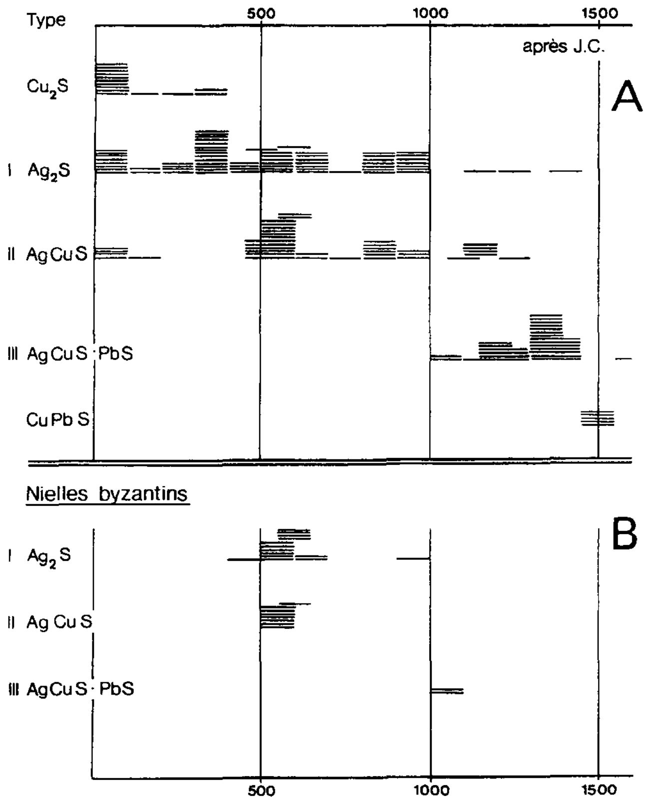

Fig. 1 Published analyses of niello inlays from 0-1500 AD, collected by Schweizer (1993). In diagram B all the Byzantine niello inlays, which were the subject of the article, are separated out. The references are Moss (1953), Schweizer (Lazovic et al. 1977), Dennis & Meyers (1979), Newman et al. (1982), Oddy et al. (1983) and La Niece (1983).

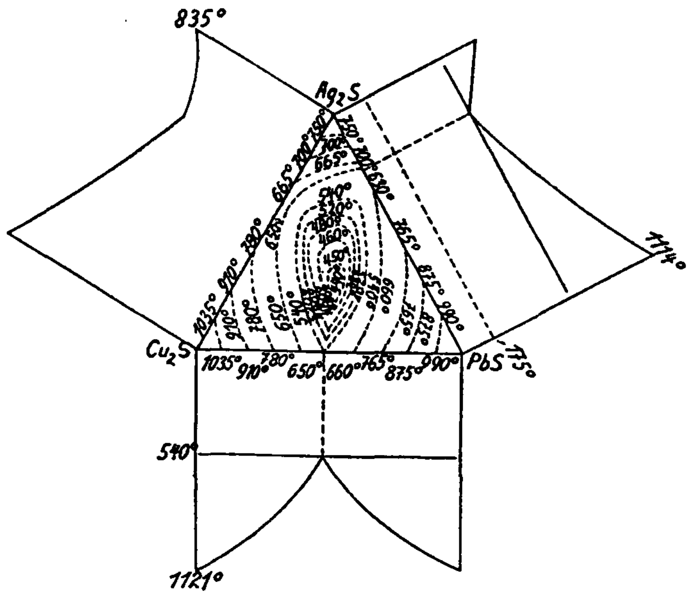

Fig. 2 Phase diagram for the tertiary system Ag_2S-Cu_2S-PbS (Schwarz & Romero 1927), where the melting points are marked with lines like contours on a map. The binary systems are drawn as foldouts.

the northwestern European material she had examined than in the few early examples from Roman times, where the parent material could have been silver of poor quality, rather than that the copper was an intentional addition. From the 11th century onwards, the use of lead-containing niello (type III) became widespread, corresponding to the formulae in medieval technical treatises. In general, the use of sulphides lacking lead was abandoned in the course of a couple of centuries in favour of the less time-consuming easily-meltable leaded niellos.

Pliny (23-79 AD)(Rackham 1968) describes the fusing together of 3 parts silver, 1 part copper plus some sulphur. In the later detailed treatises about lead-containing niello by Theophilus (1110-40 AD) (Hawthorne & Smith 1963), Cellini and others describe how the parent metals are melted together before the sulphur is added. The cast and hot-forged niello is later crushed, mixed with a flux (borax) and melted into the depressions in the artefact just like enamel powder.

Moss (1953) was aware that pure silver sulphide decomposed before it melted. He explains that the fact that silver, copper and lead could be blackened by sulphur must have been known from very early times, but the production of easily-meltable niello was not possible before mixed sulphides were introduced. The reason for this is that single metal sulphides decompose before melting i.e. the sulphur burns off. If, however, they are mixed together in suitable proportions, lower melting points are obtained, just like when metals are alloyed. Mixtures of silver sulphide, copper sulphide and lead sulphide were investigated systematically by Schwarz and Romero (1927), who examined the melting properties of various mixtures and produced a phase diagram (fig. 2). Under laboratory conditions, where the sulphur is prevented from burning off, the melting points for the sulphides of silver (Ag_2S), copper (Cu_2S) and lead (PbS) are 835° C, 1121° C and

1114° C respectively, whereas a mixture of these three sulphides in the ratio of 5:7:8 will melt at about 440° C. The niello formula used by Cellini was difficult to work with in the investigations described by Wilson (1948) and Moss (1953). Cellini was, on his own admission, self-taught in the niello technique. It must be presumed therefore that he added too much lead to the mixture in a direct attempt to achieve the lowest possible melting point.

The Danish artefacts from the Iron Age which have been investigated have no obvious air bubbles in the inlay to suggest that the method of application involved the melting on of sulphide powder. In the exhibition "From Viking to Crusader" (Roesdahl 1992, no. 27f, 304), there were, however, four onion-shaped, oriental pendants from the Swedish Vårby Hoard and a tubular neckring from the Russian Gnezdovo Treasure from the 10th century, which all had conspicuous air bubbles in the niello. These artefacts should be analysed, as they either contain melted-on niello of silver/copper sulphide or are the earliest lead-containing niello yet reported.

Of the niello formulae in eight historical sources (La Niece 1983), only a few are without lead and therefore of relevance to the Danish artefacts which have been investigated. The sources in question are a formula from Pliny (Rackham 1968), one from al-Hamdänï from 942 AD (Toll 1968; Allan 1979) and the formulae in chapters 58, 195 and 196 of "Mappae Clavicula" from the 8th-12th centuries AD (Smith & Hawthorne 1974).

In "Mappae Clavicula" (chapter 58) and al-Hamdänï, only silver and sulphur are thought to have been melted together in the production of the sulphide. Because silver sulphide, as already mentioned, decomposes to give metallic silver and sulphur before it reaches its melting point, it must be assumed that the silver used in the formulae was a silver alloy from the workshop, which contained some copper.

Rosenberg (1924) calculated the relationship between the parent elements for the metal sulphides which will be obtained according to a series of niello recipes in the historical sources and was of the opinion that generally too much sulphur was specified.

He knew of an old description of a Russian test piece, where the task was to cold-hammer a niello-inlaid plate out to double its size. Rosenberg thought that this could only be done because the niello contained some unreacted metal among the sulphides, since malleability is a characteristic of metals and not of mixed sulphides. Theophilus (Hawthorne & Smith 1963) states that cold niello shatters like glass. Moss (1953) addressed this problem and did not think that free metal in the sulphides was desirable as the excess metal would be in the form of lead which would disrupt a silver underlay. Furthermore, the excess of sulphur in the historical formulae proves that the intention was to convert the metals completely to sulphides. In an attempt to copy the Russian test piece, Moss tried to cold-roll inlaid niello so the whole surface became enlarged, but the niello cracked and came away from the silver underlay at the touch of a fingernail. On the basis of the experiments described in the following, it should perhaps be assumed that the use of the expression "cold working" with reference to the Russian test piece is due to a misinterpretation of the craftsmen who perhaps just meant that the silver should not be red hot.

Moss succeeded, to varying degrees, in inlaying pulverised silver sulphide on a heated silver piece by rubbing the powder together using a burnisher. He was therefore aware that silver sulphide becomes plastic at a much lower temperature than its melting point. A corresponding description of an attempt to inlay using sulphide powder is to be found in La Niece (1983).

In the discussion of the nature of niello, Newman et al. (1982) present the phase diagram for CU_2S-Ag_2S (fig. 3), from an investigation by Skinner (1966). The phases of the sulphide mixtures can be drawn as a two element diagram, because sulphur comprises a third of a mole in all the minerals. The melting curve (uppermost in the diagram), with a minimum melting point of 640 ± 3° C, is approximately the same as that given earlier by Schwarz and Romero (1927) (fig. 2). An important point with regard to the niello investigations presented here concerns the polymorphic conversions of the minerals in the system explained by Skinner (1966). At room temperature there are eight different stable crystal phases. There is silver sulphide (acanthite), four copper sulphides (chalcocite, covellite, digenite and djurlite), as well as three tertiary compounds (jalpaite, mckinstryite and stromeyerite). All eight crystal phases appear at specific combinations of the

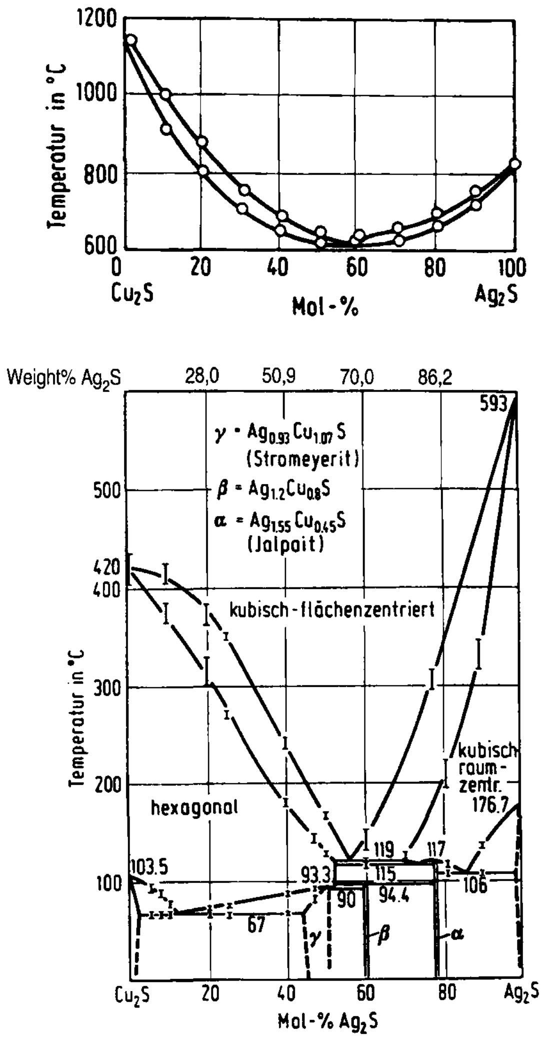

Fig. 3 Phase diagram for Ag_2S-Cu_2S: The upper part shows the melting and setting curves in the system and underneath the phases of the system in a solid state (Gmelins Handbuch 1974). The amount by weight of Ag_2S relative to the molar % has been added to the diagram.

components and the crystal structures which appear are orthorhombic, tetragonal and monoclinic.

In a solid state at higher temperatures, only three minerals occur in the system, with broad variation in composition. They have each their own lattice structure: hexagonal (hcp) at the copper side of the diagram, body-centred cubic (bcc) at the silver side and, in between, face-centred cubic (fcc), which spreads over the whole diagram with increasing temperature. These mineral compounds exist only at elevated temperatures.

In the world of metals, the body-centred cubic structure is quite malleable, while the face-centred cubic is the most malleable.

La Niece (1983) describes a swastika-shaped inlay of silver sulphide on a Roman silver dish from the 3rd century AD, where the ca. 4 mm broad bands of niello resemble cut-off strips. She presumes that this unique occurrence is evidence of an unnecessarily time-consuming application technique.

On the basis of their investigations of artefacts, La Niece (1983) and Schweizer 1993) think that silver-copper sulphide niello, like pure silver sulphide, was not melted in place, because there are no air bubbles in the niello, as there are in the later lead-containing sulphides. La Niece presumes therefore, that an (unspecified) application technique used for pure sulphides in Roman times had been continued despite copper sulphide's somewhat lower melting point.

Moss (1953) examined some bronze Roman belt plates from the 1st century AD, where the niello consisted of copper sulphide. These inlays appear to have been cut into shape before being inlaid, rather than melted into the engraved depressions. He states that "Since the melting point for cuprous sulphate (1112° C) is well above that of bronze, it could only have been inlaid in the bronze in the manner already suggested". As will be apparent from the results of the experiments described later, his interpretation appears to be correct.

Some of Newman et al.'s (1982) niello from Byzantine artefacts contained silver-copper sulphide in the form of jalpaite. When examined at 500-550 times magnification with a scanning electron microscope, the phases acanthite and jalpaite appeared finely distributed among one another. Similar visible phase division could be seen in sulphides on the Danish artefacts and the sulphides produced experimentally as part of this project.

EXPERIMENTS WITH PRODUCTION OF NIELLO AND INLAYING

In order to interpret the niello inlays on the archaeological artefacts, niellos of various compositions were produced. This was done by converting metal

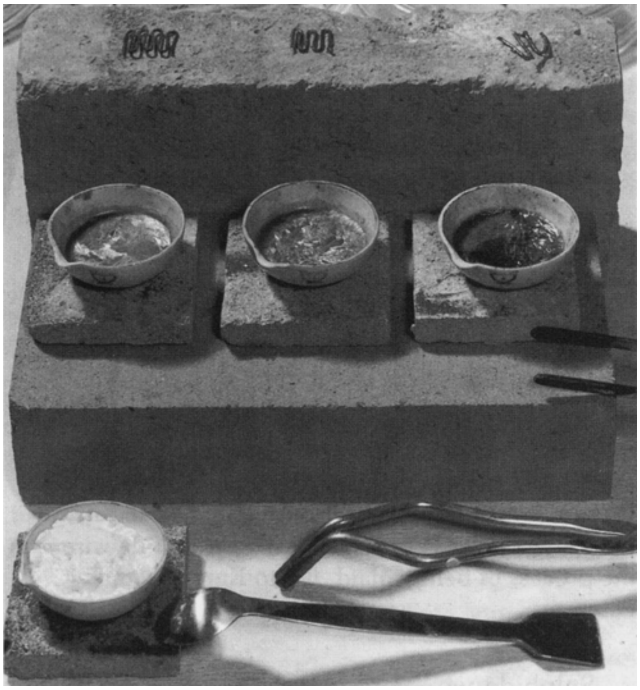

Fig. 4 Crucibles with sulphur used to convert metal wire in an oven. Some of the treated wires can be seen in the background. Photo John Lee.

wire in molten sulphur and, at higher temperatures, by melting together of the components. With sulphides in the form of wire and powder, the various inlaying techniques could then be tried out on furrows cut in sheets of silver, brass, bronze and gold.

Pure silver, pure copper and three silver-copper alloys were the starting point for the experiments. The alloys contained 92.5% (sterling silver), 80% and 70% silver respectively. According to the chemical data (Gmelins Handbuch 1971), silver sulphide can be produced from metallic silver exposed to sulphur vapour or molten sulphur. An accelerated conversion process is achieved above 177.8° C, but above 444° C the conversion of the metal occurs so rapidly that only detached flakes are produced.

Silver and sulphur ions can move freely in silver sulphide covering silver surfaces, so the sulphide does not offer protection against further action by the sulphur (Gmelins Handbuch, 1973). The experiments showed that conversion from silver wire and sterling silver wire to sulphide occurred rapidly, compared to the conversion of metal wire with a higher copper content, where the ions were less mobile in the sulphide coating. In closed bowls containing molten sulphur, 1mm thick pure silver and sterling silver wire was converted to sulphide wire in an hour at an oven temperature of 220° C (fig. 4). Alloy wire containing 80% and 70% silver and pure copper wire, required 350° C for 2 and 4 hours, respectively, for the conversion to be complete. The sulphides arising from the 80% and 70% silver alloy became very soft in the bowls, and the sulphide wires were difficult to detach from the bottom of the bowls and they stuck together in the viscous molten sulphur.

On conversion, the wires increased in volume and at the same time became hollow or developed porosities at the core. The situation is the same as that with metal artefacts which have been totally transformed by corrosion in the soil. The copper sulphide wires produced in the experiment had a hollow core 1mm in diameter, which shows that it was exclusively copper ions which had migrated out of the copper wire in order to react with the sulphur. In the other sulphide wires the hollow or porous core was less marked, which means that the sulphur ions must have moved inwards through the sulphide layer at the same time as metal ions moved out.

Silver-copper sulphide was also produced by fusing together of the components, as among others Moss (1953) has tried using Pliny's formula, at an oven temperature of ca. 1000° C. Casting of the sulphide bars made from sulphur and 70% and 80% silver alloys was done as quickly as possible, but there was, however, a small amount of finely distributed pure metallic silver in the cast sulphide. An excess of sulphur was present in the melting process, but was easily burned off. According to Newman et al. (1982) it is possible, in the laboratory, to fuse together silver sulphide containing only 10-15% copper.

In Skinner's (1966) preliminary work for the construction of the phase diagram, the measurements on silver-copper sulphide were generally uncertain above 450° C, at which point sulphur is released by evaporation and the sulphides begin to decompose. According to the tertiary phase diagram for sulphur, silver and copper at 250° C (fig. 5), pure silver will crystallise out if there is deficiency of sulphur in the mixture. The present experiments showed that the sulphur deficiency can occur during fusion, or if the

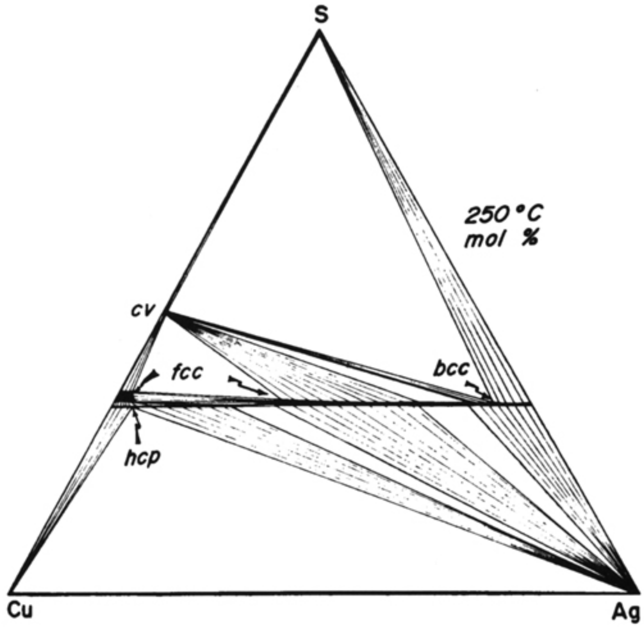

Fig. 5 Phase reactions for Ag-Cu-S at 250° C with the crystal structures marked (Skinner 1966). With a deficit of sulphur (below the horizontal line in the diagram) pure silver dissociates from the sulphide.

sulphur burns off by overheating during forging and inlaying. Overheating experiments in an oven at temperatures of 500° C and 575° C for 5 minutes showed that pure silver sulphide decomposed less readily than niello of silver-copper sulphide. Small crystalline peaks of silver could be seen on the surface of the sulphides.

In order to control the working temperatures precisely during the inlaying experiments, an electric hotplate was used. On the hotplate it was possible to shape the sulphide wires which had been produced and inlay them in furrows milled in sheet metal of sterling silver, and in some cases brass, bronze and gold. This method is illustrated in figure 6 which shows a sulphide wire being inlaid in a 1 mm spiral furrow. For the comparative experiments straight furrows were used. Copper sulphide in the hexagonal (hcp) area of the phase diagram was not malleable (fig. 3), but with further heating it was possible to inlay the sulphide at 450-500°. Copper sulphide is hard and very difficult to inlay such that the furrows easily become mis-shaped. As a consequence, it seems reasonable to conclude that the method was not continued after Roman times.

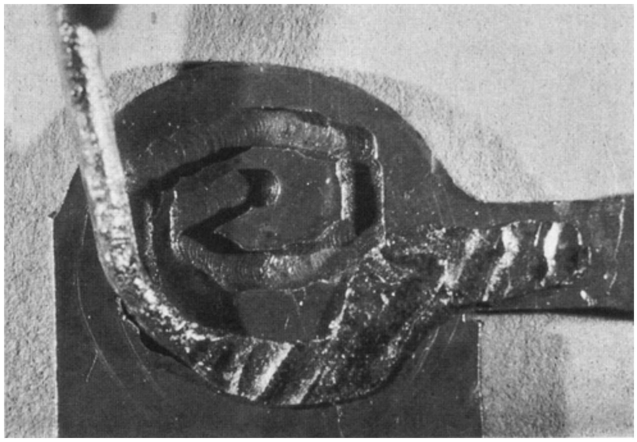

Fig. 6 Close-up photo of a milled spiral 1mm furrow in 14 carat gold sheet, in which a silver sulphide wire is in the process of being inlaid. Photo Karen Stemann Petersen.

Sulphide wires produced from pure or sterling silver could be smithed into the furrows when they were in the body-centred cubic area (bcc), but at higher temperatures they became face-centred cubic (fcc) and thus much more flexible. According to the phase diagram, sulphide with 60 molar percent Ag_2S is already face-centred cubic at 119° C, and experiments at 200° C with niello of this composition (from 70% silver) showed it to be a particularly malleable material.

If the converted wires were only slightly deformed during inlaying then porosity still existed along the core of the wire, i.e. the converted wires had to be slightly larger than the furrows for the inlay to be completely compact.

Cast sulphide bars from alloys with 70% and 80% silver could be hot forged to sheets before inlaying. All the inlaying experiments with sulphide wires and strips were convincingly easy to perform.

It is also possible to carry out inlaying by rubbing together the pulverised sulphides, as has been tried in earlier research into the niello technique, but the process is difficult and the results poor. The sulphides were crushed in a mortar. Silver-copper sulphide and pure copper sulphide were brittle, but pure silver sulphide was very tough and unsuited to pulverisation. Attempts were made to fill out the furrows in sheet sterling silver with abundant powdered silver sulphide and silver-copper sulphide

Fig. 7 Detail of a lance point from Svenskens, Endre parish, Gotland (above). Close-up of spiral-like silver lamella from the transverse decoration of the lance point, magnification ca. 70x (left) (Oldeberg, 1966).

respectively. No binding agent was employed to hold the sulphide powder together, as this would have caused inclusions and the addition of a binder in the form of excess sulphur would have resulted in a blackening of the surrounding silver. The sterling silver, with the powder in the furrows, was heated on the hotplate and the powder was rubbed in using a burnisher. It was, however, necessary to fill up the furrows several times as the powder gradually became compressed. Cross sections showed that the powder had not become completely compacted, and that there were small spaces around the conspicuous powder particles.

Experiments with inlaying of furrows with molten silver-copper sulphide, to which borax had been added, only produced a spongy mass, because the sulphide powder did not melt readily. If the borax was replaced with modern flux intended for silver solder (550-750° C), reasonable results were achieved.

In comparison, crushed and powdered silver-copper-lead niello mixed with borax melted readily into the furrows. The product did however contain small air bubbles, just like those seen in 11th century inlays. As melted-on niello does not, as mentioned earlier, occur in the investigated Danish artefacts from the Iron Age, fluxes have not been subjected to further investigation.

Delicate silver inlays, in the form of small tubes etc., within the niello itself are seen in finds from other countries (Maryon 1972; Oddy et al. 1983; Roesdahl 1992, no. 31). Inlays such as these could be easily copied when the niello inlayed piece was heated whilst the silver pieces were beaten in. It was also possible to twist silver sulphide wire together with fine silver wire in a heated state, producing something which could resemble the starting point for the lamella-like inlays (fig. 7) which Oldeberg (1966) tried to explain on the basis of the then available description of the niello technique.

Microscopic investigation of the inlays produced in the experiments showed that the lead-containing sulphide made for comparison reacted and bound with the surrounding sterling silver, such that microscopic wedges formed down into the metal. There seemed to be a slight reaction between the copper in sterling silver and inlays of pure silver sulphide. The silver sulphide appears to bind closer to the furrows than the silver-copper sulphide.

Microscopic examination of inlays in furrows cut in 14 carat gold revealed a reaction between silver sulphide and gold and the good adhesion was already noticeable during inlaying. At 300-400° C, metallic gold in contact with silver sulphide will alloy with the silver according to the following reaction: $xAg_2S + yAu \rightarrow (Ag_{2x}\ Au_y) + xS$ (Gmelins Handbuch 1971).

The experiments showed, furthermore, that it was impossible to inlay niello containing silver sulphide in bronze (with 10% Sn). Even at a temperature of 200° C, the copper of the bronze rapidly reduced the silver sulphide so that the inlay became bright and shiny. The reaction in a heated state proceeds as follows: $Ag_2S + 2Cu \rightarrow Cu_2S + 2Ag$ (Gmelins Handbuch 1971). If, however, the furrow was in brass (with 35% Zn), the silver sulphide in the inlay was less influenced by the copper in the material because of the presence of zinc. La Niece (1983) does not consider there to be any technical difficulties associated with applying silver-containing niello to copper alloys, despite the fact that Moss (1953) was not able to melt silver-copper-lead sulphide on to copper and bronze. He had more success with brass even though the niello did not flow as well as on silver.

Radiographs of the inlaid sulphide samples showed, as one would expect, that the silver-copper sulphides are more easily penetrated by the x-rays than pure silver sulphide and lead-containing sulphides. Radiographs may be able to reveal toolmarks under the niello if the inlaid objects are not too thick.

NIELLO ANALYSES ON ARTEFACTS AT THE NATIONAL MUSEUM

Most of the National Museum's niello-inlaid artefacts from the Iron Age are of silver or silver-plated bronze and the majority are further decorated with fire gilding. There is, in addition, a cast bronze reliquary cross and a pair of gold rings dating from the transition to the Nordic Middle Ages. On the basis of a visual examination of 67 artefacts, a representative group of 21 was chosen for analysis. Two or more very small particles of niello were removed with a scalpel in the same area of each item. The analytical equipment used was a scanning electron microscope linked to an EDAX x-ray detector (Philips SEM 505 with EDAX 9100). After the first round of analyses it was found that improved results were achieved if the surface of the samples was scraped gently with a scalpel to remove foreign material. During preliminary niello analyses in 1991, three objects from Jelling were analysed by placing them directly in the SEM chamber. The analyses were then carried out directly *in situ* on the niello surface.

Where the analyses revealed small amounts of Mg, Cu, Al and Si as impurities, these have not included in the results table (fig. 8). This is because these traces could be due partly to the presence of soil particles, partly to the cellulose adhesive used to hold the small particles during the analysis, and in the case of Al, partly also to the analysis chamber. The sulphur content in the sulphide minerals present can be calculated from the molar weight of the elements (fig. 9). In general, the calculated sulphur content corresponds to that found in the analyses of the artefacts. However, niello can, as mentioned, contain metallic silver and therefore have a lower sulphur content than expected. This is particularly apparent in the analyses carried out on the surfaces of the strap ornaments from Jelling and the Bonderup pendant cross which also had a bright silvery sheen on the niello-inlaid surfaces.

The oldest niello-inlaid artefacts at the National Museum are three very different silver fibulae, all of which have been imported. The silver fibula from a woman's grave from Røgnehøj, Funen, is from the late Roman Iron Age (mus. no. 59/09). Its simple niello pattern of straight lines and arcs is of silver sulphide. The slightly later silver fibula from a woman's grave from the 3rd century AD from Årslev, Funen (mus. no. 8568) also contains silver sulphide, but the niello is inlaid as more variable broad fields with diverging spirals and a narrow punched border (Storgaard 1990). The undulating margins with spirals must have been produced from very broad bands of silver sulphide. A fibula in gilded silver from the 5th century AD, found at Skjerne, Falster(mus. no. C288), contains silver sulphide, in a uniform repeating pattern of lines plus circles enclosed by interlocking arcs.

On the fibula from Skjerne, it is clear that the fire gilding was applied after the niello, as parts of the niello decoration on the side of the arch was never cleared of gold (fig. 10). Where fire gilding and niello are part of the ornamentation, this sequence is usual, as documented by Oddy et al. (1983). The gold amalgam applied to the object was heated to above the boiling point of mercury, 356.9° C, and the areas to which niello had already been applied were then scraped free of gold. Mercury from the gilding can be seen in some of the niello analyses (Oddy & Meeks 1983), as it can spread over the whole object. On the strap ornaments from Jelling there was, for example, a significant mercury content in and on the cast silver, including the now empty niello furrows.

Weapon accessories from the sacrificial bogs at Nydam, Kragehul and Ejsbøl-Syd, dating from around 450 AD, include large numbers of closely-related objects in fire gilded cast silver with Nordic

Analysed objects	"number of analyses"	S	Ag	Fe	Cu	Au	Pb	others
59/09, fibula, Røgnehøj	1	10.2	88.3	0.9	0.7			
8568, fibula, Ørslev	1	13.1	86.9					
C288, fibula, Skjerne	1	14.8	85.3					
NV448, scabbard mount, Nydam	2	15.6	34.3	12.8	37.4			
NV958, button, Nydam	2	18.5	36.1	16.0	29.3			
NV6, belt buckle, Nydam	3	19.9	27.4	24.4	28.3			
NV1, scabbard mount, Nydam	3	12.0	22.3	4.7	61.0			
NV14, clasp, Nydam	3	16.4	27.6	12.9	36.6			6.5 Zn
12524, fibula, Gummersmark	1	11.6	88.4					
C6, fibula, Bornholm	2	14.1	14.0	0.3	65.4			7.1 Hg
10/40, square strap mount, unknown site	3	9.5	50.6		36.6			1.6 Hg
C325, oval brooch, Holbæk county	3	12.7	64.2		23.0			
CCCLXXII, beaker, Jelling, niello on silver	1	11.3	63.0	6.0	19.7			
same, niello on gilding	1	12.0	68.7	8.6	10.6			
Strap ornament A, Jelling	3	8.5	86.6		1.1		0.4	2.3 Hg
Strap ornament B, Jelling	1	2.8	84.2		3.1		0.5	7.7 Hg
J.nr.7375/92, trefoil sword mount, Trabjerg Bakker	1	13.1	86.9					
16370, penannular brooch, Mølleløkken	1	12.0	88.0					
D97-1982, mount, Lundby Krat	2	12.3	87.7					
14190, Bonderup Cross	1	6.6	32.8		60.6			
D12124, Randers Cross	2	13.2	50.0	0.4	35.6			0.9 Zn
MCMXXX, gold ring, Snoghøj	1	12.0	83.0		0.8	4.2		

Fig. 8 Results of the EDAX analyses of niello from 21 artefacts, given as percent by weight. Where there are several analyses from an object a calculated average is given. Small amounts of Mg, Ca, Al and Si have been omitted from the table.

Minerals	S	Ag	Fe	Cu
acanthite, Ag_2S	12.94	87.06		
jalpaite, $Ag_{1,55}Cu_{0,45}S$	14.07	73.38		12.55
mackinstryite, $Ag_{1,2}Cu_{0,8}S$	15.10	60.96		23.95
stromeyerite, $Cu_{1.07}Ag_{0,93}S$	16.01	50.05		33.94
chalcocite, Cu_2S	20.14			79.86

Fig. 9 For the purposes of comparison with the niello analyses in fig. 8 the sulphur content in the corresponding minerals has been calculated.

animal ornamentation, carved relief and with niello inlay. Five items from Nydam, excavated in 1990-1991 (fig. 11) (Rieck 1994; Vang Petersen 1994), were analysed (NV6, 448, 958, 1, 14). The inlays were of silver-copper sulphide with a substantial iron content which will be dealt with later. Like La Niece (1983), I believe that in this period, when the sulphides appear with a significant copper content, it must be an intentional alloying.

The appearance of the niello pattern on the weapon sacrifices is different from that described earlier, in that there are quite narrow inlaid patterns of punched semi-circular marks and opposing triangular depressions (zig-zag pattern). Niello inlay must have been a particularly popular form of ornamentation with the warriors who were defeated and whose weapons were thrown in to the bogs as sacrificial gifts.

It is possible to follow the similar carved reliefs and animal ornamentation with niello inlay in zigzag patterns etc. through the many fibulae from the 6th century, for example mus. no. C6, which is inlaid with silver-copper sulphide. Many fibulae come from graves on Bornholm and areas close to the Baltic Coast, including, in particular, many Swedish finds from Gotland.

Fig. 10 The fibula from Skjerne (mus. No. C288) with niello also on the side of the arch. Under production, partly gold-covered niello has been left after the fire gilding. The length of the fibula is 15 cm. Photo Svend Erik Andersen.

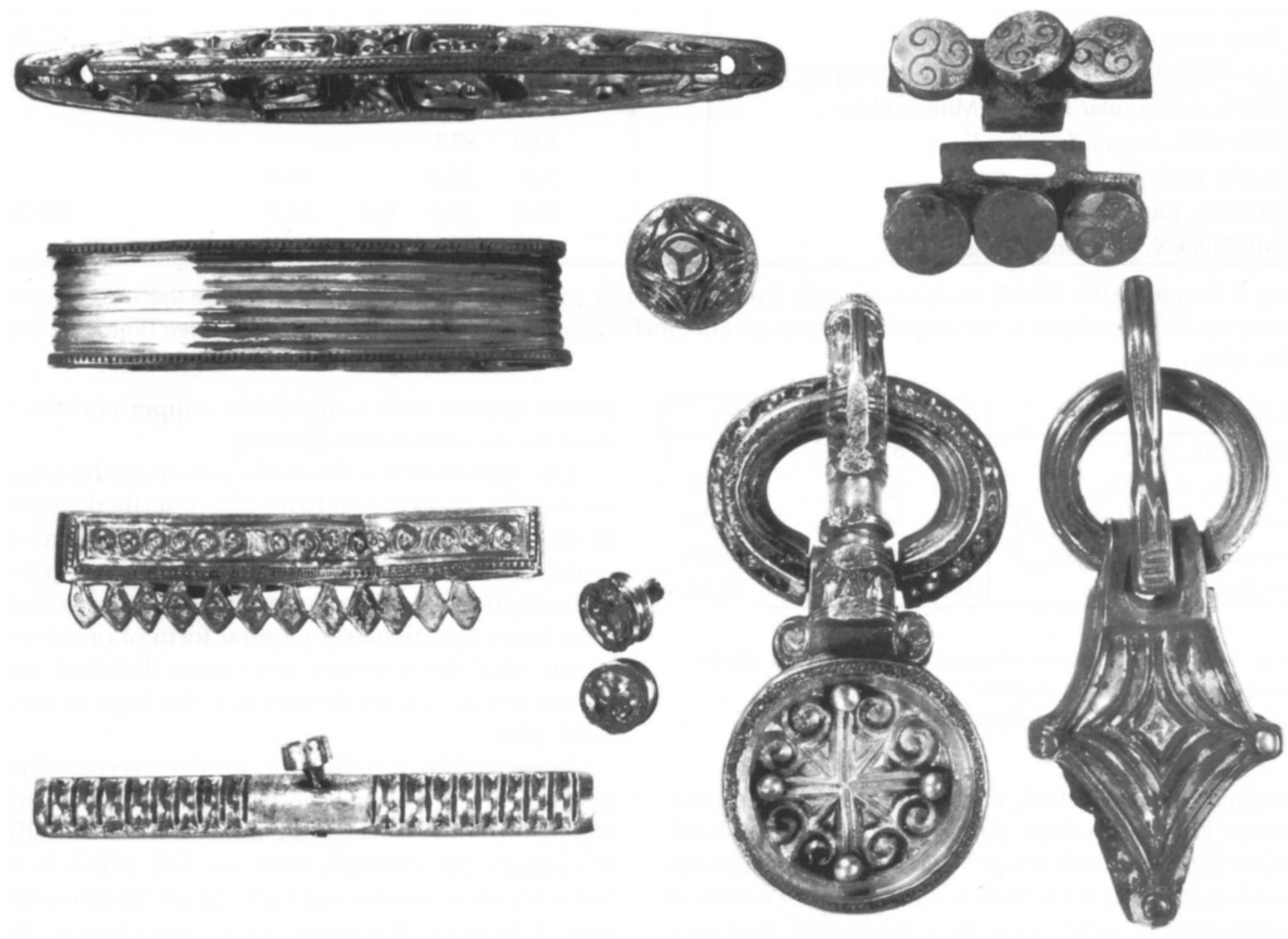

Fig. 11 From a number of different niello-inlaid weapon mounts and ornaments excavated in Nydam Mose in 1990-91, five of the above shown niello inlays have been analysed. Photo Kit Weiss.

Fig. 12 The trefoil sword mount (J.no. 7375/92) from Trabjerg Bakker, Jutland (breadth 5.9 cm). The piece had only been partially cleaned with a scalpel, so the corrosion layer could be investigated before the conservation work was completed. Photo John Lee.

La Niece (1983) found silver-copper sulphide niello on seven out of eight Swedish artefacts from this period, including a scabbard mount and a fibula from Sjörup, as well as three fibulae from Gillberga, Häste and an unknown site. The Swedish artefacts can thus, both in form and niello composition, be compared directly with the Danish finds. An exception to this, where the niello is in the form of pure silver sulphide, is however the fibula from Gummersmark (mus. no. 12524) with some special traces in the inlay which will be discussed later.

From the 7th century, there are some square bronze cross-strap mounts, where a thin covering of sheet silver forms the underlay for niello decorations with zig-zag patterns. The analysed mount (mus. no. 10/40) contains niello of silver-copper sulphide.

The artefact groups from around the 5th to the 7th centuries are related in form and have narrow bands of punched inlays. On the basis of the above mentioned analyses it appears likely that silver-copper sulphide is the most utilised niello type in this period, and the experiments proved this mixed sulphide to be exceptionally pliable and thus suitable for detailed inlays.

Among the Viking Age finds at the National Museum, there are many different silver artefacts inlaid with niello. Analysis of some of these inlays showed, in addition to silver-copper sulphide, also several inlays of pure or almost pure silver sulphide. The artefacts concerned are a trefoil sword mount from Trabjerg Bakker (J.no. 7375/92) (fig. 12), two strap ornaments from Jelling Church, the large 10th century penannular brooch (mus. no. 16370) and the crumpled sheet metal mount from Lundby Krat (mus. no. D97-1982). The silver sulphide inlays on these objects appear in very regular, relatively plain bands.

The Jelling beaker (mus. no. CCCLXXII) has rather less regular engraved lines and the inlay is of silver-copper sulphide. Niello analyses were, as mentioned earlier, carried out directly on the surfaces of the beaker and the strap ornaments in 1991, and the niello analyses from the beaker showed, in addition to silver-copper sulphide, the presence of 6% and 8.7% iron.

The beaker has, in addition to the niello inlays in the silver itself, also niello inlays in shallow depressions on bands of gilding. The smooth delimited lines of sulphide do not appear to be the result

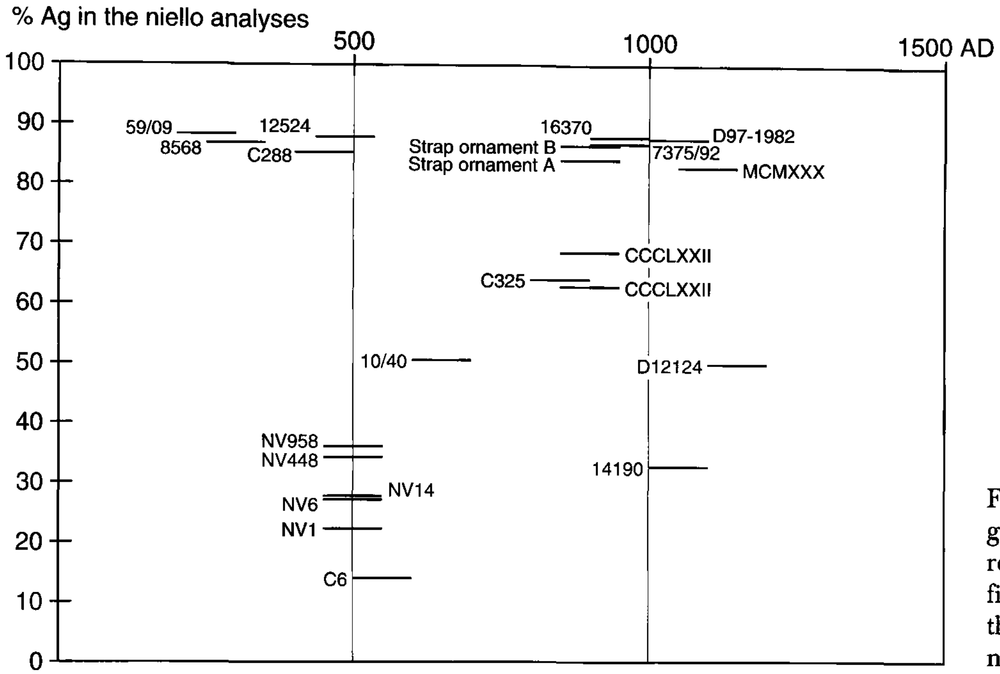

Fig. 13 A chronological graphical presentation of the results of the analyses from fig. 8 arranged according to the silver content in the niello.

of corrosion. The fact that niello can be applied over gilding seems not to have been described previously, but contributes to proving that niello was applied below a temperature, which could cause the already applied gilded areas to dissolve into the underlying silver. The beaker will be deskribed in: Krogh & Leth-Larsen, Vikingekongernes monumenter i Jelling, vol. II.

A very simple pattern of lines in niello on a silver-plated bronze oval brooch (mus. no. C325) consists of silver-copper sulphide. On the silver-plated bronze oval brooches and mounts for harness bows from Mammen (mus. no. C1063) (where the niello has not been analysed), the furrows in the silver are, in several places, cut so deeply that the niello is in direct contact with the underlying bronze.

The niello on the cast silver pendant cross from Bonderup (mus. no. 14190) is engraved with designs in the Ringerike style with the inlay consisting of silver-copper sulphide.

The 12th century Randers Reliquary cross (D12124) is of cast brass, inlaid with silver-copper sulphide niello, just like the Irish St. Cuillean's Bell in iron and brass, dating from the end of the 11th century, which was investigated by Moss (1953) (Roesdahl, 1992 no. 430). The ornamentation of the Randers Cross has been compared with British and Irish artefacts (Lindahl 1990). The choice of brass rather than bronze as an underlay for the sulphides must, as already mentioned, be because the zinc content of the brass prevents a reaction between the silver sulphide and metallic copper.

The National Museum has faceted finger rings in gold, silver and gilded copper, dating from the transition between the Viking Age and the Medieval Period, which bear inscriptions filled out with niello. In the gold ring from Snoghøj (mus. no. MCMXXX), which dates from the end of the 12th century, pure silver sulphide has been inlaid in the engraved letters. A detected gold content in the niello must come from a reaction with the underlay. According to La Niece's (1983) comprehensive list of analyses, gold only appears in niello inlays on gold artefacts, and up until the 13th century, these inlays consist solely of silver sulphide. The ring from Snoghøj is thus consistent with this pattern of events. In "Mappae Clavicula", chapters 195 and 196 (Smith & Hawthorne 1974) a distinction is made between whether the underlay is of gold or of silver. With the former, use of the most silver-rich silver-copper sulphide is recommended.

The graphical presentation of the results of the analyses (fig. 13) has been modified with respect to that of Schweizer (1993) (fig. 1) in that it was possible here to plot the silver content on the y-axis. In this way it is made clear that the group containing pure or almost pure silver sulphides stands apart from the copper-containing sulphides. There is a noteable shift in the sulphides used though time, which is in agreement with earlier analyses.

The niello analysed from Nydam Mose showed a significant iron content within the silver-copper sulphide (fig. 8). In an EDAX analysis technique, revealing the distribution of individual elements, it could be seen that the iron is located in the most copper-rich phase. With the present state of our knowledge of the corrosion of sulphides, it cannot be determined whether the iron could come from the manufacturing process, or from the local concentration of corroded iron in the burial of Nydam Mose. Although, according to the phase diagram, fig. 5, there should be no free copper in the niello inlay with which the iron in the earth could bind.

Arne Jouttijärvi has carried out a niello analysis on an animal-formed brooch from Lindholm Høje on behalf of Aalborg Historiske Museum. The silver-copper sulphide contained 2.9% iron, which with point analysis with EDAX was also found to be bound to the most copper-rich phase of the niello.

Schweizer (1993; Lazovic 1977) has previously demonstrated iron contents of up to 4.7% on the surfaces of Byzantine silver sulphide niello, and Newman et al. (1982), using emission spectrography, found traces of iron on Byzantine niello of both silver sulphide and silver-copper sulphide.

THE MICRO-TOPOGRAPHY OF THE INLAYS

As niello inlays on artefacts found in the soil can be confused with corrosion in the form of sulphides, it is useful to have some diagnostic micro-topographical characters. On bronze artefacts, silver coating is, for example, used as an underlay for the niello decoration, because, as mentioned earlier, it is not possible to apply silver-containing sulphides directly to bronze.

The mount from Lundby Krat (mus. no. D97-1982) is forged and only ca. 1mm thick, in contrast to the majority of objects containing niello, which are cast and are considerably thicker, and thus would have been able to retain heat for some time during application of the sulphide.

As a rule, the inlaid areas occur on raised or flat surfaces, so later smoothing out or scraping of the inlaid material was possible.

Oldeberg (1966) observed niello which occurred above the level of the copper alloy in which it was inlaid and it could be, as he presumes it to be, a result of corrosion of the surrounding metal. Slight differences in level were seen on some Danish artefacts, perhaps because the niello had not been scraped completely flush with the surrounding metal. Smith & Hawthorn (1974) state that in European niello work the decoration lies flush with the rest of the surface, whereas they noticed that in some Sassanian and Byzantine pieces, the niello stood in relatively high relief.

There have been several descriptions of the furrows in the metal underlay. Smith & Hawthorne (1974) saw inlays that were prepared by coarse chisel work to ensure good adhesion in the furrows. La Niece (1983) describes punchmarks which probably correspond to the punched depressions referred to in this investigation. Silicone casts of the tool marks on the strap ornaments from Jelling revealed that a graver with a flat edge had cut the regular notches (Leth-Larsen & Krogh in print). Several other artefacts examined have similar furrows with a rectangular cross-section and notches. Both punched patterns and engraved notches produce uneven surfaces in the furrows, which help, to a certain extent, to anchor the niello. Despite this, there are also niello inlays in furrows with, for example, v-shaped cross-sections cut with a pointed graver and with furrows which are unbroken and quite smooth.

There are several similarities between the encrustation technique and niello inlay. For example, on the silver penannular brooch (mus. no. 16370), three spheres have niello inlay over almost all their surface. These correspond to the closely spaced spiral furrows for inlaying metal wire described by Holmqvist & Arrhenius (1964). According to them, encrustation was a technique commonly-used by Nordic craftsmen from the 5th-12th centuries AD, but apparently with declining popularity in the 6th to 9th centuries. Because of the widespread use of niello in punched patterns around the 6th-7th centuries, it seems likely, that the craftsmen continued using the same inlaying technique, but with niello instead of metal wire. A relationship between the two techniques is also gathered from studying Allan's (1977)

Fig. 14 The fibula (mus. no. 12524) from Gummersmark, Zealand (length 15.5 cm). On the close-up the arrows show that in every inlaid triangular depression there is a cavity in the niello. As the niello is shiny, this appears lighter on the picture. Photo John Lee.

investigation of early Islamic craft traditions. He describes how silversmiths in 11th century had to abandon niello inlay on silver objects, as silver became scarce and expensive, in favour of encrustation work on brass objects.

Experience gained from converting silver wire to sulphide in molten sulphur, suggests that this manufacturing technique could be the reason why on some artefacts there are porous areas running longitudinally in the centre of the inlay. Likewise there is some special repeated flaws on the inlays on the small punched zig-zag patterns on the fibula from Gummersmark (mus. no. 12524). There is a distinctive cavity in the centre of every triangular inlay, fig. 14. In contrast to the other analysed niello inlays with zig-zag patterns of silver-copper sulphide, the niello here consisted of silver sulphide. The repeated cavity might be the result of inlaying a hollow wire, but could certainly not have been produced by rubbing silver sulphide powder into the depressions or by a melting process.

Medieval lead-containing niello appears rather more blue-black than earlier niello of silver sulphide and silver-copper sulphide. This could be the reason that the early types are often found together with fire gilding, so that the colour contrast was accentuated.

BREAKDOWN OF INLAID NIELLO

The sulphides are thought to be stable and are not altered in the soil, but metallic silver, which can occur in the niello matrix will be subject to corrosion in the same way as, for example, the silver object in which the niello is inlaid. The presence of metallic silver on and in the niello on some of the artefacts means that the inlays are not dark. The circumstances regarding niello destruction have been described in several publications.

According to Oddy et al. (1983), care has to be taken in interpreting analyses carried out directly on niello surfaces. The inlay on the large drinking horn from Sutton Hoo consisted solely of silver chloride at the surface, whereas inside it consisted of silver sulphide. Oddy & Meeks (1983) consider it probable that the growth of silver chloride crystals on the surface arises from corrosion of the surrounding silver. In its raw (i.e. uncleaned) state the trefoil sword mount (J.no. 7375/92), fig. 12, had no free silver in the sulphide which appears to be quite intact. The surface covering of silver chloride must therefore stem from the surrounding cast silver.

La Niece lists the reasons why metallic silver occurs in niello as follows: not all the silver has been converted to sulphide during production; silver sulphide is unstable at the elevated temperatures which can arise during inlaying; a possible chemical reduction occurs during certain conservation treatments of the artefacts. With reference to the latter, the strap ornaments from Jelling and the Bonderup Cross (mus. no. 14190), among others, have silver coatings over the niello inlays, which it is hard to imagine were there when the decorations were new. In 1979, the strap ornaments from Jelling were treated by electrolysis in an EDTA solution to remove heavy green corrosion. The metallic silver uppermost in the niello could be a result of this method of cleaning. Half of the niello particle investigated by Jouttijärvi (1995) from the animal- shaped brooch from Lindholm Høje, was of metallic silver, whilst the rest was of silver-copper sulphide with the abovementioned iron content. It must be presumed that it was the uppermost half which was of silver, as previous surface analyses of the inlays have only revealed silver. Most of the niello investigated during this project was however well preserved, including the newly-conserved artefacts from Nydam Mose. The situation regarding corrosion and conservation should be investigated further in connection with new finds.

CONCLUSION

Studies of the common features of the artefacts and the tool marks seen on them, support the theory proposed here with regard to inlaying niello in the Iron Age. Through a discussion of the characteristics of the sulphides and the results of the experiments, it has been demonstrated that Iron Age niello inlaying was in all probability carried out using heated sulphides in strip form. On the raised edge surfaces, for example between carved ornamental reliefs, it would have been impossible to apply sulphides in the form of powder or in a molten state. Furthermore, silver sulphide is quite unsuitable for crushing to powder.

Silver sulphide is easy to produce from silver, which in solid form is exposed to sulphur vapour or molten sulphur. Because silver-copper sulphide is more difficult to make by this method, it apparently was produced by fusion, as described by Pliny (Rackham 1968). This suggests therefore that the changes which the analyses have revealed in niello inlays through time reflect corresponding and significant shifts in production techniques.

The fact that pure silver sulphide decomposes less readily at high temperatures than niello containing copper, perhaps explains why quite pure silver sulphide was the preferred material for early Iron Age inlay, where the work was carried out at high temperatures. From the experiments it appeared that silver sulphide binds closer to the furrows than the silver-copper sulphides. This relationship, as well as the fact that silver sulphide does not decompose so readily at high temperatures and is quite easy to produce, can be the reason why pure silver sulphide was preferred during a substantial part of the Iron Age.

From the middle of the 5th century onwards a significant copper content is found in the niello. The silver-copper sulphide becomes very pliable, and thus suitable for use in detailed inlays, at relatively low temperatures where there is little danger of decomposition.

Whilst bronze is unsuitable as an underlay for silver-containing niello and has to have a silver coating applied before it can be ornamented, brass can be inlayed directly. With the knowledge we have amassed so far regarding the good adhesion of silver sulphides on gold, it would be interesting in the future to conduct a search among Viking Age finds for technical parallels to the Jelling beaker with its niello inlay over gilt silver. It will also require further analyses of Viking Age niello before we are able, in collaboration with archaeologists, to elucidate the parallel use of silver sulphide and silver-copper sulphide during this period.

An explanation for the substantial iron content in the niello from Nydam Mose could possibly be found by carrying out future analyses of the newly found weapon fittings from the bog and other well documented niello finds.

The Iron Age techniques which have been outlined here, can also be of interest to present-day craftsmen, in the light of reservations concerning the health risks associated with the more well-known lead-containing niello.

SUMMARY

In previous research much attention has been focused on the nature of niello and the medieval easily-meltable lead-containing sulphides have been thoroughly investigated. The technological investigations described here were inspired by observations made on archaeological artefacts and by studies of the literature on the chemistry of sulphides. Silver sulphide can be produced by conversion of solid silver in molten sulphur, but this sulphide will decompose in air before it melts. Even though it is possible to produce silver-copper sulphide in the same manner, this form of niello was probably manufactured by fusion of the components. The changes through time which the niello analyses have revealed seem therefore to reflect a corresponding shift in production techniques. With the practical use of the phase diagram Ag_2S - Cu_2S it can be demonstrated that the encrustation technique, which was the technique used in the Iron Age for decorative inlaying of metal wire, could also have been applied to the inlaying of both silver sulphide and silver-copper sulphide in a heated state.

The sulphides bind or react differently with the surfaces of different metals, which explains the choice of materials for niello-inlaid artefacts.

Analyses of Danish niello-inlaid artefacts from the Iron Age revealed unanimously the same changes in sulphide composition through time, as demonstrated in analyses from other countries. The artefacts with niello inlay found at Danish sites dating from the end of the 5th century until the 7th century are related in their Nordic ornamentation and many are thought to be inlaid with silver-copper sulphide. There is a substantial iron content in the sulphides from Nydam Mose, which might be a result of a corrosion phenomenon.

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