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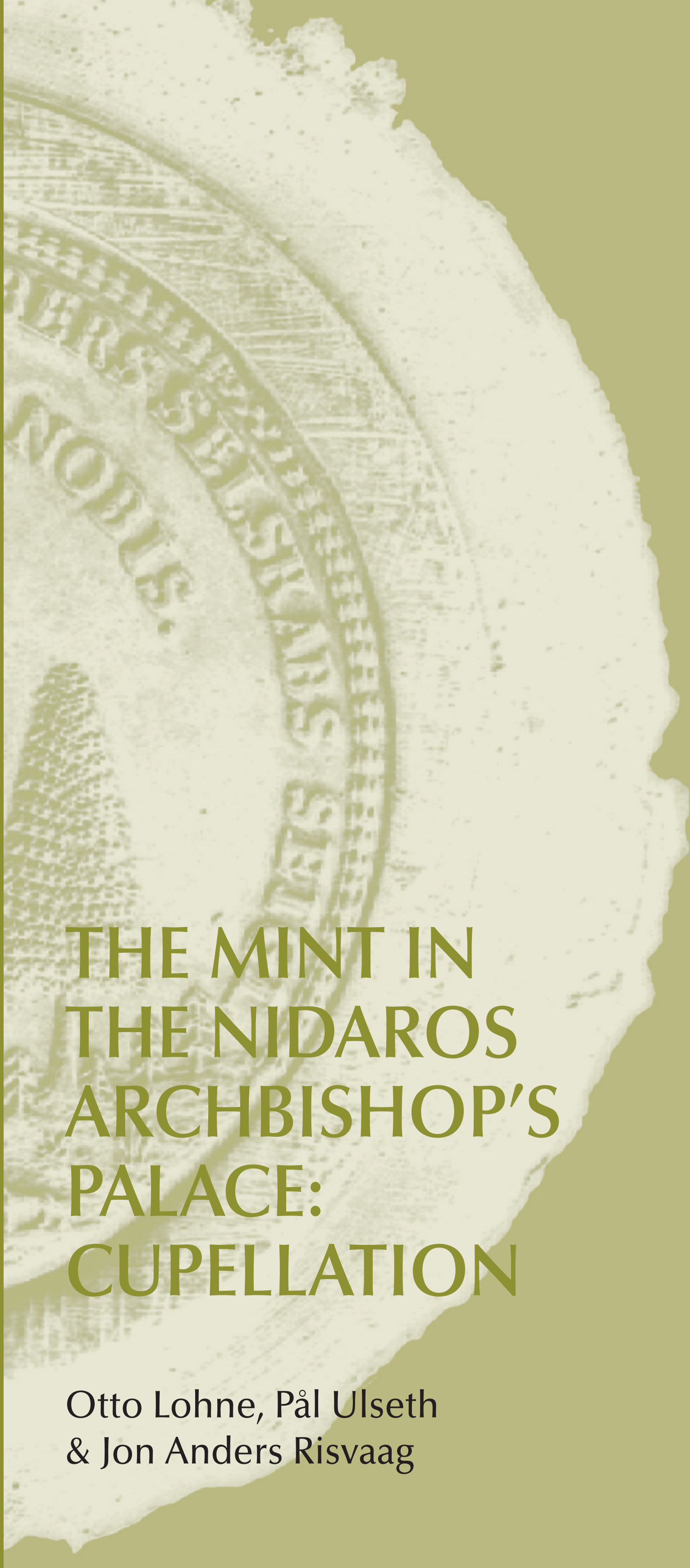

THE MINT IN THE NIDAROS ARCHBISHOP'S PALACE: CUPELLATION

Otto Lohne, Pål Ulseth
& Jon Anders Risvaag

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PREFACE

The Archbishop's Palace in Trondheim (Nidaros) was the centre of the largest diocese in Western Christendom. Established in 1153, the diocese encompassed Norway, Iceland, the Faeroe Islands, Greenland, Isle of Man, the Orkneys, and Hebrides. The oldest parts of the Archbishop's Palace date back to the twelfth century. Different parts of the palace have been developed and rebuilt in different stages to fit different purposes throughout the centuries right up to the present day.

In 1222 the archbishops were granted coin privileges by King Håkon Håkonsson (1217–63), which subsequently was revoked by the Regency government in the minority of King Eirik Magnusson (1281–99). The coin privileges were restored by the Danish-Norwegian King Hans (1483–1513) in 1483, and the archbishops' coin production continued seemingly uninterrupted until the Reformation in 1537.

A devastating fire on 18 August 1983 destroyed the southern and eastern wings of the Archbishop's Palace, consisting of eighteenth-century military magazine/storage buildings. Following this tragic event extensive archaeological excavations led by Sæbjørg Walaker Nordeide (1956–2020) took place in the years 1991–95. The excavations revealed the remains of three consecutive late medieval mint complexes within the palace structures. The oldest of which is at present exposed *in situ* in the museum constructed on the excavation site.

A vast array of objects originates from these mints, including bone-ash cupels, crucibles (fragments) and flans. Based on these finds and known coins, and with a generous grant from The Royal Norwegian Society of Science and Letters (Det Kongelige Norske Videnskabers Selskab – DKNVS), Otto Lohne, Jon Anders Risvaag, Pål Ulseth and

Jardar Lohne were able to conduct an interdisciplinary study of the coin production of Archbishop Gaute Ivarsson (1475–1510) as part of the project *Erkebiskopenes utmynting på 1500-tallet* (launched in 2003), which resulted in the publication *The Mint in the Nidaros Archbishop's Palace. Coin Production under Archbishop Gaute Ivarsson (1474–1510)* (DKNVS Skrifter 2010:1). The publication encompassed historical, archaeological, numismatic, and metallurgical analyses of the archbishop's coin production, aiming for a broader understanding of its political and economic role. The results from the metallurgical analysis underlined both the importance and the advanced technology of the mint, but also its limitations.

In the continuation of the project, the research group focused more specifically on technical sides of the archbishops' coin production, particularly the process of assaying, also referred to as cupellation. In these studies, used bone-ash cupels and fragments from the excavations have been closely investigated and new cupels have been produced to examine the assaying process itself. Several international studies have previously been conducted and are referred to in the individual articles.

This volume presents three articles resulting from the investigations of the excavated bone-ash cupels and the experimental processes: 1) 'A catalogue of bone-ash cupels dating from AD 1500–1537' which presents a morphological study of all identified bone-ash cupels/fragments from the archaeological excavations of the Archbishop's Palace. 2) 'From calves' sculls to finished bone-ash cupels and testing of precious metals' which presents experiments involving the whole production chain of bone-ash cupels and their use in the assaying process, and lastly 3) 'A metallurgical investigation of the formation of silver beads during cupellation of a 925 ‰ silver alloy' which presents a metallurgical analysis of the formation of silver beads during the cupellation process. A model based on the metallurgical processes taking place during heating, annealing, and cooling is presented.

The papers represent an interdisciplinary combination of archaeology, numismatic and metallurgical expertise. Access to world class scientific instruments and skilled operators has been a great advantage when investigating the various metallurgical processes taking place during cupellation.

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Trondheim
23 August 2021

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A CATALOGUE OF BONE-ASH CUPELS DATING FROM AD 1500–1537

found in the Mint in the Archbishop's Palace, Trondheim, during archaeological excavations in 1991–1995

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Abstract

During the archaeological excavations in the Archbishop's Palace in Trondheim in 1991–1995, three successive mints dating from about AD 1500–1537 were uncovered. A high number of bone-ash cupels used for assaying precious metals were found. The assemblage, which is now stored in the NTNU University Museum, Trondheim, represents one of the largest collections of late medieval bone-ash cupels found in a European excavation to date. The catalogue comprises a brief presentation of the use of bone-ash cupels in medieval times and individual descriptions of 186 uncovered cupels and fragments of cupels. The cupels were made in moulds but no such moulds have been found. The upper, outer rim diameter of each cupel has been measured and the results show that the cupels in the assemblage may be grouped into five size classes: 25, 30, 35, 40, and 45 mm. The majority have a rim diameter of 35 mm (54%).

Introduction

During the archaeological excavations of the Archbishop's Palace in 1991–1995, three successive mint complexes, one above the other, were uncovered (McLees, 1994; Saunders, 2001; Nordeide, 2003; Lohne et al., 2010). The mints date from the period of the last three archbishops, from about AD 1500–1537. Among metalworking debris there was a high number of used bone-ash cupels. Together in the archaeological excavation records, 174 cupels or fragments of cupels of various sizes have earlier been reported (Saunders, 2001, p. 27). In the catalogue presented in this paper the number of cupels is increased to 186 cupels, due to identification of cupels previously recorded as crucibles. Both the cupels and fragments of cupels are systematically described, albeit not in very much detail. The main purpose of the catalogue is to give a qualitative overview of the size, shape and condition of each cupel or fragment.

Background

Several treatises from the 16th century describe the manufacture of cupels and the cupellation process, including those by Biringuccio (1966 [1540]), Agricola (1950 [1556]), and Ercker (1951 [1574]). According to the aforementioned authors, good cupels were made using ground and washed ash that is moistened with a binder, such as strong beer, packed into a mould, and beaten with a mallet to make a shallow bowl on top of the compacted body. The top surface was then faced with a finely ground bone-ash, which was beaten onto the surface, so it adhered evenly. Bone-ash from calves' heads was recommended as the best material for the purpose. However, the authors also informed that in the 16th century the materials selected, and assayers' practices varied.

Many publications deal with archaeologically excavated cupels from various places dating from the 16th century and earlier, including Oberstockstall, Austria (c.100 cupels) and Castle of Pymont, France (c.20 cupels) (Martinón-Torres et al., 2009), Legge's Mount, the Tower of London (14 cupels) (White, 2010), and Porto, Portugal (171 cupels) (Hsu and Martinón-Torres, 2019). The main investigations described in the aforementioned publications concerned the material used in the manufacture of the cupels and their microstructure. Except for four cupels (White, 2010), all investigations were of used cupels. Although only a small fraction of the excavated cupels have been investigated, the main material used is reported as bone-ash, mainly pure bone-ash or in some cases mixed in with some other material, accounting for up to 10% in cupels from Pymont and 30% in cupels from Oberstockstall. In cupels from Porto, wood ash might have been mixed in with the bone-ash.

The use of bone-ash cupels

In medieval times, bone-ash cupels were the main equipment used when assaying precious alloys, such as measuring the silver content of coins. However, the process of cupellation can be traced back as far as the Early Bronze Age (Kohlmeyer, 1994). The four main steps in the cupellation process are schematically shown in Fig. 1 (for details, see Lohne et al., 2021, this volume; Lohne and Ulseth, 2021, this volume).

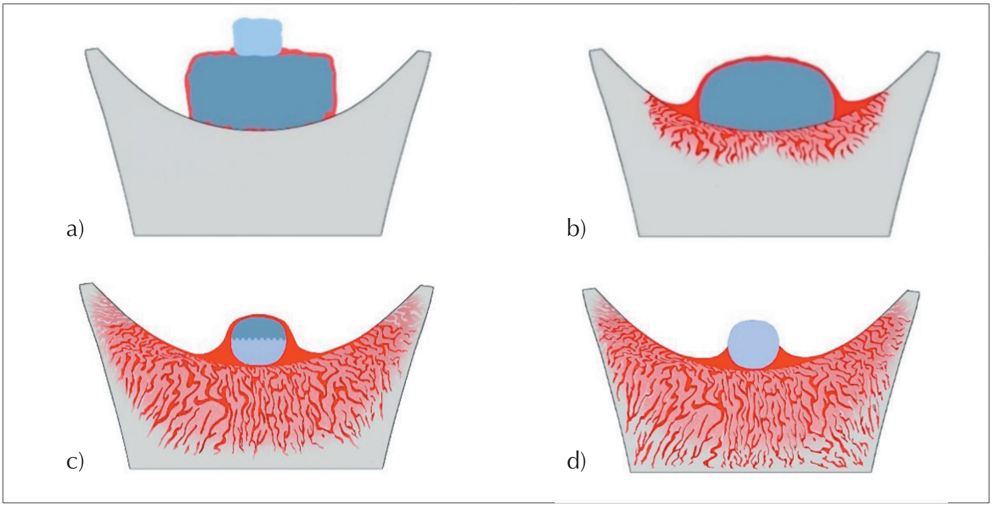

Figure 1. a) A small piece of metal of unknown composition (pale blue) is placed on a pre-heated bone-ash cupel (grey) together with about ten times as much lead (dark blue) and then the cupel is placed in a hot furnace. On the lead surface, solid lead oxide is shown as red; b) During heating, lead melts and the metal of unknown composition is dissolved in the melt. At a temperature just below 900°C lead oxide melts, dissolves impurities (not silver) and is soaked into the cupel by capillary action. Some lead oxide evaporates. c) More and more lead transforms into lead oxide by reacting with oxygen above the melting lead button and is soaked into the cupel. Consequently, the concentration of silver in the molten pool increases. When the concentration reaches c.90%, solid silver starts to precipitate in a dendritic shape. Dendrites grow, impinge on other dendrites, and are transformed into grains (pale blue). d) All lead has oxidized, and the silver has solidified into a bead of almost pure silver. When all the lead oxide has been soaked into the cupel, the process is complete. After the cupel and its contents have cooled, the bead is removed. The ratio between the weight of the bead and the original metal of unknown composition gives the fraction of precious metal (assuming that the bead is pure silver, that no silver has been soaked into the cupel, that the bead has been cleaned and/or brushed properly, and that the lead did not contain any silver at the start of the process).

Results and discussion

Some typical bone-ash cupels from the excavations in the Archbishop's Palace are shown in Figs. 2–4. It is clear that the cupels were made in moulds. The diameter of the upper outer rim of each cupel is listed in Table 1. The results of examinations of the assemblage revealed that the cupels may be grouped into five size classes according to their rim diameters: 25 mm, 30 mm, 35 mm, 40 mm, and 45 mm. The upper rim diameter in each class may vary ± 1 mm. The majority of cupels had a rim diameter of 35 mm (54%). Although there must have been moulds for making cupels in least five different sizes, no such moulds were found during the excavations.

Many of the cupels that lack part of their base are ball shaped. A total of 154 (83%) ball-shaped cupels were found and measured. For all cupels with a base or part of their base surviving, the heights of the cupels were measured too.

Figure 2. An original silver bead seen at the bottom of cupel N124867. Diameter of bead: 0.7 mm. Photograph: Åge Hojem, NTNU University Museum.

Figure 3. Four bone-ash cupels viewed from two angels. From left to right, they have an outer rim diameter of 30 mm (N125123), 35 mm (N122514), 40 mm (N124858), and 45 mm (N124857), respectively. Note the depression at the bottom of the largest cupels, N124857 and N124858. Photograph: Åge Hojem, NTNU University Museum.

A fine-grained surface layer, called facing, has been registered. With the exception of one or two cupels, all cupels have a fine-grained facing layer, which made it easier for all silver to be collected in one bead through cupellation. Erker (1951 (1574) p. 31) underlines the importance to have a good cupel facing. At flaws in the surface layer silver may be lost (Ulseth et al., 2015).

Some cupels with an upper rim diameter of 40 mm or 45 mm have a depressed area at the bottom – 15 out of 27 (56%) and 8 out of 17 (47%), respectively. Such depressions have been seen also on cupels from Pymont (Martinón-Torres et al., 2009). The depressed area might have facilitated the process of collecting all silver into one bead. One question that arose during this compilation of the catalogue, was whether there might be an identifiable chronological development from cupels without a depression to cupels with a depression. However, archaeological data do not support this theory: of the 22 cupels with a rim diameter of 40 mm and 14 cupels with a rim diameter of 45 mm, which could be placed within a time frame corresponding to their use, cupels either with or without a depression were equally distributed. In total, 61% (22 of 36 cupels), with an almost 50:50 division based on the presence or absence of a depression or not, were found in a levelling layer relating to a fire of 1532, which devastated the eastern and southern parts of Archbishop's Palace, including the second workshop. The fire was caused by the troops of the Protestant king, Frederik I (1523-1533), who attacked the palace in retribution for the archbishop's support to the deposed Catholic king, Christian II (1513-1523), who tried to reclaim the throne in 1532.

Figure 4. Cross-section of cupel N124860. Upper rim diameter 35 mm, height 23 mm. Note the fine-grained surface layer (i.e. the facing) at the bottom of the bowl and the two dark areas at the bottom of the cupel where lead oxide has filled both cavities between the bone-ash particles and the canals in the bone-ash, in contrast to just in the canals in the bone-ash particles higher up. Photograph: Åge Hojem, NTNU University Museum.

Bone-ash cupels are brittle. However, in the parts of used cupels that are 'impregnated' with molten lead oxide during the cupellation process, the bone-ash particles are strengthened and 'glued' together. As a consequence, these parts might survive handling during excavations, whereas excavated cupels might lack parts that were not impregnated (Fig. 5) and might have a ball-shaped bottom and lack parts of their upper rim. Bone-ash in cupels that have not been impregnated by molten lead oxide might have been reused by assayers (Lohne et al., 2021, this volume).

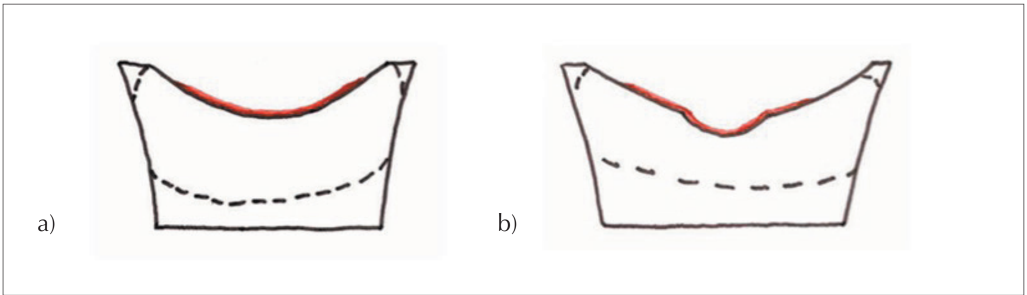

Figure 5. Sketches of two cupels with an upper rim diameter of 35 mm (left) and 40 mm (right), the latter with a depression at the bottom. The dashed lines indicate where many cupels are broken (the majority of them ball-shaped cupels). Red lines illustrate the facing. (Reproduced from Ulseth et al., 2015, p. 270)

Two cupels were cut in two halves and then metallographic specimens were prepared and investigated using a scanning electron microscope (SEM) equipped with energy dispersive x-ray spectrometer (EDS), for chemical analyses. In addition to lead oxide, the dominant elements were calcium (Ca) and phosphorous (P), with low concentrations of magnesium, aluminium and silicon. The results show that the cupels were made mainly from bone-ash, without the addition of clay. The precence of canals and analysis of the ash show that the ashes were derived from animals (Ulseth et al., 2015).

Earlier, we conducted laboratory experiments on making bone-ash cupels, ranging from calves' sculls to finished cupels with facing, and we tested a silver and a gold alloy with known purity with good results (Lohne et al., 2021, this volume)[1]. From the experiments, we concluded that it is most likely that the cupels excavated from the Archbishop's Palace were produced on the Mint's premises.

[1] See also: Lohne, O. and Ulseth, P., 2016. *Framstilling av beinaskekupeller fra grunnen av og erfaringer med kupellering*. Notat. 15 pages in Norwegian.

Table descriptions and comments

Systematic descriptions of the 186 bone-ash cupels are listed in Table 1. The descriptions are not very detailed. Rather, the catalogue is intended to provide a qualitative overview of the size, shape and condition of the cupels. The weight of the cupels indicates that all of them were used. The cupels have each a specific museum number, prefixed with the letter N (e.g. N115972).

Similar sample presentations are missing at an international level. By presenting the catalogue, we aim to contribute results that might be used when researchers compare manufacturing techniques and use of cupels across Europe and across various time periods.

The number of unearthed cupels presented in different papers from the excavations of the three archbishop's mints at the Archbishop's Palace in Trondheim vary. This is partly because the cupels were found in different areas and in different layers. Our catalogue includes a further 12 cupels to the 174 cupels and cupel fragments presented in earlier publications of the excavations. From our experience, some cupels have been stored in boxes together with crucibles and consequently they have been registered as crucibles rather than as cupels. However, in some boxes containing cupels also contain small crucibles, which have been registered and counted as cupels. Furthermore, it remains unclear whether some of the fragments we have identified as belonging together were perceived during the excavations as separate entities or parts of the same object. Therefore, there may still be some unidentified or wrongly identified cupels stored among other objects from the excavations.

In addition, it should be noted that of the 17 objects registered as N124864, one includes the note 'Plastic bag (921043)'. The reason for the object being stored with the rest of the objects under the same number, but separately in a plastic bag with another number, is unknown.

Table 1. A description of the 186 cupels/fragments of cupels

Museum Number	Comments	Diameter – outer upper rim (mm)					Diameter (mm)	Facing		Bottom shape and thickness		Full height	Height (mm)	Period	Phase	Period dating
		25	30	35	40	45	??	Yes	No	Ball	Thickness					
N 115972				X				X		X	13			11	2	c.1700-c.1783
N 121955					X			X		X	15			6	2	c.1500-1532
N 121964						X		X		X	16			6	1	c.1500-1532
N 121965				X					X	X	13			6	1	c.1500-1532
N 121976:1				X				X		X	10			6	3	c.1500-1532
N 121976:2	In 3 pieces, possibly belonging together			X				X		X				6	3	c.1500-1532
N 122015:1			X					X		X	7					
N 122015:2			X					X		X	11					
N 122015:3			X						X?	X	12					
N 122155:1				X				X		X	9					
N 122155:2				X				X		X	10					
N 122155:3				X				X		X	11					
N 122155:4			X					X		X	10					
N 122248			X					X		X	8					
N 122250	Depression				X			X		X		X	21	7	2	1532-c.1590
N 122267:1				X				X		X	10			7	2	1532-c.1590
N 122267:2	In two parts			X				X		X	12			7	2	1532-c.1590
N 122267:3	Small piece			X				X		X	9			7	2	1532-c.1590
N 122278				X				X				X	19	7	2	1532-c.1590
N 122294				X				X				X	21	6	3	c.1500-1532
N 122514:1				X				X				X	20	7	2	1532-c.1590
N 122514:2				X				X				X	23	7	2	1532-c.1590
N 122514:3				X				X		X	10			7	2	1532-c.1590
N 122514:4				X				X				X	22	7	2	1532-c.1590
N 122514:5				X				X		X	10			7	2	1532-c.1590

Museum Number	Comments	Diameter – outer upper rim (mm)					Diameter (mm)	Facing		Bottom shape and thickness		Full height	Height (mm)	Period	Phase	Period dating
N 122514:6	Well preserved facing			X				X		X	11			7	2	1532-c.1590
N 122514:7				X				X				X	18	7	2	1532-c.1590
N 122514:8				X				X		X				7	2	1532-c.1590
N 122514:9				X				X		X	11			7	2	1532-c.1590
N 122514:10				X				X		X	9			7	2	1532-c.1590
N 122514:11				X				X		X	8			7	2	1532-c.1590
N 122524:1						X		X				X	31	7	3	1532-c.1590
N 122524:2	Depression					X		X		X	10			7	3	1532-c.1590
N 122525														7	3	1532-c.1590
N 122536		X						X		X	5			7	3	1532-c.1590
N 122563	Small piece			X						X				7	3	1532-c.1590
N 122649:1				X				?		X	9			7	3	1532-c.1590
N 122649:2				X				X		X	8			7	3	1532-c.1590
N 122661:1						X		X		X	18					
N 122662:1	Perfect			X				X				X	18	7	3	1532-c.1590
N 122662:2				X				X		X		X		7	3	1532-c.1590
N 122662:3				X				?		X				7	3	1532-c.1590
N 122666:1					X			X		X	29			7	3	1532-c.1590
N 122666:2						X		X		X	20			7	3	1532-c.1590
N 122666:3	Small piece					X		?		?	?			7	3	1532-c.1590
N 122667	Not clean		X					?		X	?			7	3	1532-c.1590
N 122668:1				X				X				X	19	7	3	1532-c.1590
N 122668:2				X				X		X	9			7	3	1532-c.1590
N 122668:3				X				X		X	7			7	3	1532-c.1590
N 122674				X				X		X	7			7	3	1532-c.1590
N 122677				X				X				X	20	7	3	1532-c.1590
N 122681				X					X?	X	6			7	3	1532-c.1590

Museum Number	Comments	Diameter – outer upper rim (mm)					Diameter (mm)	Facing		Bottom shape and thickness		Full height	Height (mm)	Period	Phase	Period dating
N 122690				X				X				X	19	7	3	1532-c.1590
N 122696:1				X				X				X	19			
N 122696:2			X					X		X	8					
N 123089			X					X		X	7			7	3	1532-c.1590
N 124837				X				?		X	12			11	4	c.1700-c.1783
N 124839				X				X		X	10			9	1	1640-1672
N 124852:1	3x+14 exhibited in the Archbishops Palace			X				X		X	9			6	3	c.1500-1532
N 124852:2	Half			X				X		X	10			6	3	c.1500-1532
N 124852:3				X				X		X	8			6	3	c.1500-1532
N 124852:4	Depression				X			X		X	6			6	3	c.1500-1532
N 124852:5			X					X		X	11			6	3	c.1500-1532
N 124852:6			X					X		X	8			6	3	c.1500-1532
N 124852:7				X				X		X	8			6	3	c.1500-1532
N 124852:8				X				X		X	10			6	3	c.1500-1532
N 124852:9	Depression				X			X		X	10			6	3	c.1500-1532
N 124852:10	Depression				X			X		X	9			6	3	c.1500-1532
N 124852:11				X				X		X	9			6	3	c.1500-1532
N 124852:12				X				X		X	12			6	3	c.1500-1532
N 124852:13				X				X		X	8			6	3	c.1500-1532
N 124852:14	Depression				X			X		X	7			6	3	c.1500-1532
N 124852:15	Depression				X			X		X	9			6	3	c.1500-1532
N 124852:16			X					X		X	9			6	3	c.1500-1532
N 124852:17							32	X		X	11			6	3	c.1500-1532
N 124855	Two layers + facing		X					X		X	9			6	3	c.1500-1532
N 124857:1	Depression					X		X		X				6	3	c.1500-1532
N 124857:2	Depression					X		X		X				6	3	c.1500-1532
N 124857:3	Depression					X		X		X				6	3	c.1500-1532
N 124857:4	In 3 pieces					X		X		X				6	3	c.1500-1532

Museum Number	Comments	Diameter – outer upper rim (mm)					Diameter (mm)	Facing		Bottom shape and thickness		Full height	Height (mm)	Period	Phase	Period dating
N 124858:1	Depression				X			X		X				6	3	c.1500-1532
N 124858:2	Depression				X			X		X				6	3	c.1500-1532
N 124858:3				X				X		X				6	3	c.1500-1532
N 124858:4			X					X		X				6	3	c.1500-1532
N 124858:5			X					X						6	3	c.1500-1532
N 124859:1					X			X		X				9	1	1640-1672
N 124859:2				X				X				X	21	9	1	1640-1672
N 124859:3				X				X		X				9	1	1640-1672
N 124859:4				X				X		X				9	1	1640-1672
N 124860														9	1	1640-1672
N 124862				X				X		X				7	2	1532-c.1590
N 124864:1	3 × with steps (Smaller monk?)		X					X		X	10			7	3	1532-c.1590
N 124864:2				X				X		X	11-15			7	3	1532-c.1590
N 124864:3			X					X		X	9-13			7	3	1532-c.1590
N 124864:4				X				X		X	7-18			7	3	1532-c.1590
N 124864:5				X				X		X	6-15			7	3	1532-c.1590
N 124864:6			X					X		X	7-10			7	3	1532-c.1590
N 124864:7				X				X		X	9-21			7	3	1532-c.1590
N 124864:8				X				X		X	8-10			7	3	1532-c.1590
N 124864:9				X				X		X	10-13			7	3	1532-c.1590
N 124864:10				X				X		X	11-17			7	3	1532-c.1590
N 124864:11				X				X		X	11-21			7	3	1532-c.1590
N 124864:12			X					X		X	9-10			7	3	1532-c.1590
N 124864:13				X				X		X	9-10			7	3	1532-c.1590
N 124864:14			X					X		X	8-9			7	3	1532-c.1590
N 124864:15			?					?		?	??			7	3	1532-c.1590
N 124864:16			X					X		X	9			7	3	1532-c.1590
N 124864:17	Plastic bag (921043)			X				X		X	9-14			7	3	1532-c.1590
N 124866	Depression				X			X		X	10			7	3	1532-c.1590

Museum Number	Comments	Diameter – outer upper rim (mm)					Diameter (mm)	Facing		Bottom shape and thickness		Full height	Height (mm)	Period	Phase	Period dating
N 124867:1	Depression				X			X		X				7	2	1532-c.1590
N 124867:2				X				X		X				7	2	1532-c.1590
N 124867:3	With bead			X				X		X				7	2	1532-c.1590
N 124867:4			X					X		X				7	2	1532-c.1590
N 124867:5			X					X		X				7	2	1532-c.1590
N 124868:1				X				X		X				7	2	1532-c.1590
N 124868:2				X				X		X				7	2	1532-c.1590
N 124876				X				X		X	7			6	4	c.1500-1532
N 124879				X				X				X	20	6	4	c.1500-1532
N 124881:1				X				X				X	20	6	4	c.1500-1532
N 124881:2				X				X				X	21	6	4	c.1500-1532
N 124881:3				X				X		X	13			6	4	c.1500-1532
N 124883:1				X				X		X	10			7	1	1532-c.1590
N 124883:2	Depression					X		X		X	13-15			7	1	1532-c.1590
N 124885				X				X		X	16			7	2	1532-c.1590
N 124886				X				X				X	20			
N 124887:1				X				X		X	8-12			6	4	c.1500-1532
N 124891				X				X		X	10			7	3	1532-c.1590
N 124894:1	Depression					X		X		X	13			6	3	c.1500-1532
N 124894:2				X				X		X	9			6	3	c.1500-1532
N 124894:3				X				X		X	11			6	3	c.1500-1532
N 124894:4				X				X		X	8			6	3	c.1500-1532
N 124895:1	Depression				X			X		X	8-16			6	3	c.1500-1532
N 124895:2			X					X		X	17			6	3	c.1500-1532
N 124896:1	Depression			X		X		X		X	9			6	3	c.1500-1532
N 124896:2	PbO left?			X				X		X	8			6	3	c.1500-1532
N 124896:3				X				X		X	10			6	3	c.1500-1532
N 124896:4				X				X		X	8			6	3	c.1500-1532
N 124897				X				X		X	9			6	4	c.1500-1532
N 124911	In 2 pieces			X				X		X	8-17			6	4	c.1500-1532

Museum Number	Comments	Diameter – outer upper rim (mm)					Diameter (mm)	Facing		Bottom shape and thickness		Full height	Height (mm)	Period	Phase	Period dating
N 124912	Depression				X			X		X	10			6	3	c.1500-1532
N 124913				X				X				X	19	6	3	c.1500-1532
N 124917:1	Ag bead?				X			X		X	8			6	3	c.1500-1532
N 124917:2	Pb on outside?				X			X		X	9			6	3	c.1500-1532
N 124918			X					X		X	10					
N 124920:1			X					X		X	6-12					
N 124923					X			X		X	11			6	3	c.1500-1532
N 124924					X			X				X?	?	6	4	c.1500-1532
N 124925						X		X			13			6	4	c.1500-1532
N 124926	Brown facing				X			X		X	8			6	3	c.1500-1532
N 124929	Fragment				X			??		X	??					
N 124930				X				X				X	20	6	3	c.1500-1532
N 124931					X			X		X	11			6	3	c.1500-1532
N 124945:1					X			X				X	17	12	2	c.1783-1991
N 124945:2					X			X		X	10			12	2	c.1783-1991
N 124946:1				X				X		X	11			12	2	c.1783-1991
N 124946:2				X				X		X	11			12	2	c.1783-1991
N 124954	Dirty			X				?		X	19			6	3	c.1500-1532
N 124957	Depression				X			X		X	11			9	1	1640-1672
N 124960				X				X		X	8			9	1	1640-1672
N 125118				X				X		X	14			7	3	1532-c.1590
N 125122			X					X		X	8			7	3	1532-c.1590
N 125123		X						X		X	10			7	3	1532-c.1590
N 125124:1		X						X		X	7			7	3	1532-c.1590
N 125125			X					X		X	10					
N 125126		X						X		X	9			7	3	1532-c.1590
N 125127:1		X						X		X	9			7	2	1532-c.1590
N 125127:2		X						X		X	9			7	2	1532-c.1590
N 125127:3		X						X		X	8			7	2	1532-c.1590
N 125127:4		X						X		X	9			7	2	1532-c.1590

Museum Number	Comments	Diameter – outer upper rim (mm)					Diameter (mm)	Facing		Bottom shape and thickness		Full height	Height (mm)	Period	Phase	Period dating
N 125127:5		X						X		X	10			7	2	1532-c.1590
N 125128		X						X		X	9			7	2	1532-c.1590
N 125132:1				X				X		X	18					
N 125141			X					X		X				9	1	
N 125144				X				X		X	10			9	1	
N 125145	In 2 pieces					X		X		X	14-22			7	1	1532-c.1590
N 125151:1				X				X				X	25	7	3	1532-c.1590
N 125151:2				X				X				X	21	7	3	1532-c.1590
N 125151:3				X				X		X	7			7	3	1532-c.1590
N 125151:4				X				X				X	21	7	3	1532-c.1590
N 125153:1				X				X		X	12			7	3	1532-c.1590
N 125153:1				X				X		X	14			7	3	1532-c.1590
N 125154			X	X				X		X	8					
N 134396	Depression					X		X		X				8	1	
N 138757	Cu particle?					X		X?		X				6	2	c.1500-1532
N 139439	Depression				X			X		X				2	1	Late Viking Age / Early Medieval
N 139622	Depression				X			X				X	24	6	2	c.1500-1532

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FROM CALVES' SCULLS TO FINISHED BONE-ASH CUPELS AND TESTING OF PRECIOUS METALS

An investigation of the cupellation practised in the Mint in the Archbishop's Palace, Trondheim, in medieval times

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Abstract

The paper discusses three topics: (1) the production process of bone-ash cupels from bones in calves' sculls to finished cupels, (2) cupellation of a silver and a gold alloy with known compositions in replica cupels and (3) how lead oxide is distributed in the cupel after cupellation. Following the precept of Ercker in his treatise published in 1574, the authors' production of cupels is described in detail, from boiling the calves' heads to the finished cupels, each with a fine-grained surface layer. The microstructure of bone-ash particles and the flow of molten lead oxide into the cupel in a replica cupel and a cupel found during the archaeological excavations in the Archbishop's Palace, Trondheim, in 1991–1995 are compared. The experience gained from making cupels and later use of the cupels for cupellation led the authors to the conclusion that the cupels found during the archaeological excavations on the premises of the 16th century Mint in the Archbishop's Palace were most likely produced locally at the Mint. By using his own cupels, the contemporary moneyer might have measured the fineness of precious metals with an acceptable degree of precision.

Introduction

Following a fire in 1983 that ravaged the wooden buildings forming the eastern and southern wings of the Archbishop's Palace of Nidaros (present-day Trondheim), archaeological excavations took place between 1991 and 1995 (McLees, 1996; Saunders 2001; Nordeide 2003). The excavations identified three successive mint complexes, one above the other, in the northern part of the eastern wing. All of them were near the perimeter wall of the Palace. Their structure and layout, with workbenches, tiled floors and hearths, clearly indicate the purpose of the buildings as workshops of medieval moneyers, corresponding to illustrations and instructions of minting in contemporary treatises. The interpretation of the buildings as mints was further corroborated by archaeological evidence from the excavations, with important finds of metalworking debris from coin production, such as bone-ash cupels, crucible fragments, hammered rods, blanks, and coins. The finds are mainly from the site's archaeological period 6, corresponding to AD 1500–1532, and period 7, corresponding to AD 1532–1590. The majority of the cupels relate either to destruction layers associated with the second and third workshop or to waste layers. Judging from dendrochronological measurements of timber in the walls, the oldest mint complex was established about AD 1500 (Olsson, 2000) and is today exhibited *in situ* in the museum part of the Archbishop's Palace, Trondheim.

During the excavations, 143 bone-ash cupels and 31 cupel fragments were unearthed (Saunders, 2001, pp. 27–33).[1] The total has since been increased from 174 to 186 cupels and cupel fragments (Risvaag et al., 2021, this volume).

The use of bone-ash cupels was a standard technique in the assaying of precious metals in the 16th century. The cupels have an inherent capacity to enable the separation of precious metals from other metals, thereby permitting calculations of the fineness (i.e. purity) of a sample. Unused cupels have a low weight. The weights of the excavated cupels indicated that all of them had been used. As the archbishops in medieval Trondheim did not have any silver mines, bone-ash cupels were used to measure the fineness of precious metals being paid to the archbishops and to establish the correct alloy composition when making bullion for use in coin production.

Guidelines on how to make cupels are given in several contemporary treatises, including those by Biringuccio (1966 [1540]), Agricola (1950 [1556]) and Ercker (1951 [1574]). For our project in which we attempted to make bone-ash cupels, we followed the recommendations provided by Ercker. As pointed out by Bartels (2010),

1 Se also: Bergstøl, S., & S. W. Nordeide. 1993. *"Analyses of crucibles from the late medieval mint in the Archbishop's Palace, Trondheim,"*. Unpublished.

even though Ercker was as a skilled university-educated theorist, his knowledge relied as much on hands-on experience as would an apprentice. Thus, knowledge of procedure in practice was as important as theory, which in turn meant there were some gaps in his treatise. Although Erker was quite meticulous in his description, both of the making of cupels and the cupellation process, some degree of interpretation is left to the modern reader. What he regarded as a hands-on procedure remained unsaid, possibly to avoid stating the obvious. Ryle's classical distinction of 'knowledge-how' and 'knowledge-that' springs to mind (Ryle, 1949 (1990)).

Modern investigations of 16th century cupels from the Tower of London (White, 2010) have shown that they were made from bone-ash, while cupels from Oberstockstall, Austria (Rehren, 1998; Martinon-Torres and Rehren, 2005) were made of about two-thirds bone-ash and one-third clay mixed in as a binding agent. Mid-14th century bone-ash cupels from Pymont, France (Rehren and Eckstein, 1998) contained c.10% by weight of other materials.

Furthermore, investigations have provided detailed information about testing methods (e.g. Bayley, 1991; Sieblist, 2006). A modern description of how to assay precious metals is available in *ASTM E1335 - 08(2017): Standard Test Methods for Determination of Gold in Bullion by Fire Assay Cupellation Analysis* (ASTM International).

However, few papers deal with the microstructure of cupels and the resulting beads after cupellation in general (Téreygeol and Thomas, 2003). This gap in knowledge was addressed by an investigation of bone-ash cupels from the Mint in the Archbishop's Palace in Trondheim (Ulseth et al., 2015). However, one question remained open: Were the cupels imported, similar to the crucibles that were imported from Germany,[2] or were the cupels made at the Mint in Nidaros (present-day Trondheim)? In an attempt to answer this question we decided to produce cupels from scratch and to conduct some experiments in the cupellation of precious metals with known fineness. We anticipated that if we managed to make cupels that allowed for acceptable results in terms of fineness of the samples after cupellation, it would be likely that a skilled moneyer appointed by the Archbishop of Nidaros would have managed to produce good cupels (Lohne and Ulseth, 2021, this volume).[3]

As it is possible to make metallographically prepared specimens from used coupels impregnated with molten lead oxide, we compared a cross-section of a used replica cupel with a cross-section of a cupel (N124860) found during archaeological excavations in 1991–1995.

2 Lohne, O., Ulseth, P. and Risvaag, J.A., 2015. Undersøkelse av digelfragmenter fra funn i Erkebispegården. En statusrapport. 24 pp. [Unpublished report in Norwegian]

3 See also: Lohne, O. and Ulseth, P., 2016. *Framstilling av beinaskekupeller fra grunnen av og erfaringer med kupellering*. Notat. 15 pages in Norwegian.

Experiments

Preparation of bone-ash cupels

From bones to bone-ash powder: Agricola (1950 [1556]) and Ercker (1951 [1574]) proposed both that calves' sculls were the best material for bone-ash cupels. Our investigations of cupels unearthed in the archbishops' mints showed that they were made from animal bones (Ulseth et al., 2015). When replicating the manufacture of the cupels, rather than using commercial bone-ash as described from experiments by Téreygeol and Thomas, (2003), we went to a local slaughterhouse, where we were given two calves' heads. The heads were boiled to remove the flesh (Fig. 1) and the bones subsequently cleaned by brushing.

Figure 1. A calf's head during boiling.

The different bones that made up a calf scull were separated and then calcinated by putting them in a wood-fired oven. After 20 minutes the bones were taken out and cleaned to remove the ashes (Fig. 2). Bones from the forehead were broken into pieces, ground in a mortar and sieved (Fig. 3).

Coarser particles were removed and then ground and sieved once again. The bone-ash powder of 173 g from one scull had a volume of 200 ml in a beaker.

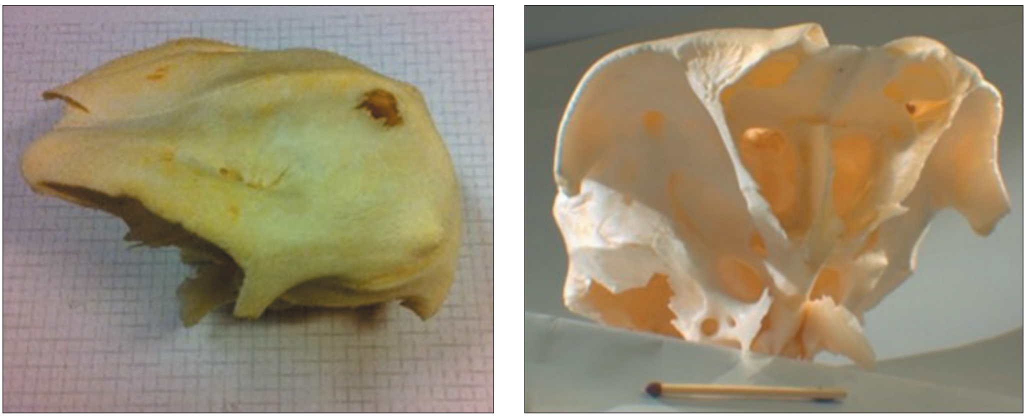

Figure 2. A cleaned calf's scull viewed from two directions. Dimensions of paper squares: 5 × 5 mm.

Figure 3. The forehead bones ground in a mortar and then sieved. Sieve mesh aperture: 1 × 1 mm.

Washing and drying the powder: Warm water was added to the beaker while stirring. The volume of powder + water was 600 ml. Coarse particles settled, while fine particles remained as a muddy liquid. The liquid had a pH of 9–10.

After 30 minutes the water was still muddy, but a thin layer of foam had appeared on the surface of the water. The foam was skimmed off and 370 ml of the muddy water was poured into another beaker. Thereafter, 370 ml warm water was added to the beaker with the remaining coarse particles while stirring. After standing for 16 hours at room temperature, the water appeared clean in both beakers, with settled powders at the bottom (Fig. 4).

The water in both beakers was poured off and the powders taken out with a spoon. The fine powder was placed in a separate glass dish and the coarse powder placed in

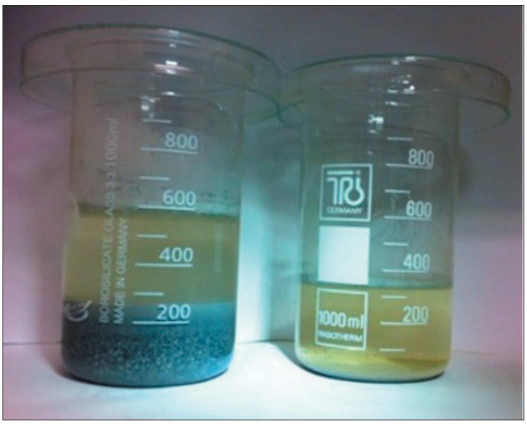

Figure 4. The beakers and their contents after standing for 16 hours at room temperature. The water appears clean in both beakers, with coarse powder (left) and fine powder (right) at the bottom.

six heaps in a further two glass dishes (three heaps in each dish) (Fig. 5). All three dishes were placed in a drying cabinet heated at 50°C for 24 hours until the powders were dry.

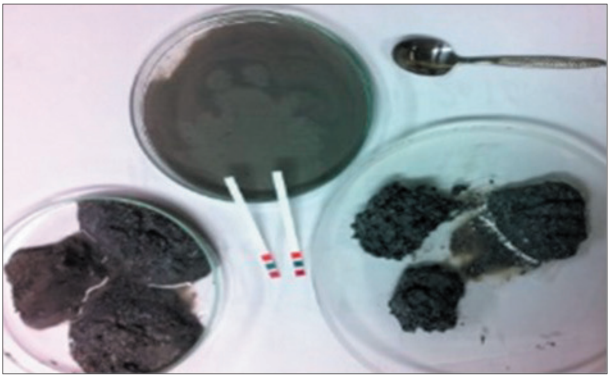

Figure 5. Wet powder before the drying process was started.

After drying, both the coarse and fine powders are ground separately and sieved (Fig.6). The fine powder was later used for the surface of the cupel (i.e. for the 'facing'), while the coarse powders were ground and sieved once again before mixing. The latter material was later used to make the main body of the cupels.

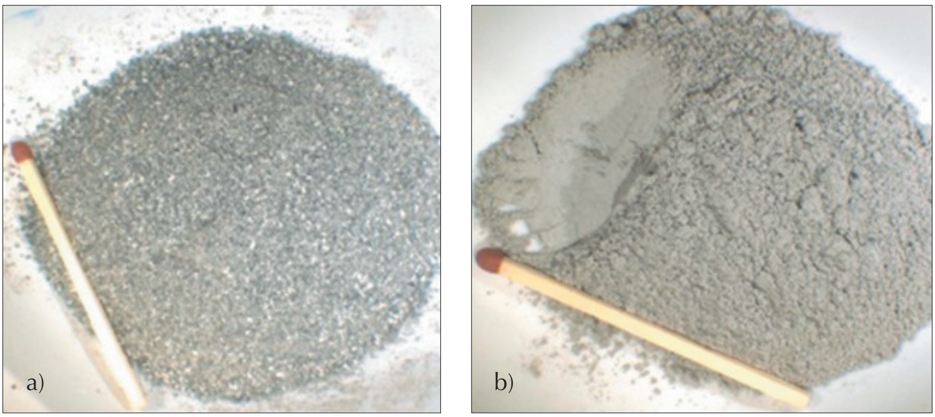

Figure 6. Powders after drying and being ground and sieved once again. a) Coarse powder, b) Fine powder.

Shaping and drying the cupels: A special tool for making cupels has been locally turned from brass (Fig. 7). It consists of a conical ring, 20 mm high and with an inner top diameter of 34.6 mm and an inner bottom diameter of 27.4 mm, together with a stamp, the 'monk', which fits into the top of the ring. The size of the tool corresponds to the most frequent dimension (35 mm) of cupels found during the excavations of the Archbishop's Palace in 1991–1995 (Risvaag et al., 2021, this volume).

Ercker (1951 [1574], p. 29) explained the process of shaping cupels as follows:

> *Moisten the powder with strong beer. Do not use too much of the liquid. When you press some in your hand the lump should just hold together. Then pack the cupel mold full of ashes, scrape or wipe off excess, and with one to four blows of the mallet drive the monk into the mold. Wipe the monk clean and with a little wooden spoon sprinkle good facing ashes on the cupel while it is still in the mold. Spread the facing ashes with your fingers. Then put the monk back in again, fit it on straight, and with two or three straight blows, whichever the case may require, beat down the facing in the cupel so that it adheres very evenly. Then lift out the monk and press out the cupel from the mold.*

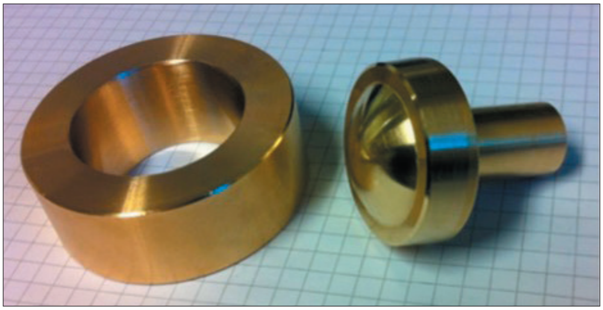

Figure 7. The cupel tool, ring (left) and the 'monk' (right).

We used the following process:
1 We used strong ale from the brewery Nøgne Ø. The ale had a 10% ABV (alcohol by volume) and was unfiltered. We did not test the use of water instead of ale.
2 It was important that the ashes were not too wet. When liquid was observed seeping out between the ring and the monk it indicated that the ashes were too wet.
3 Two ways of drying the cupels were tested: (1) 24 hours in a drying cabinet at 50°C, and (2) two weeks at room temperature. Drying at room temperature for two weeks gave the strongest cupels.

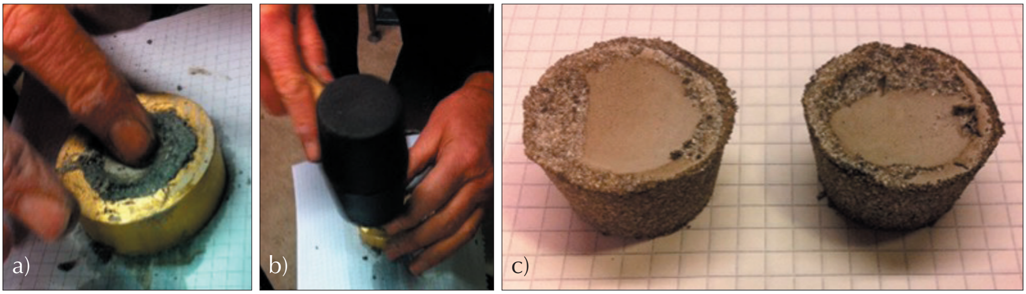

Figure 8. a) Filling the mould. b) Driving the 'monk' into the mould. c) Cupels with fine grained facing on the bowl surface. Dimensions of paper squares: 5 × 5 mm.

Cupellation of a silver alloy and a gold alloy

Two alloys were tested: a silver alloy with a fineness of 925 ‰ silver (Ag) (the rest was mainly copper (Cu) and zinc (Zn)), and a gold alloy with 585 ‰ gold (Au) and 198 ‰ Ag. When testing the 925 silver alloy, a piece of thin lead plate weighing 1.40 g was folded around the piece of silver weighing 0.15 g and put into a pre-heated cupel lying in a vessel and placed in a furnace operated at a temperature of 900°C. We were able to follow the process by looking through a quartz glass window. The specimen melted quickly and turned into a yellowish white sphere (Fig. 9).

Figure 9. The yellowish white melted metal sphere in the cupel placed in a vessel in a furnace operated at a temperature of 900°C.

During annealing some evaporation above the melt button was observed and a 'flash' was seen. After five minutes, the vessel containing the cupel was taken out of the furnace, placed on a table at room temperature and left to cool. The surface of the bead from the silver alloy was smooth and shiny in some places and rough elsewhere (Fig. 10a, and Fig. 10c). Investigations using a scanning electron microscope (SEM) showed that the smooth area was almost pure silver, and the erupted material was silver and lead oxide with a small amount of copper and traces of calcium and phosphorous (from the bone-ash). The weight of the bead after cupellation of the silver alloy was 0.14 g.

The ASTM - E1335-08 standard recommends that the assaying procedure should be repeated if 'spitting' occurs (ASTM International).

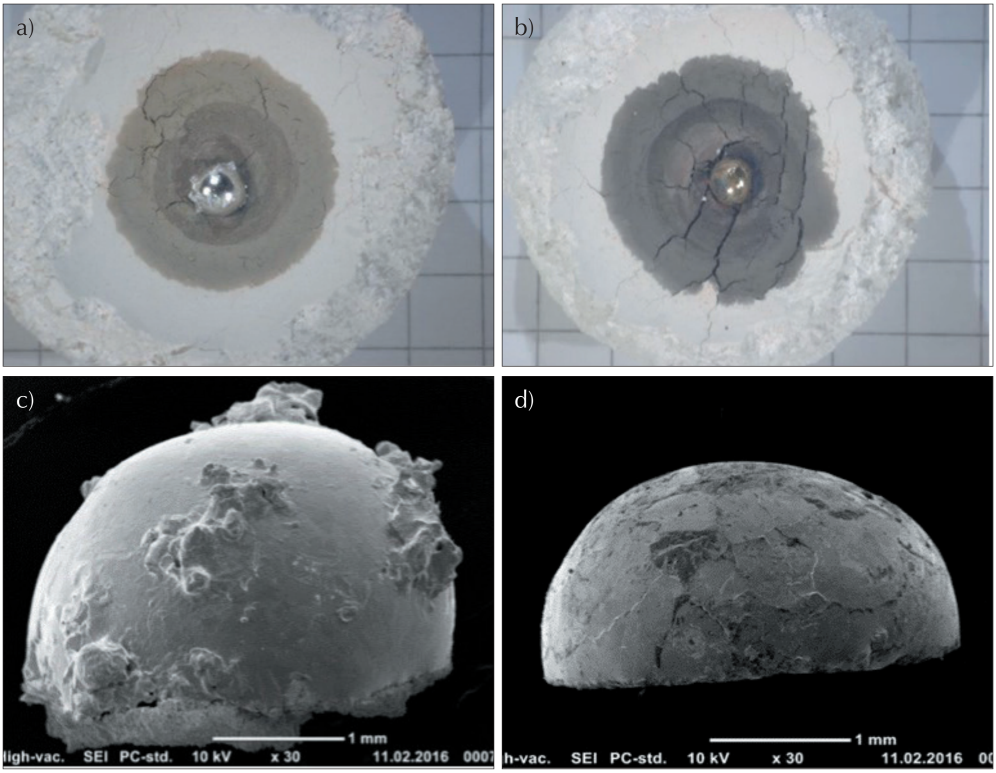

Figure 10. Cupels with a bead after cupellation of a) 925 ‰ silver alloy and b) 585 ‰ gold alloy. Dimensions of paper squares: 5 × 5 mm. In images c) and d) the beads have been removed from the cupels and investigated using SEM (side view). Note the rough surface in c), which shows that the silver bead was 'spitting'. No spitting is visible on the gold bead in image d).

Later experiments conducted using the silver alloy and with longer annealing time at 900°C and slower cooling did not result in spitting but there was a small shrinkage cavity on top of the bead and 'islands' of thin lead oxide flakes on the surface (Lohne and Ulseth, 2021, this volume).

The same procedure was used when testing the gold alloy (Fig. 10b). However, no spitting was observed. Instead, some surface grains were displaced, resulting in a rough surface (Fig. 10d).

Investigations of the surface layer of cupels after cupellation

Used replica cupels: A piece of the surface layer of a cupel after cupellation was cut out for further investigation. The piece was cast in resin (EpoFix) and then the cast was cut to show a cross-section of an area, where subsequently we observed that the lead oxide had been sucked into the cupel. After grinding on SiC (silicon carbide) grinding paper and diamond polishing, the specimen was carbon coated and investigated by using an electron probe microanalyser (EPMA) equipped with an energy dispersive x-ray spectrometer (EDS) and wavelength dispersive x-ray spectrometers (WDS) to measure the chemical compositions of microscopic areas (Figs. 11–13).

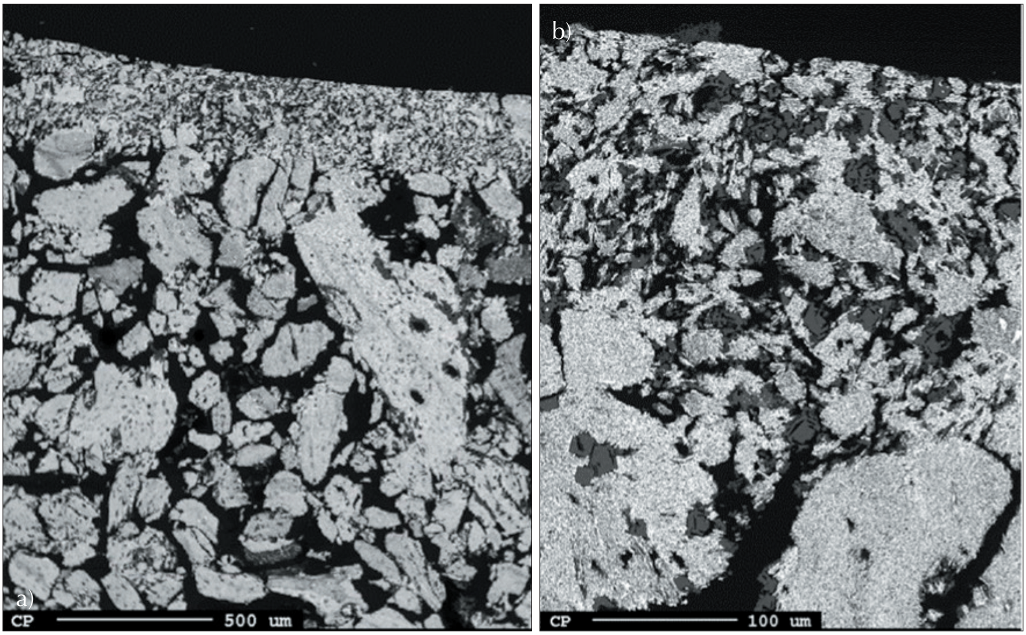

Figure 11. Microprobe images at different magnifications of a cross-section of a used replica cupel taken near to the bowl surface. a) Microstructures near the surface, with a fine-grained bone-ash layer with a thickness of c.200 µm on top. Black areas are pores filled with resin. Bone-ash particles (grey) are filled with lead oxide (white) (see Figure 12). b) The fine-grained surface layer at higher magnification.

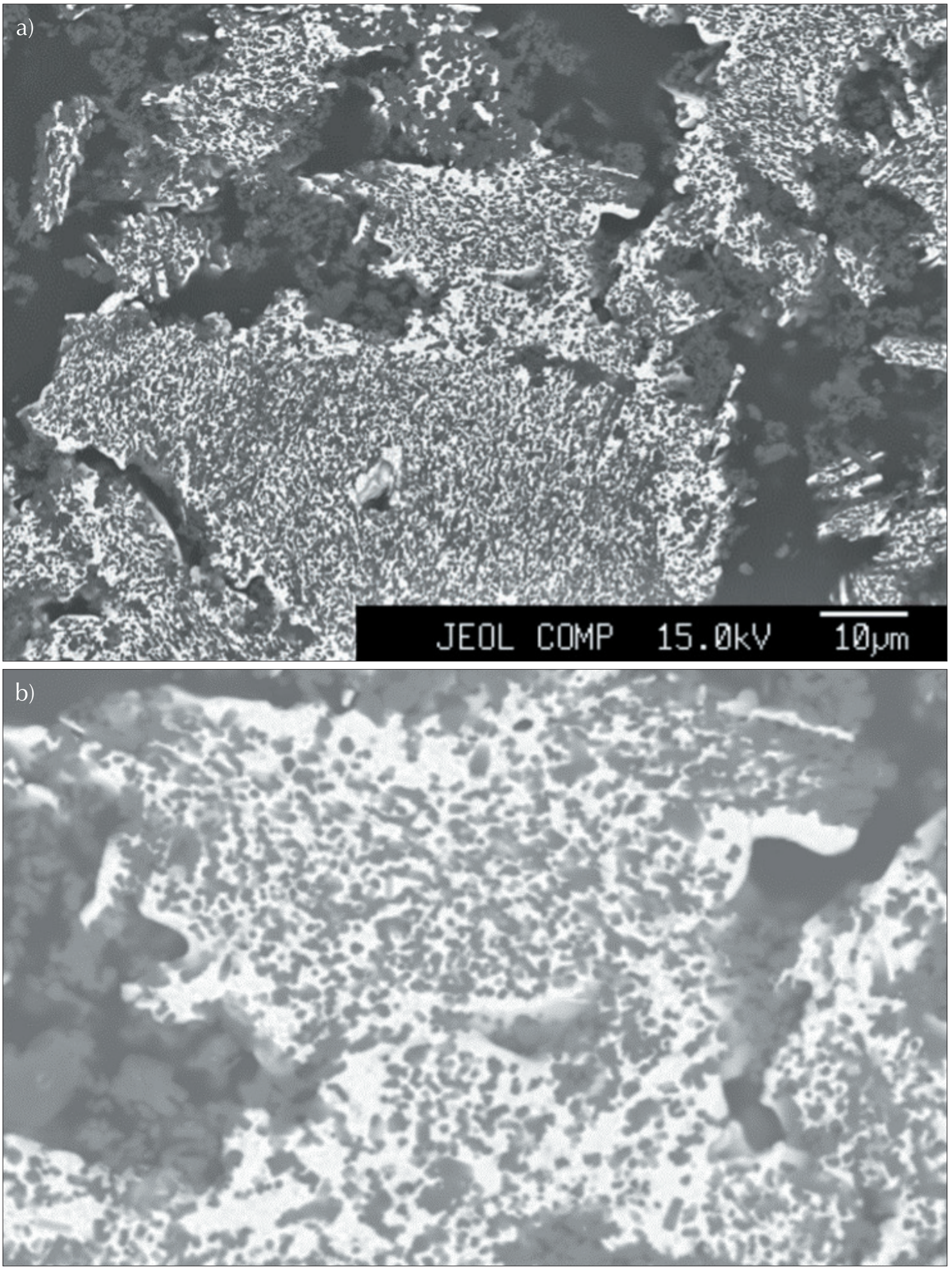

Figure 12. Microprobe images. a) Coarse particles from just below the bowl surface layer. Dark areas are cavities (filled with resins), grey are bone-ash and white are lead oxide. The porous bone-ash particles are filled with lead oxide. b) A central grain seen in a) is magnified 3 ×. Note that the lead oxide is both lying on the surface of the bone-ash particles and filling the fine bone-ash canals. The cavities between the coarse particles (see Figure 11), are not filled with lead oxide.

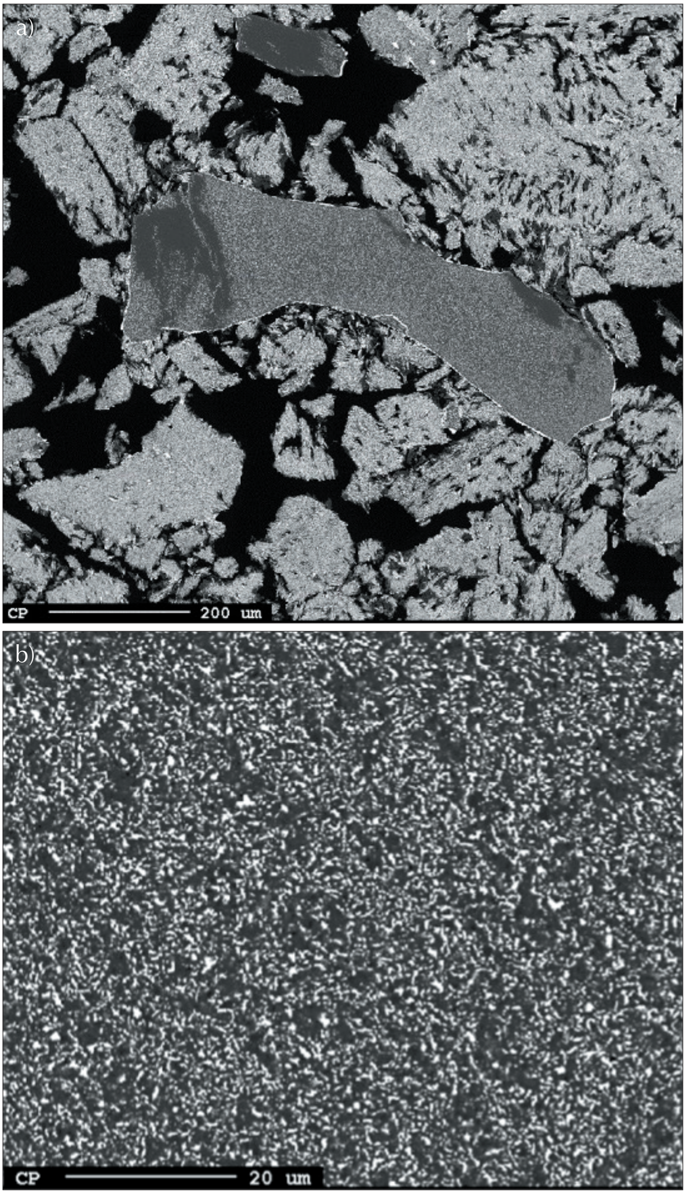

Figure 13. Microprobe images. a) Bone-ash particles appear in different shades of grey according to the size of the lead oxide filled canals (white). Note the white rim of the dark central particle. b) Part of the central dark particle in a) shown at higher magnification. White lead oxide patterns are clearly visible. Molten lead oxide has filled even the finest canals.

Comparison of a used replica cupel and the excavated cupel N124860

Cross sections of cupels are shown in Figs. 14-16:

Figure 14. A cross-section of the excavated cupel N124860. The upper, outer rim diameter is 35 mm and the height 23 mm. Note the fine-grained surface layer (i.e. the facing) at the bottom of the bowl. The two dark areas at the bottom of the cupel show where lead oxide has filled the cavities between the bone-ash particles, as well as the interior of the particles (see Figure 16). Photo: Åge Hojem, NTNU University Museum.

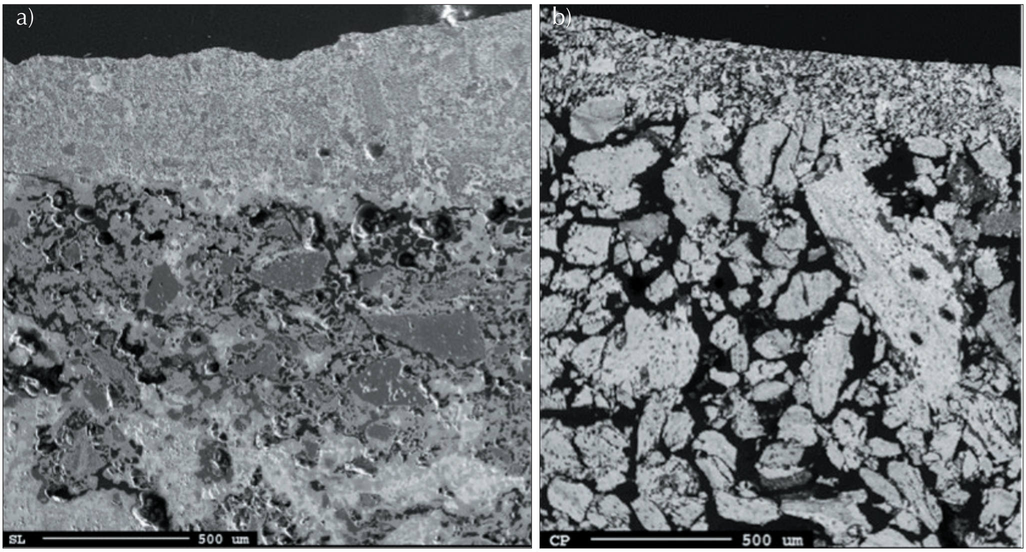

Figure 15. Microprobe images from material near the bowl surface of the cupel. a) N124860 and b) used replica cupel. The area fraction of cavities (black) is higher in the replica cupel than in the excavated cupel. Additionally, the facing is thicker and denser in N124860 than in the replica cupel.

Discussion

No tools for making cupels were found during the archaeological excavations in 1991–1995. This raises the question of how and where the cupels were produced. To address this question the main aim of our experiments was to generate some background information. We wanted to determine whether the excavated bone-ash cupels were produced locally in the Mint at the Archbishop's Palace or whether they were imported, as were the crucibles that had production marks pointing to the Hessian town of Großalmerode (Stephan, 1995).[4]

Microstructure of cupels

Unused bone-ash cupels are brittle. By contrast, the used cupels filled with lead oxide are strong and gave the opportunity to produce, by metallographic techniques, specimens that could be used to compare the microstructures of the replica cupels with cupels found during the archaeological excavations.

In Fig. 15 two main differences are seen:

1 The fine-grained facing layer in N124860 is denser and at least 50 % thicker than in our replica cupel. There will of course be variations, and no statistical variations are available, but the tendency may be clear. Except from one or two cupels all excavated cupels had a facing layer (Risvaag et al., 2021, this volume).

2 In N124860 there is a greater variation in the size of bone-ash particles in bulk than in our replica cupel, and the porosity is less. The same structure could have been obtained in our replica cupel if some fine-grained bone-ash powder had been mixed in, as proposed by Ercker, and that the compaction of the wet lump of ashes had been better.

Although there may be a greater variation in bone quality in N124860 than in the replica cupels, the structure of N124860 may be better suited for cupellation than our replica cupels.

4 See also: Lohne, O., Ulseth, P. and Risvaag, J.A., 2015. Undersøkelse av digelfragmenter fra funn i Erkebispegården. En statusrapport. 24 pp. [Unpublished report in Norwegian]

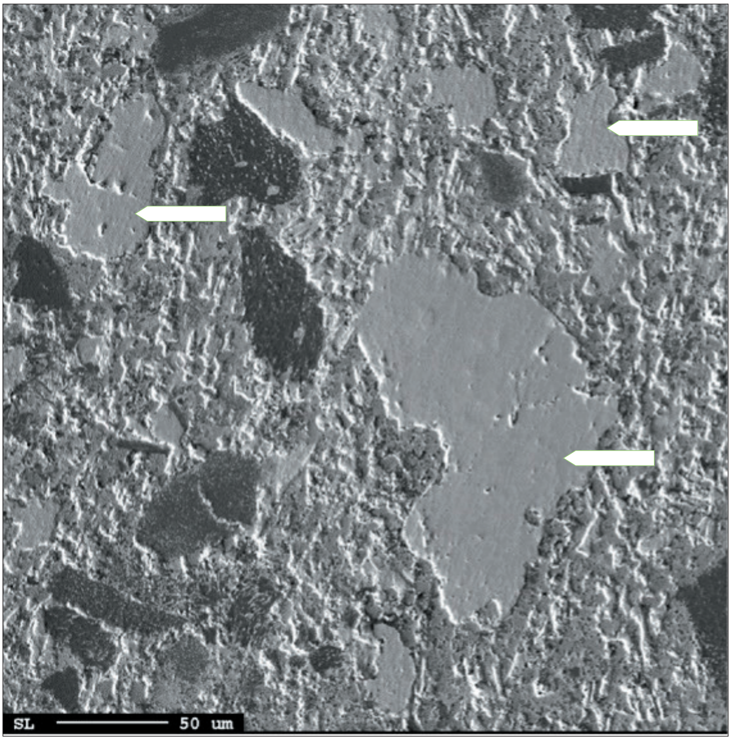

Figure 16. Microprobe images of dark areas from N124860 (see Figure 14). 'Lakes' of lead oxide (pale grey areas, indicted by arrows) have filled the cavities between bone-ash particles. Filled bone-ash particles appear in different shades of grey.

The flow of liquid lead oxide into the cupel

Figures 11–16 show what happens inside the cupel during cupellation. Molten lead oxide wets the bone-ash, flows over the surface of the particles, and through capillary action is sucked into the canals (whether fine or coarse) in the bone-ash. The bone-ash particles are in contact with each other and the molten lead oxide may flow from one particle to the next without filling the cavities between the particles. When the lead oxide has solidified, the filled bone-ash particles become stronger, and are glued together. Volumes in which the particles have not been hardened this way will, after the cupellation process, continue to be brittle and easily crumble away. This may account for why most of the excavated cupels (83%) lack part of their original bottom part and upper rim (Fig. 3) (Risvaag et al. 2021, this volume).

On the other hand, re-use of bone-ash from parts of the cupels that had not been impregnated with molten lead oxide has been tested in other new replicas, with good results. Thus, it is possible that the moneyer in Nidaros reused bone-ash from such materials without problems.

If there is still some molten lead oxide left around the silver bead in the cupel when all bone-ash particles in the cupel have been filled, the melt will start to fill the cavities between particles from the bottom (see the dark areas in Fig. 14 and details in Fig. 16). Therefore, it would have been necessary for the moneyer to select the size of the cupels according to the expected amount of lead oxide produced during the cupellation process and the capacity of the cupel to absorb molten lead oxide. Among the 186 cupels of five size classes, the majority (83%) lacked part of their bottom (Risvaag et al., 2021, this volume). This indicates that the assayer normally selected cupels of the right size when he performed his cupellation measurements.

To summarize, molten lead oxide wets the bone-ash particles, covers the surface of the particles, and is sucked into the particles by capillary action (Fig. 12). The molten lead oxide does not fill the pores between the particles until all bone-ash particles have been filled. When all particles have been filled, the pores between the bone-ash particles start to fill, from the bottom up (Figs. 14 and 16).

Estimation of fineness

At Kongsberg, Norway, the mining of silver started in AD 1625. The Royal Mint Museum, Kongsberg, has some working precepts that inform how the fineness is calculated in a specimen after cupellation:

> *The silver bead is picked out of the cupel with a pair of tweezers and carefully cleaned. The bead thus produced, which consists of pure silver, is then weighed on the specified pair of balances. The weight of the silver bead compared with the weight of the starting specimen gives the percentage of silver in the material from which the specimen has been taken.*[5]

The silver bead shown in Fig. 10c had a weight of 0.14 g, indicating a content of 930 ‰ Ag in the starting piece of 0.15 g. This is close to the fineness given by the metal producer (K.A. Rasmussen, Hamar), 925 ‰ However, we know that some copper would have been dissolved in the silver and possibly some lead and/or lead oxide and copper and/or copper oxide in between the grains in the bead (Lohne and Ulseth, 2021, this volume). Fig. 10c shows a small piece of facing of low weight sticking to the bottom of the bead. These factors might have resulted in a higher weight than the weight of the pure silver in the bead. However, some silver might have been lost to the lead oxide and either evaporated or sucked into the cupel. The

5 From: Katalog for Sølvverksmuseet, Kongsberg, 1942. Handwritten, possibly by a secretary under dictation of the foreman of miners Bjarne Sanness. Our translation. Ref. Per Øyvind Østensen, 2019. Cupellation is found under “Probering” in ch. “Oppredning”.

silver content of the lead plate was measured as c.0.5%. This would have added to the weight of the bead. Consequently, we should have measured weight of the beads with a balance scale with a higher precision than the one used in the experiments. Although the result is slightly uncertain, a more accurate result could have been obtained by repeating the process with longer holding times at 900°C, slower cooling, better cleaning of the bead, and more precise weighing.

The cupellation procedure for the gold alloy was the same as for the silver alloy. However, the cupellation process does not distinguish between silver and gold; it only gives the sum of both. Without cleaning the gold bead and correcting for copper dissolved in the alloy, the result was 778 ‰ Au+Ag compared with 753 ‰ (Au + Ag), as given by the producer of the gold (K.A. Rasmussen, Hamar). The same arguments about uncertainty for the silver alloy apply also to the gold alloy.

We may conclude that the replica bone-ash cupels gave fairly good results when silver and gold alloys with known fineness were tested. However, a skilled assayer might have done better.

The phenomenon of spitting

Spitting is a well-known effect when pure silver solidifies. At melting point, in liquid form pure silver contains 0.32 wt % (2.1 at %) oxygen and solid silver contains 0.006 wt % oxygen (at equilibrium) (see phase diagram in Massalski 1986, p. 49). This means that 0.314 wt % oxygen must leave the melt during solidification. If the melt in the button is sufficiently clean after annealing for 5 minutes at 900°C that it behaves like pure silver, and if the silver bead is cooled quickly after the annealing, the metal may freeze on the surface before all internal melt has solidified. Then, internal gas pressure may build up when melt inside the bead solidifies, causing an eruption from within and through the solid surface of melt. If the solidification is more complete and the cooling slower, the oxygen may leave through the surface in canals of melt and not cause any eruption.

It is known that by adding gold to silver the melt will contain less oxygen. In our case, the gold alloy contained more gold than silver and the surface did not show any eruptions (Fig. 10d). Therefore, in the tested gold alloy, the solubility of oxygen in the melt was low and we did not expect to observe eruptions at the surface under cooling conditions.

Conclusions

Following the guidelines given by Ercker in his book *Treatise on Ores and Assaying* from 1574 (Ercker 1951 [1574]), the procedure for making bone-ash cupels is, in our experience, a straightforward process. Although the book was printed many years later than the operative time of the Mint in the Archbishop's Palace, the knowledge of cupellation is likely to have been common among skilled people.

Unused bone-ash cupels are brittle and not suitable for transportation. However, the moneyer in Nidaros in the period AD 1500–1537 would have had access to the necessary ingredients for making bone-ash, tools, and furnaces on his premises. It is therefore reasonable to assume that the bone-ash cupels found during the archaeological excavations in 1991–1995 in the Archbishop's Palace were made locally at the Mint.

The excavated cupel investigated, N124860, might have been of better quality than our replica cupels, which gave acceptable results when used for testing two alloys with known chemical composition. Thus, the moneyer in Nidaraos might have produced cupels of a high standard and measured the fineness of precious metals with an acceptable degree of precision.

Acknowledgements

We thank Goldsmith Helge Karlgård for the providing of silver and gold alloys and for valuable discussions, the organizers of workshop for making the cupel tools in the Department of Physics, NTNU, Birgitte Karlsen, SINTEF, for metallographic preparation of specimens, and Morten Raanes, NTNU, for the microprobe analysis.

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A METALLURGICAL INVESTIGATION OF THE FORMATION OF SILVER BEADS DURING CUPELLATION OF A 925 ‰ SILVER ALLOY

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Abstract

In the late 15th century and first half of 16th century, the moneyer in the Archbishop's Palace in Nidaros (present-day Trondheim) struck coins for the last three Norwegian archbishops of Nidaros in the period AD 1475–1537. In his profession he would have to master how to make bullion with the correct silver content for the coins. This raises the question of to what degree of accuracy could be achieved in the measurement of the silver content with the use of bone-ash cupels at that time. To address this question the authors made replica bone-ash cupels from scratch and performed cupellation experiments on a silver alloy with known composition. Both the annealing time at 900°C and the cooling rate were varied. The results of the experiments were used to create a model of the metallurgical processes that took place during annealing and cooling. One important result was that the silver bead, which resulted from the cupellation process, was not pure silver once the 'flash' had been observed. However, the authors suggest that with prolonged annealing and slow cooling, the bead may become close to having a content of pure silver, depending upon type of impurities in the silver.

Introduction

The main sources of income of the last three archbishops of Nidaros (present-day Trondheim), Gaute Ivarsson (1475–1510), Erik Valkendorf (1510–1522) and Olav Engelbrektsssson (1523–1537), were tithes, land rents and royal revenues which were collected as *lensherre* (suzerain) in Trøndelag (including land rent, fines, and leidang[1]), as well as trade surplus on trading, especially stockfish from the north of Norway. Almost all income from land rents and tithes were paid in kind, such as corn, malt, meat, fish, and furs (Nissen, 1998). However, most fines were paid in money or silver. To evaluate the value of the precious materials paid as fines it was important to have knowledge and the necessary tools for measuring the quality of precious metals.

In the Halmstad-recess of 1483, the protocol conditioning King Hans' election as king of Norway, one of several concessions was the King's reconfirmation and reinstatement of the privilege of the archbishop of Nidaros to strike coins of the same quality as the coins issued by the King. As a consequence, the last three archbishops had the privilege to issue coins. During the archaeological excavations of the Archbishop's Palace in 1991–1995, three mint complexes were found, one above the other (McLees, 1996; Saunders, 2001; Nordeide, 2003; Lohne et al., 2010). The mints have been documented as having been in operation from about AD 1500–1537 (Olsson, 2000). Among a large number of metalworking debris, 174 used bone-ash cupels and cupel fragments were found (Saunders 2001).[2] The total has since been increased to 186 (Risvaag et al., 2021, this volume). The finds show that fire assay by cupellation was quite common at the Mint in the first half of the 16th century. Therefore, it can be assumed that the moneyer had the ability to make cupels and measure the silver content of the coins produced in the Mint (Lohne et al., 2021, this volume).

Furthermore, we know that in 1532 the archbishop's moneyer in Nidaros, Jacob Schult, was commissioned by King Christian II to assay the silver being used for mountings on St. Halvard's shrine, possibly because the king needed silver to pay his soldiers. In a letter written in Nidaros on 6 November 1532, Jacob Schult presented the results of his assaying as follows: 'The Mark to contain 15 Lod and 6 gren' (our translation) (DN, X No. 640). If we use 1 mark = 16 lots and 1 lot = 18 grains (Ercker (1951 (1574) p.37) it follows that the metal assayed by Jacob Schult was 958 ‰ silver. Thus, the moneyer had the ability to measure the silver content of silver alloys and

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1 The *leidang* (Old Norse: *leiðangr*) was the organization of the coastal defense, which included ships, crews and provision for a certain amount of time. Around AD 1200m the *leidang* was converted into a fixed tax and by the mid-1300s the *leidang* was a fixed tax on properties in both urban and rural areas.

2 Bergstøl, S. and Nordeide, S.W. Bergstøl, S., & S. W. Nordeide. 1993. "Analyses of crucibles from the late medieval mint in the Archbishop's Palace, Trondheim,". Unpublished.

enjoyed a high reputation, given that he was commissioned by the king to carry out the analysis. To what degree of accuracy could the silver content in silver alloys be measured in the medieval times?

The main steps in the cupellation process are described by Lohne et al. (2021, this volume), whose main finding is that the melting of a silver alloy together with about ten times as much lead in a bone-ash cupel may end up with a bead of almost pure silver after annealing at 900°C. The percentage of silver in the starting material can be found by dividing the weight of the final bead, assuming it is pure silver, by the weight of the starting material, and multiplying the sum by 100. However, the surface appearance and internal microstructure of the bead will vary according to the chemistry of the tested material, the annealing time and the cooling rate. We investigated the external shape and internal microstructure of the beads in order to reveal the metallurgical processes that take place during the annealing and cooling, and thus to evaluate the parameters that determine the fineness (i.e. purity) of the silver in the beads.

Experiments

Replica bone-ash cupels (Lohne et al., 2021, this volume) were used in several experiments to investigate the external shape and the internal microstructure of the silver beads after cupellation of a 925 ‰ silver alloy. The material used comprised cut-offs from gating systems after casting silver jewellery in a silversmith's workshop. Fig. 1 shows a pair of replica bone-ash cupels. The outer upper rim diameters are c.35 mm and the bowls have a fine-grained surface layer – 'facing'.

A piece of thin lead plate was folded around pieces of the silver alloy, in which the silver weighed 0.15–0.38 g and the lead weighed about nine times the weight of the silver (i.e. c.1.35–3.42 g). The object was put into a pre-heated cupel, which was placed in a ceramic vessel in a furnace operating at 900°C (Fig. 2a). The annealing temperature was

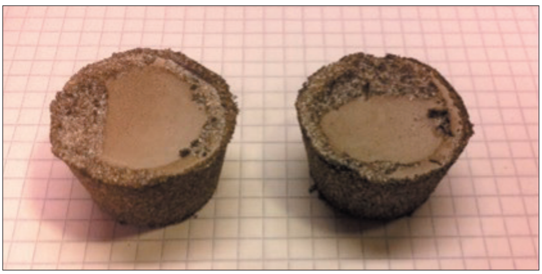

Figure 1. Replica bone-ash cupels. Dimensions of paper squares: 5 × 5 mm (Lohne et al., 2021, this volume)

selected according to the ASTM proposal for gold in bullion in *ASTM E1335 - 08(2017): Standard Test Methods for Determination of Gold in Bullion by Fire Assay Cupellation Analysis* (ASTM International). The effect of annealing at other temperatures than 900°C was not investigated.

Annealing time in the experiments varied from a few minutes to 30 minutes. After annealing, the cupels were cooled at various rates: rapid cooling by taking the vessel with the cupel straight out of the furnace and let it cool down on a table at room temperature or slow cooling by turning off the heating in the furnace and later partially opening the furnace door. The cooling rate of the beads was not recorded. However, the cooling rate of the air when slowly cooled was measured with a thermocouple in the vicinity of the vessel and was of the order of 10°C per minute down to 800°C and thereafter slower until the temperature was the same as room temperature.

Silver beads (Fig. 2b) were removed from the cupels and investigated by using a scanning electron microscope (SEM) equipped with energy dispersive x-ray spectrometer (EDS). Some beads were cast in EpoFix resin and the cast was cut by a saw with a diamond-bound cutting wheel and cut surface was ground and polished. The specimens were carbon coated before being investigated using the SEM and an electron probe microanalyser (EPMA) equipped with wavelength dispersive x-ray-spectrometers (WDS). EDS and WDS were used to measure the chemical compositions

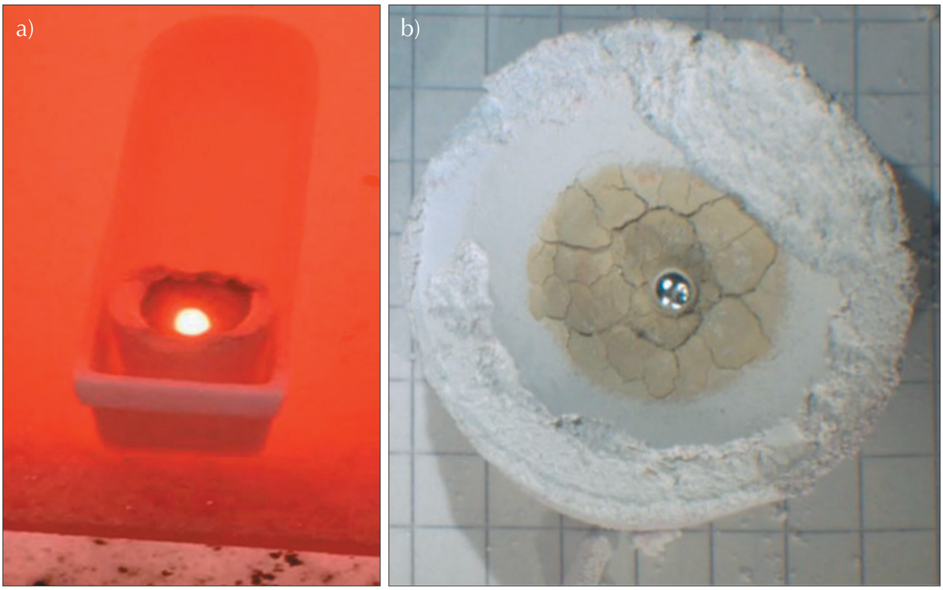

Figure 2. a) The cupel in a ceramic vessel is viewed through a quartz glass window in the furnace door. The yellowish white pool is the melt of lead and silver. b) The cupellation process results in a shiny silver bead at the bottom of the cupel. The cracks in the facing layer appeared during the cooling. Dimensions of paper squares: 5 × 5 mm.

of small areas (e.g. microscopic particles) on the surface of the specimens.

We conducted three tests:

1) Short annealing at 900°C (a few minutes) and rapid cooling (as described above in this section)

2) Short annealing at 900°C (after c.5 minutes, when a 'flash' was observed) and cooling (slower than rapid, but not slow).

3) Long annealing, 30 minutes at 900°C, and slow cooling. The vessel was lightly tapped at the end of the annealing and twice during cooling, as suggested in the ASTM Standard E1335 – 08(2017) (ASTM International). Weight of material at the start of the test: 0.3808 g. Weight of the bead: 0.3486 g (without cleaning off lead oxide). Diameter of bead: c.4 mm.

Long annealing at 900°C and slow cooling: Figures 4–8 show how lead oxide was squeezed out onto the surface at grain boundaries. The lead oxide contained some copper.

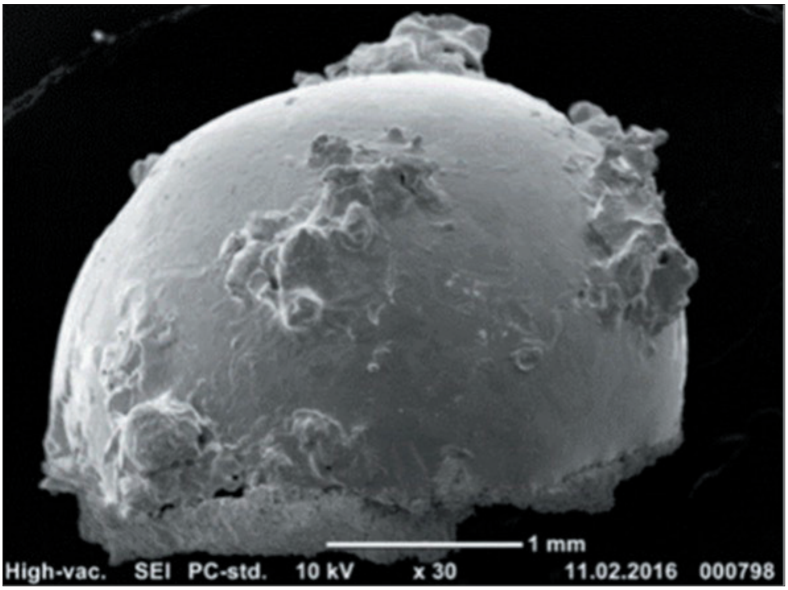

Figure 3. SEM image of the hemispherical bead after annealing for a few minutes at 900°C and rapid cooling. Exhaled melt from the inside of the bead has solidified on the surface. Spitting is a well-known effect when pure silver solidifies, as liquid silver contains much more oxygen than solid silver at equilibrium. Oxygen must therefore be rejected during solidification (Lohne et al., 2021, this volume).

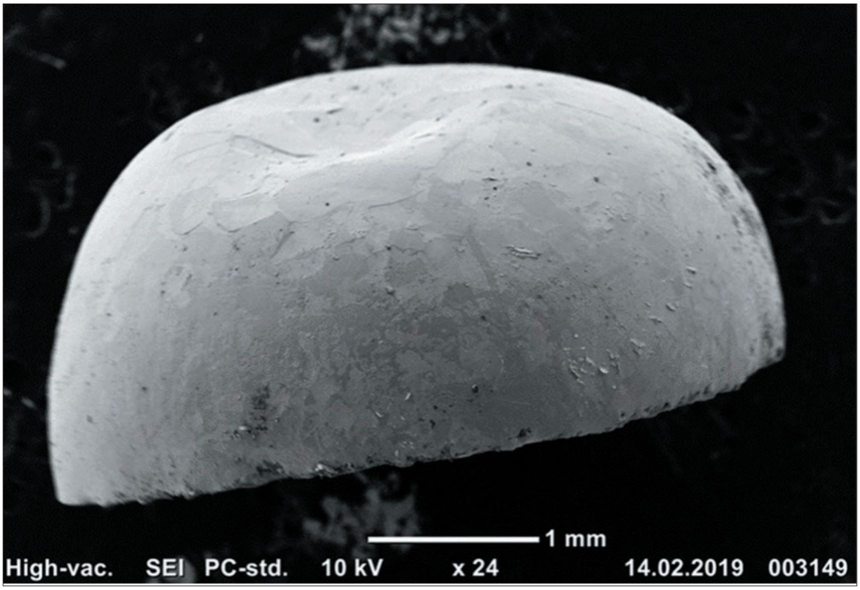

Figure 4. SEM image of the hemispherical silver bead after annealing for 30 minutes at 900°C and slow cooling. No 'volcanoes' are visible but there is a shrinkage cavity at the top. The surface is partially covered with flakes of lead oxide.

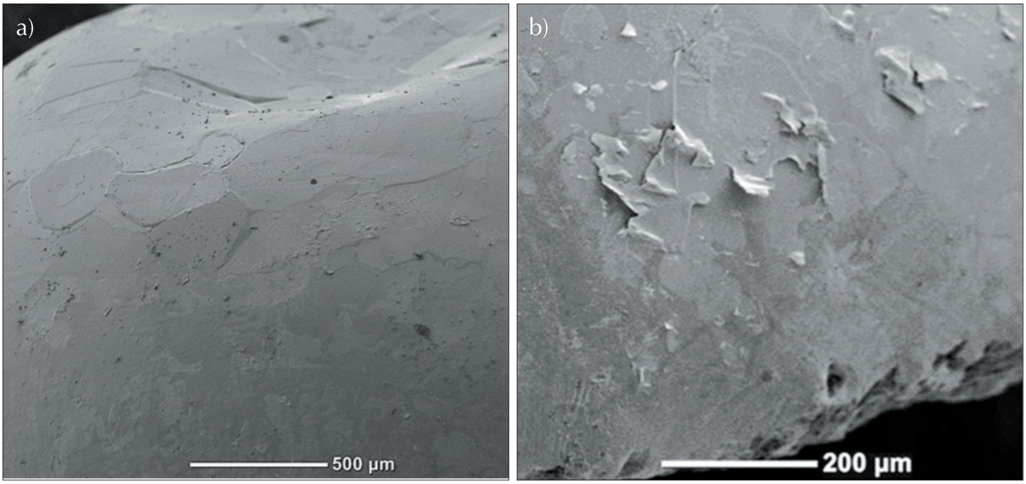

Figure 5. SEM images. a) The top of the bead in Figure 4 at a higher magnification. Lead oxide flakes are clearly visible on the rim of the shrinkage cavity. b) The 'steeper' side of the bead where some lead oxide flakes have burst.

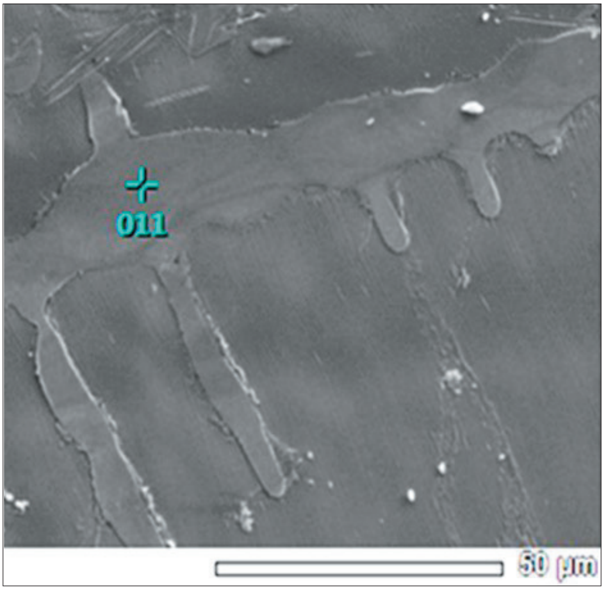

Figure 6. SEM image of the melt flow pattern. Lead oxide has flowed from a boundary between silver grains and solidified. Point 011 marks lead oxide. Beyond the edges of the lead oxide flakes the metal is silver. Scale mark: 50 µm.

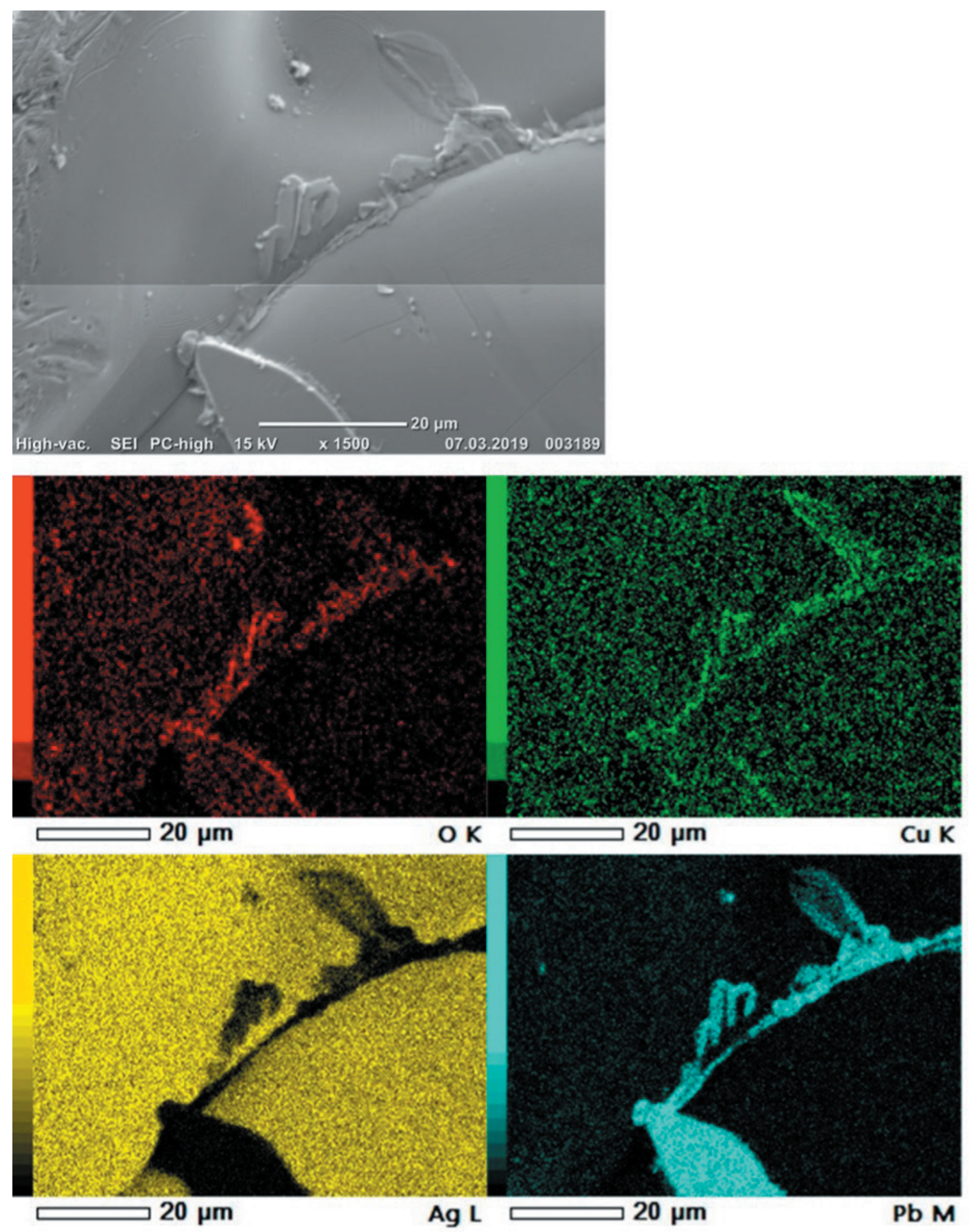

Figure 7. SEM image and EDS maps showing the distribution of oxygen (red), copper (green), silver(yellow), and lead (blue) on the surface. (An electronical fault in the lower part of the SEM image has resulted in a small change in the grey tone). Melt has been squeezed out from boundaries between silver grains.

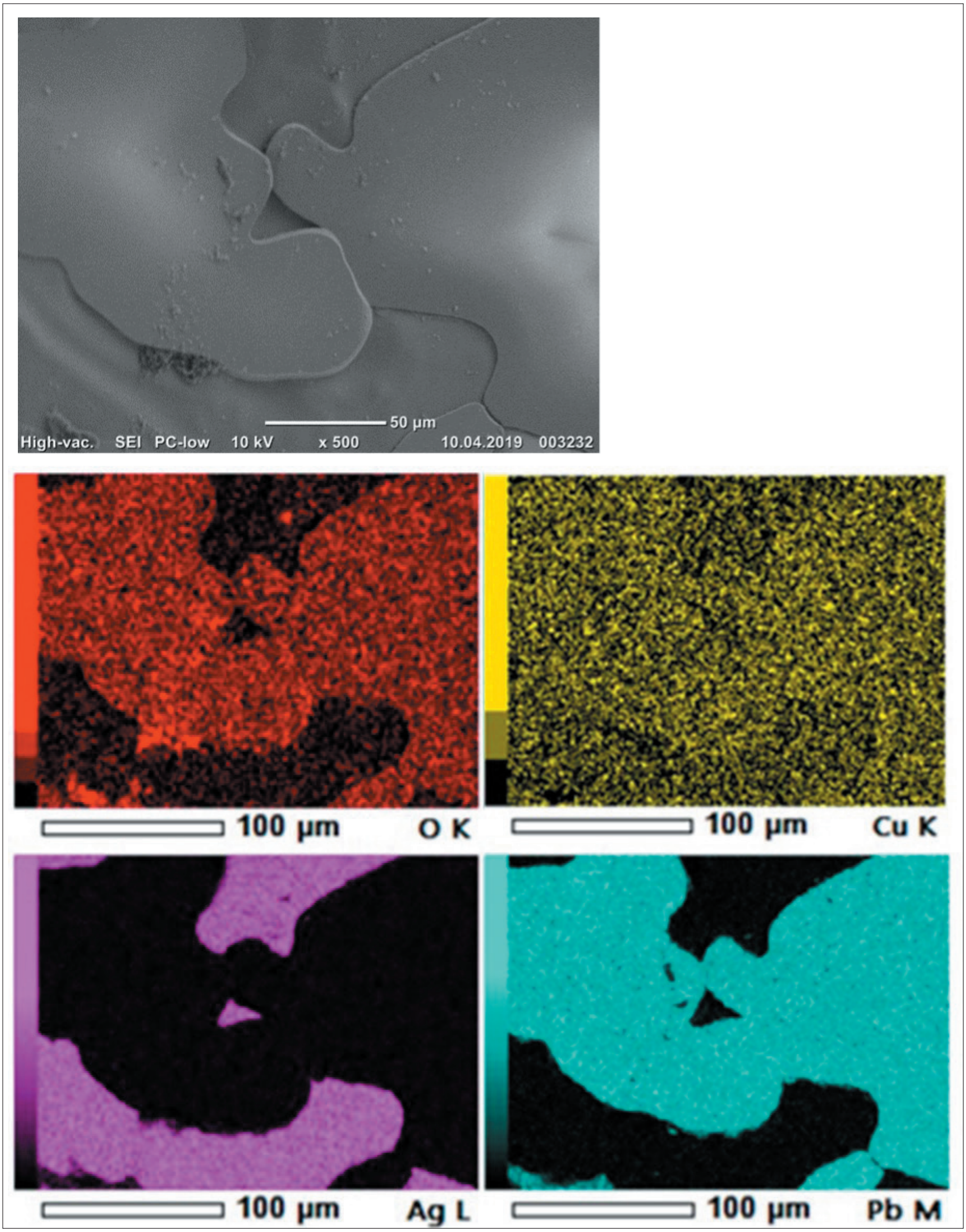

Figure 8. An SEM image and EDS maps showing the lead oxide flakes (red and turquoise), with some copper that has flowed over the silver surface (purple).

Bead shape

After short annealing at 900°C and cooling after an observed flash

The microstructure of the cross section of the bead is shown in Fig. 9.

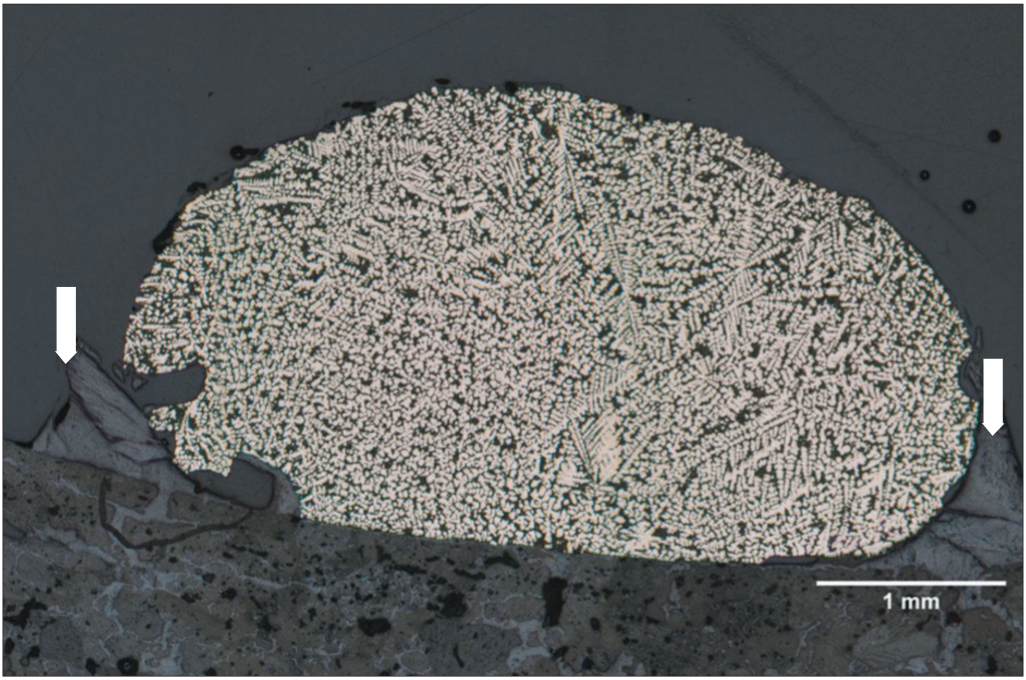

Figure 9. Light microscope (LM) image. The cross-section shows the shape of a bead when the annealing at 900°C was stopped before all lead oxide had soaked into the cupel and then cooled fairly rapidly. Inside the bead, silver dendrites (white) are surrounded by solidified melt of silver, lead and/or lead oxide and copper and/or copper oxide. Outside lead oxide is creeping under the bead, see arrows, and into the cupel (Some small pieces of the bead have been pulled out during preparation).

After long annealing at 900°C and slow cooling

A cross section of the bead after slow cooling is shown in Fig. 10.

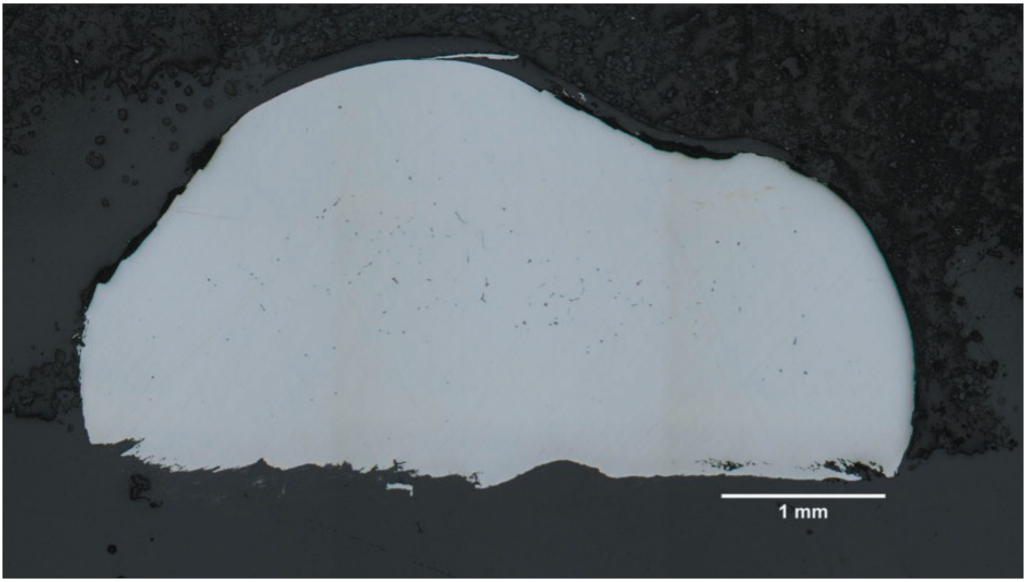

Figure 10. Light microscope (LM) image of a cross-section of the bead after long annealing at 900°C and slow cooling. No dendrites are visible. The faint spots are precipitates of copper, silver and lead (see Figure 14 for details).

The bead shown in Fig. 11 was cast in EpoFix resin and cut. After grinding, a layer of lead oxide spalled off the silver showing that the bonding between the silver and lead oxide was rather weak, thus indicating that molten lead oxide does not wet solid silver very well.

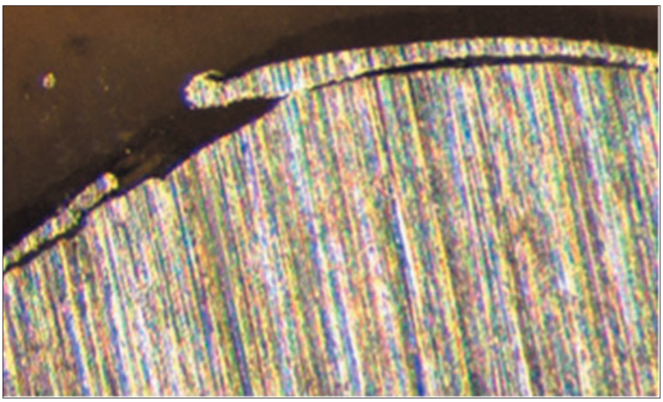

Figure 11. Light microscope (LM) image of a cross-section of a bead annealed for 30 minutes at 900°C and slowly cooled (see Figure 4). A layer of lead oxide loosened from the bead during grinding. Width of original image: 1 mm. Thickness of the loosened lead oxide layer at the surface: 50–100 µm.

Internal structures

After short annealing at 900°C and cooling after an observed flash

Figure 12 shows how lead oxide filled the voids between the silver dendrites. There was no continuous film of lead and/or lead oxide on the surface of the bead.

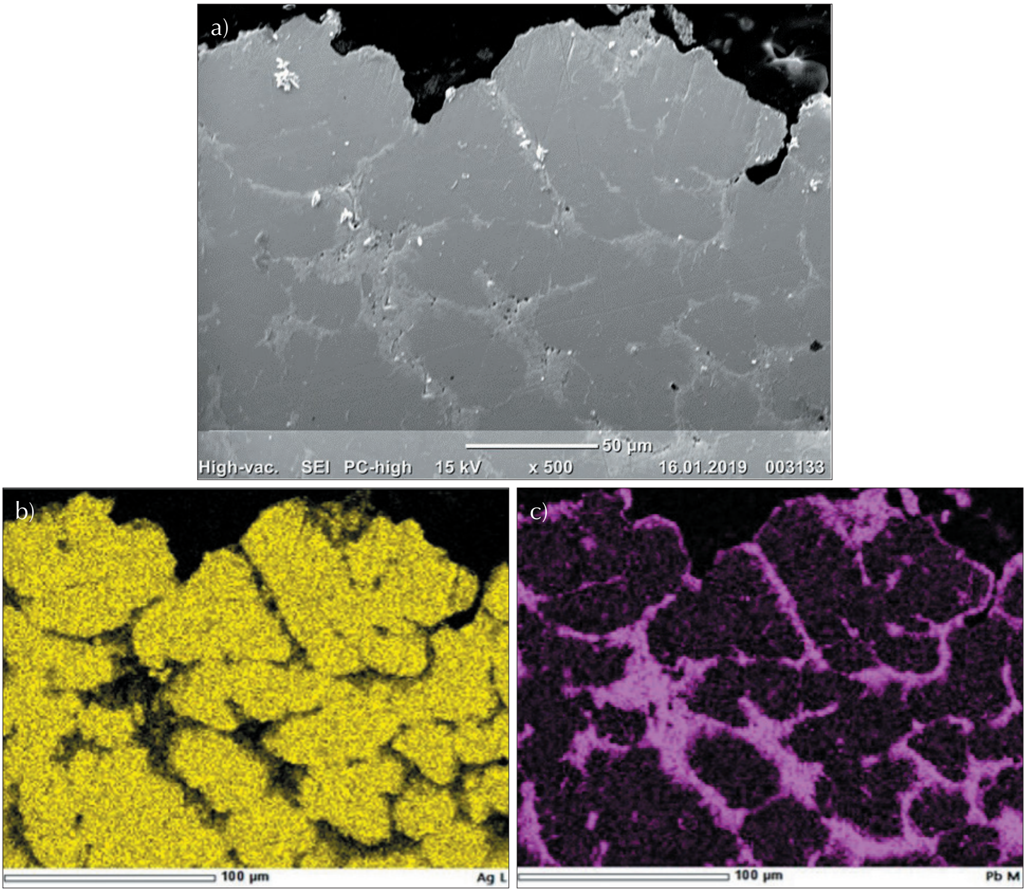

Figure 12. SEM image (a) and two EDS maps (b and c). Cross-section near the upper surface of the bead seen in Figure 9. Lead and/or lead oxide (red) in c) lies in between the dendritic branches of silver (yellow) seen in b). Black spots in a) are pores between the silver dendrites. (Some white spots are dust particles.)

Fig. 13 shows silver dendrites surrounded by lead and/or lead oxide. With the use of an image analysing program, the silver dendrites in Fig. 13 were measured as covering an area fraction of about 67% of the area. The results of chemical analysis performed using a microprobe showed that of the material between the dendrites was 1–2% silver, 98% lead and/or lead oxide, and a small amount of copper and/or copper oxide. This corresponded to the silver content of a pure lead oxide-silver alloy annealed at 900°C

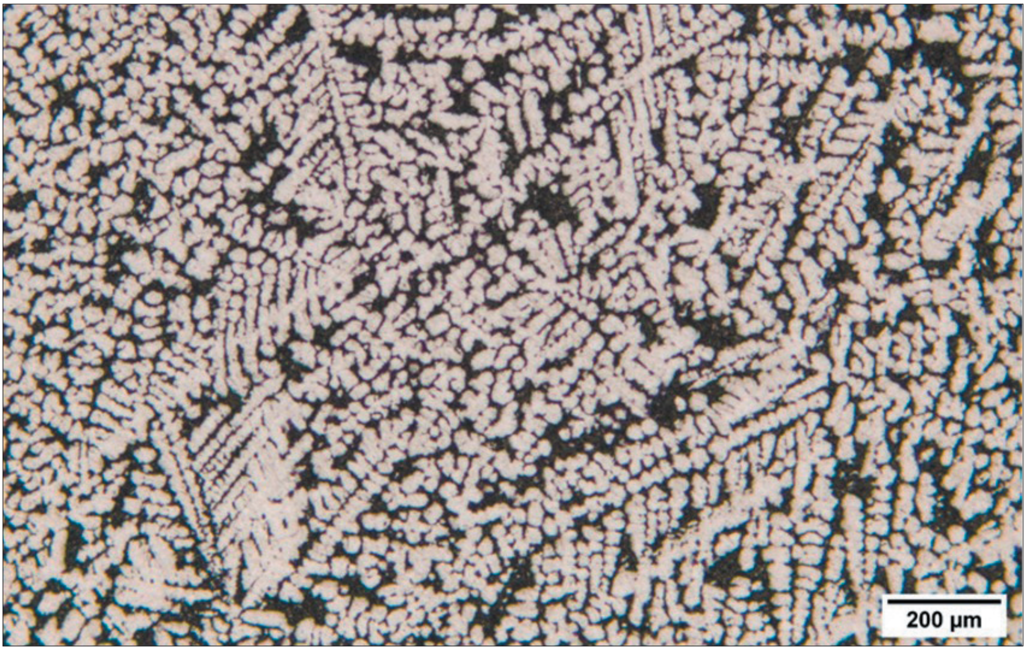

Figure 13. Enlarged light microscope (LM) image of a ground and polished cross-section of an inner part of the bead shown Figure 9. Dendrites of silver (white) are surrounded by solidified melt of lead and/or lead oxide, silver, and copper and/or copper oxide.

(Fig. 18), where 10 mol % corresponded to c. 2.0 wt% silver. The dendrites contained more than 95% silver, and both lead and copper were present. However, when the chemical composition of the dendrites and the material in between the dendrites were analysed, the results were uncertain because it was difficult to avoid soft lead being smeared over the entire surface during grinding and/or mechanical polishing.

After long annealing at 900°C and slow cooling

After annealing for 30 minutes at 900℃ and slow cooling, the microstructure of the bead was multicrystalline. Elongated precipitates were found on the grain boundaries (Fig. 14a). The precipitates consisted of particles of copper, lead and silver. Smaller, spherical shaped precipitates were found in the interior of the grains. The composition of the precipitates was in accordance with known phase diagrams with no intermediate phases in either the binary or ternary systems of silver-copper-lead. Thus, the precipitates, either on grain boundaries or in the silver matrix, were a mixture of copper/copper oxide, silver and lead/lead oxide particles, as shown in Fig. 14b. The area fraction of precipitates was c.2%. From visual inspection of photographs from the outer regions of the bead there appeared a somewhat lower density of precipitates than in the central region. When measured by a microprobe, it was found that the silver content in the interior of the grains was c.99.5% silver and higher outside the particles.

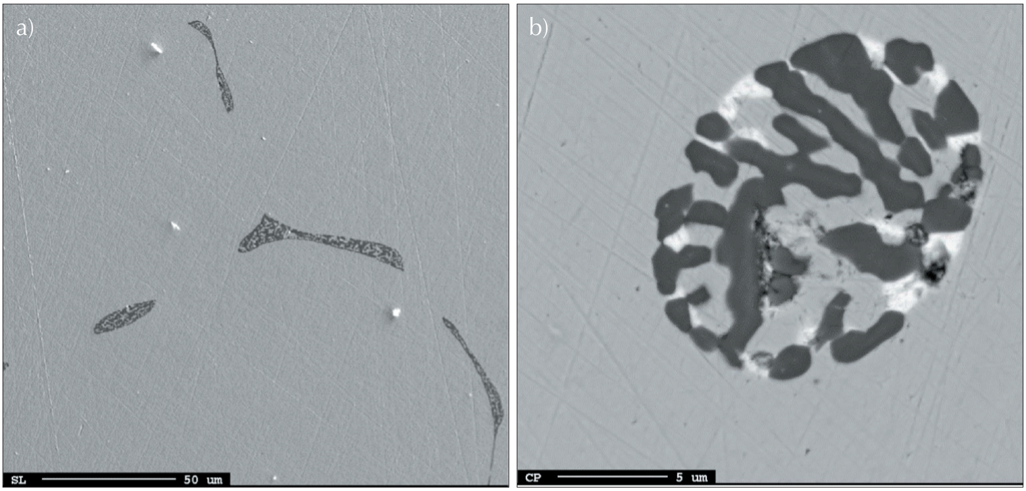

Figure 14. Microprobe image of a ground and polished cross-section of the bead shown in Figure 4. a) Copper-silver-lead particles along grain boundaries (the three white spots are dust particles). b) A precipitate in the interior of a silver grain. Copper (dark grey), lead (white), silver (pale grey), pores (black). Scale marks: 50 µm in a) and 5 µm in b).

Testing the silver alloy used in the experiment

As mentioned at the beginning of the section headed 'Experiment', the material used comprised 925 ‰ silver alloy cut-offs from gating systems from casting silver jewellery. A cross-section of the gating material is shown in Fig. 15. The black spots are evenly distributed precipitates mainly of copper, which show that the alloy used was homogeneous. EDS analysis performed over an area of 2 mm × 2 mm gave a mean result of several measurements (in wt %) for the main elements as follows:

91.5% Ag + 4.6% Cu + 2.4% Zn + 1.0% Pb + 0.2% O

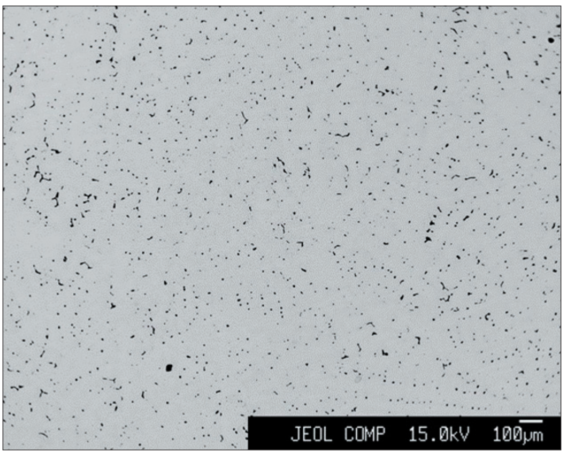

Figure 15. Microprobe image of a cross-section of the starting silver material used for the cupellation experiments. The black spots are precipitates mainly of copper.

Discussion

The cupellation process

In this section we discuss the results of the experiments with reference to a model illustrated in Fig. 16 and further extends the description in more detail in 12 steps. In Fig. 16a the concentration of lead in the melt button has been reduced to c.10% by transforming lead to lead oxide, which is evaporating or being sucked into the cupel wall. Thus, the alloy concentration has passed the liquidus line in the phase diagram of the silver-lead system (Fig. 17). If we disregard the presence of copper and zinc, the melt now consists of c.90% silver and 10% lead. Dendrites of solid silver are nucleated, probably near the surface where the silver concentration may be higher than in the lower part. A liquid of lead oxide covers the surface of the melt button. Molten lead oxide wets the cupel surface and 'creeps' under the button and into the cupel. Dendrites of solid silver are heavier than the melt and will sink.

Fig. 16b shows that more lead has been transformed to lead oxide. Dendrites grow and impinge, and new dendrites are nucleated. Molten lead oxide covers the surface of the button. The melt consists of silver, lead and/or lead oxide, and copper and/or copper oxide. Lead oxide and copper oxide may form in the melt due to oxygen being rejected from the silver during solidification because the concentration of oxygen in liquid silver is higher than in solid silver at equilibrium.

Fig. 16c shows how the dendrites grow together and form grains with melt on some grain boundaries. On the surface, the molten lead oxide has broken, leaving a ring around the bottom of the bead. A shrinkage cavity appears on the top.

Fig. 16d shows the silver when it has solidified, and liquid lead oxide is being squeezed out onto the surface. During cooling the lead oxide may be liquid down to c.825°C (Fig. 18) in a pure mixture of lead oxide and silver. However, due to impurities, in this case copper, the eutectic point may be further lowered, even to 750°C (Ulseth et al., 2015). Lead oxide will be squeezed out and solidify on the surface due to the contraction of silver both during solidification and linear contraction during cooling down. Elements in solid solution in silver may precipitate, preferentially at grain boundaries.

Twelve steps in a model of the cupellation process

Twelve steps in the cupellation process are outlined in the following list, where steps 1–3 represent heating, 4–10 annealing at 900°C, and 11–12 cooling. These steps are based on the phase diagrams in Fig. 17 (Ag-Pb) and Fig. 18 (PbO-Ag), and that the

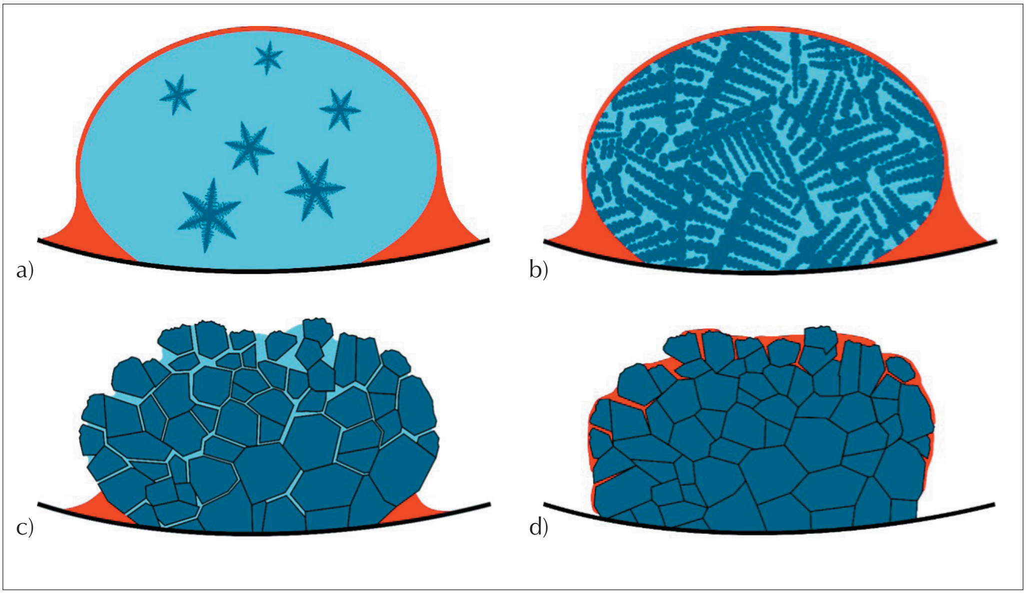

Figure 16. Model of the cupellation process. A melt button is lying at the bottom of the cupel. Red – molten lead oxide (with some copper), dark blue – solid silver, and pale blue – melt of silver, lead and/or lead oxide and copper and/or copper oxide (The flow of lead oxide into the cupel wall is not shown.

concentration of oxygen is much higher in liquid silver (0.32wt%) than in solid silver (0.006wt%) (Massalski, 1986, p. 49) at equilibrium, and that molten lead oxide, but not lead or silver, is wetting the bone-ash cupel:

1) During heating lead melts at 327.5°C. The silver alloy to be investigated is dissolved in the lead melt. The shape of the melt is a flattened sphere covered by solid lead oxide (PbO) on the surface. The chemistry of the melt is c.90% Pb, 9.2% Ag, 0.5% Cu, and 0.2% Zn at the starting of the annealing process (zinc is omitted in the following steps).

2) Just below the annealing temperature, 900°C, PbO melts (Fig. 18). During annealing, lead oxide vapourizes and some is sucked into the wall of the cupel. New lead oxide is formed by reactions between the molten lead and the air above the melt.

3) Copper and other impurities in the silver alloy are dissolved in the molten lead oxide and are either vaporized with the lead oxide or sucked into the wall of the cupel.

4) During annealing at 900°C the amount of lead in the melt will be reduced due to the continuous formation of lead oxide; the same applies to copper, but not silver (Fig. 17). In this way, the melt will be enriched with silver over time.

5) When the concentration of lead in the melt has been lowered to c.10% (the liquidus concentration of Ag-Pb at 900°C is 90% Ag + 10% Pb), solid silver, with c.1.5% of lead and some copper in solid solution, starts to precipitate in the melt as dendrites, probably near to the surface. The volume of the melt has now been reduced by c.90% since the start of annealing.

6) During further annealing, the amount of silver in the melt is reduced (but the concentration is constant) as solid silver dendrites grow (Fig. 9) and new dendrites are formed. The silver dendrites are heavier than the melt and will sink, impinge on each other and grow together. There will be a reduction by volume when silver solidifies by 4–5%.

7) During solidification of silver, oxygen will be rejected and either form pores between the silver dendrites (Fig. 12a) or react with lead to form lead oxide or with copper to form copper oxide (CuO). Thus, between the dendrite arms the melt contains lead and/or lead oxide, some copper and/or copper oxide, and silver.

8) The rejection of oxygen from the silver melt, the transformation of lead into lead oxide (and copper to copper oxide) will increase the volume of the melt, which will then be squeezed out at the surface (Fig. 4-8).

9) As the annealing proceeds, the dendrite arms at the surface 'puncture' the continuous layer of molten lead oxide due to roughness and a reduced area of lead in contact with air (Fig. 12). As the liquid of lead oxide poorly wets solid silver (Fig. 11), the lead oxide layer will, at a certain stage, burst and leave a bright silver surface. This process may be observed as a flash. The liquid lead oxide may contract to form a ring around the bottom of the bead and disappear as it is sucked into the cupel. There may still be melt between the dendrites, which upon contact coalesce and form grains.

10) When the silver in the melt has solidified, the contact area between grains increases and the remaining melt of lead oxide, with some copper and/or copper oxide, is squeezed out onto the surface, slides down (Fig. 5b) and is sucked into the cupel.

11) During cooling from 900°C the lead oxide may still be in a liquid state to temperatures far below 900°C (see Fig. 18, which shows a eutecticum at 825°C or lower due to the presence of impurities, in this case mainly copper). Liquid lead oxide that is still present between grains of silver may be squeezed out due to the contraction of the solid silver and then solidify on the surface of the silver bead.

12) During cooling, impurities in solid solution in silver at 900°C may precipitate. Preferred sites for the precipitation will be grain boundaries and/or grain boundary nodes (Fig.14).

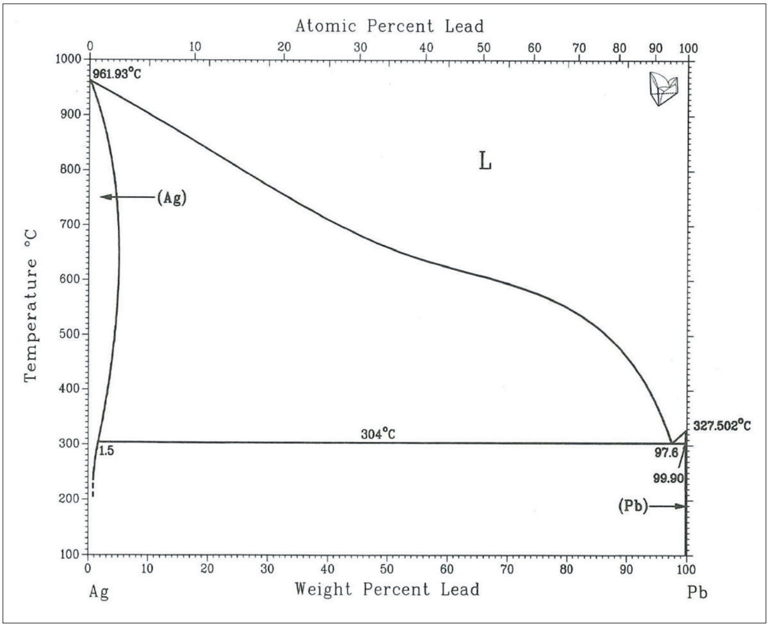

Figure 17. Phase diagram of Silver – Lead (Ag – Pb) (Massalski, 1986, p.53).

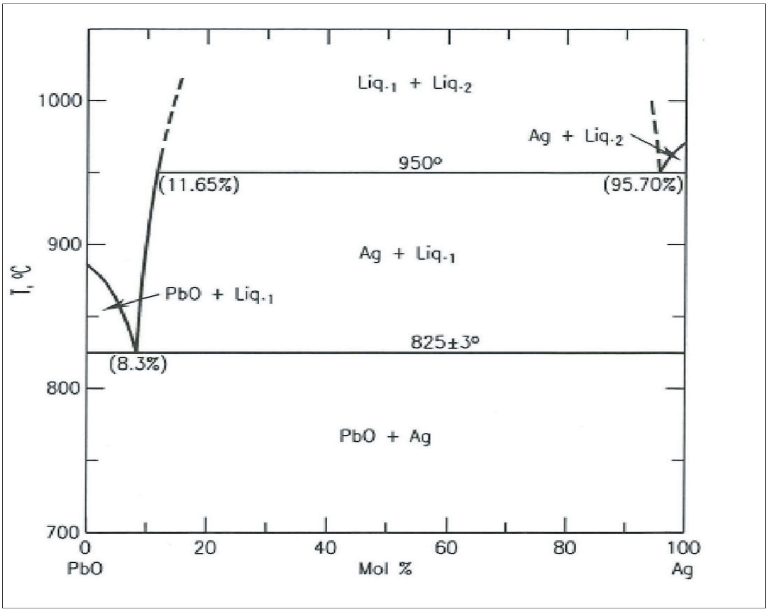

Figure 18. Phase diagram of Lead oxide – Silver (PbO – Ag) (Roth, 2001).

How to clean the bead

With the exception of impurities on the surface, earlier assayers assumed that after brushing the beads were pure silver (Sieblist, 2008; Mongiatti et al., 2009; White, 2010). Ercker (1951 [1574], p. 60)[3] recommended that the beads should be prised from the cupels while still warm:

> *Then they will come cleanly off the cupel facing. If something should stick to the beads, they should be squeezed with pliers or flat-nosed tongs, and the dirt will spall off. Then brush them with a small hard brush made from bristles.*

During our experiments we experienced that material from the facing could stick to the lower side of the bead, but could be brushed away. We also observed that the lead oxide spalled off the silver surface during grinding (Fig. 11). As a consequence, lead oxide on the surface could break away from the surface when the bead was squeezed. Thus, after brushing, the surface of the bead may be quite clean.

The purity of the beads

In addition to melt of lead and/or lead oxide and copper and/or copper oxide being trapped between solid silver grains, impurities in solid solution in silver grains inside the bead will still remain. Correct annealing will keep such impurities to a minimum. When annealed at 900°C' the bead will contain c.1–1.5 w% of lead. At room temperature, copper will be found both as precipitates on grain boundaries and in solid solution in the silver. The concentration of elements in solid solution will depend also on the cooling conditions.

What about other alloying elements that were normally found in silver alloys from the Archbishops Palace? S. Bergstøl and S.W. Nordeide (Cf. Footnote 2) tested five bone-ash cupels by using XRF (X-rays fluorescence) on the cupel surface. In addition to silver, they found lead, copper, and small amounts of zinc (Zn). In one of the cupels they registered some tin (Sn) and in three cupels small amounts of nickel (Ni). Zinc might have been present due to the use of brass (Cu-Zn) instead of pure copper when making bullion for coins produced in this 16th century mint, which normally contained c.70% copper. The presence of zinc reduces the tendency of pore formation during casting and might have been added at will. The zinc would have been in solid solution in the silver. The presence of tin could have resulted

3 See also: *Katalog for Sølvverksmuseet, Kongsberg*, 1942. Handwritten, possibly by a secretary under dictation of the foreman of miners Bjarne Sanness.

from bronze (Cu-Sn) being used instead of copper in bullions for coins. The solid solubility of tin in silver is high (c.10%).

The solid solubility of nickel in silver is low. However, in a binary lead-nickel alloy c.2–3%, nickel may be dissolved in liquid lead at 900°C and at the surface form a high-temperature stable oxide that is not dissolved in lead oxide (Moesta and Franke, 1995, p. 66). Therefore, nickel would not have been sucked into the cupel wall but would rather be found on the surface of the cupel.

A small amount of silver would have been lost to the lead oxide during annealing and ultimately sucked into the cupel or solidified on the surface. However, some silver might have come from the lead material (the lead used contained c.0.5% Ag). Ercker underlined that both the silver lost to the lead oxide and silver gained from the lead had to be taken into account when evaluating the results after cupellation.

Ercker (1951 [1574], p. 60) recommended that 'When the beads […] have just blicked (flashed) clearly, lift the cupels from the furnace and pry the beads out while the crucibles are still hot'. However, as shown in Fig. 13, a substantial amount of lead and/or lead oxide and copper and/or copper oxide may be locked between the silver dendrites if the cooling starts just after a flash. Fig. 13 shows dendrites with a concentration of more than 95% silver covering about 67% of the cross-section. The melt covers about 33% and has a concentration of c.1–2% silver. Together, these results show that the fineness of the bead in the image might have been c.70% silver. If the weight of the bead was supposed to constitute pure silver, a mistake or overestimation of c.30% was made.

Furthermore, Ercker (1951 [1574]) recommended that two cupellations with the same amount of material should be done. Then, if the weight of the resulting beads is equal, the cupellation has been done correctly.

Estimation of fineness in the starting material after long annealing and slow cooling

If, after long annealing and slow cooling, the bead was well cleaned before being weighed the uncertainties of the fineness would have to be estimated from several parameters: How much silver followed the lead oxide into vapour or into the cupel or solidified on the surface of the bead? How much silver was gained from the lead applied in the process? What are the contributions of impurity elements present as precipitates on the grain boundaries or in solid solution? If we assume that silver lost balances silver gained, the effect of precipitates and elements in solid solution may be evaluated as follows: the area fraction of precipitates was measured as c.2%, of which

silver might have constituted c.50% of the precipitates; the purity of the interior of the grains was measured as higher than 99.5% silver. Together, the two evaluations give the fineness of the bead as between 98–99% silver, not 100% (Téreygeol and Thomas, 2003).
Other contributions to the uncertainties of the estimated fineness of the starting material in our case may be calculated as follows:

1) The silver bead after long annealing and slow cooling was not cleaned for lead oxide. Using the weight of the starting material, 0.3808 g, and the final weight, 0.3486 g, without cleaning for lead oxide on the surface, a fineness of 91.5% is obtained, which is the same percentage as found in the EDS analysis of the gating material. However, we know that this is an overestimate because the bead was not clean.

2) If we suppose that a 50 μm thick layer of lead oxide covers c.25% of the bead surface, the weight of the oxide layer will be c.0.005 g. Then, the fineness will be about 90.2% Ag, which is a deviation of 1.3% and an overestimate.

3) If we also correct for precipitates and elements in solid solution and use a value of 98.5% silver in the bead, the estimated fineness of the starting material will be 88.9%, which is a deviation of 2.6%.

The right annealing temperature

The active part in the cupellation process is liquid lead oxide (PbO). In the experiments, the annealing temperature was kept constant at 900°C. However, pure lead oxide (PbO) melts at 888°C (Fig. 18) and may be kept molten at lower temperatures if other elements, such as copper, are absorbed (Ulseth et al., 2015). The phase diagram of silver and lead in Fig. 17 shows that at 900°C solid silver will contain c.1.5% of lead in solid solution at equilibrium. At higher temperatures, the concentration of lead in silver will be lower. The same will apply for copper. However, by increasing the temperature, the loss of silver by evaporation may become a problem. Ercker discussed the problem of regulation of temperature during cupellation of copper containing silver alloys and considered it 'a master's assay'. Essentially, the assaying temperature should not be too high during the start of annealing but increased towards the end of the annealing process. The phase diagram in Fig. 17 shows that if the temperature is raised to the melting temperature of pure silver, 961.93°C, it will be possible to remove lead and copper if done correctly. However, it is not possible for beginners to master such temperature regulations.

Conclusions

1 The processes that take place during cupellation (i.e. heating, annealing and cooling) may be understood as sequences of the metallurgical processes: melting, melt enrichment, dendrite nucleation, dendrite growth and dendrite impingement, internal and surface oxidation, and metal shrinkage.

2 Due to the dendrite and/or grain structure there may be some lead and/or lead oxide locked inside the silver bead. The amount will vary with impurity content, annealing temperature and time, and cooling conditions. Therefore, merely measuring the weight of the bead after the surface has been cleaned well will give results that have some errors. We found an error of about +30% when the cupellation process was terminated just after a flash was observed. The results may be better than 3% or 1–3% after long annealing at 900°C and slow cooling, and even better if the assayer manages to follow Ercker's advice to increase the annealing temperature to the right level at the end of the annealing. Without knowing how the moneyer performed the assaying process in detail, the uncertainties may lie somewhere in between the above given percentages.

Acknowledgements

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