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METALConsn-info

Metal working group

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BROMEC10

Editorial

The number of abstracts in this new issue is rather low, but still, we are happy that some of you managed to contribute even though in the last months you had to provide abstracts both for the next Interim Metal WG meeting in Canberra, and more recently for the next ICOM-CC triennial meeting in the Hague. Again, it shows how active the group is.

This time we have an interesting paper on an ongoing research project performed in Malta that describes a much less invasive technique to study the metallography of iron/steel armours. The second abstract is about copper based gilded medieval altars and the coating that was applied on the top surface. A symposium should soon be organised and the author is interested in any possible contributions. The next two abstracts are about the conservation of tin based materials and the last one is about an innovative electrolytic technique to protect copper, iron, silver and lead based materials with a transparent organic film.

Some of you were wondering whether the results presented in the past ten BROMEC issues had been published or would be soon? I have in fact carried out a survey asking all the past BROMEC contributors whether they had published or would be willing to publish their work. The outcomes of this survey can be found on the Metal homepage of the ICOM-CC website (http://icom-cc.icom.museum/WG/Metals/) as an updated **list of abstracts per topics** where a mention of any paper published or any project of a paper to publish is given.

This survey clearly shows that the BROMEC contributors use the Metal proceedings as a support to their publications. It is clear too that there is the will to publish the work presented in BROMEC. It shows, first, the high quality of that work which is worth publishing and, secondly, that BROMEC achieves its goals in informing the professionals on research projects in progress and gives opportunities for them to contact the contributors, to be involved in and to discuss the results obtained.

After some months, the BROMEC also helps raise awareness of what research is being performed, and by whom, and facilitates the organisation of meetings where research in progress is discussed between professionals active in the field (example of WG COST Action meetings where experts can be invited).

Hoping you will find this new issue as useful and interesting as usual.

Editor
Christian DEGRIGNY
Assistant editor
James CRAWFORD

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Ongoing research projects

Metallographic examination of armour artefacts – an 'in-situ' metallographic observation method (***MCR/ WC***)

Valuable information can be retrieved from a metallographic examination of armour metal. This includes information related to the technology of manufacture (whether any attempts have been made at hardening the metal, whether a high quality bloom was utilized for production purposes, blacksmith skills at forging and forge welding armour plates) and information on the composition of the steel (determining a rough idea of the weight percent carbon in steel). The presence or absence of slag inclusions determines whether an artefact is authentic. Furthermore, the microstructure of modern steel is easily recognizable from that of historic steel.

Unfortunately, a technique capable of observing the microstructure of an armour plate without necessarily interfering 'invasively' with the artefact is not available. For this reason, we usually rely on examination of a small fragment of metal that is extracted from the inner side of the armour. The fragment is prepared into a cross-section by grinding and polishing after a preliminary embedding step. The microstructure of the metal is examined under a metallographic microscope after etching with Nital (2% (v/v) nitric acid in ethanol).

The investigation of the microstructure of armour artefacts can also be performed via the so-called *in-situ* method. This involves embedding part of the armour (a plate edge) into polyester resin to obtain a cast block; grinding the surface of the latter to expose the embedded metal; polishing the metal to a 'mirror surface' finish; etching; and finally observing the microstructure using an inverted metallographic microscope. Dr Alan Williams, an expert on amour metallurgy devised the method. He is currently serving as armour consultant to the Wallace Collection, London, as well as other armour museums around Europe. This method seems to be the preferred one with most curators of armour collections since fragments are not detached from their artefacts.

Requirements for the in-situ procedure include:

- Identification of a suitable plate edge for polishing. The artefact is examined thoroughly beforehand in order to find an edge where the cross-sectional surface can be investigated. Armour artefacts are very complicated geometrical objects and it is not always easy to find a site for embedding. The situation becomes even more complicated when the artefact is highly decorated, for example where the edges are worked into a roped decoration.
- Reversibility of the embedding procedure. When the examination is complete, the resin cast must be easily removed. This is achieved if a polyester resin is used for embedding. Furthermore, any gilding or etching decoration that might end up embedded within the resin must not be damaged upon removal of the cast.
- Having an inverted metallographic microscope capable of carrying the weight of the armour piece under investigation. This microscope set-up is an essential requirement when it comes to investigating embedded edges because it allows you to place the whole artefact onto the microscope. Embedded samples investigated using a conventional metallographic microscope require that the upper and lower surfaces of the object under investigation run in parallel. If this is not the case, the observer will

encounter difficulties whilst focusing. This is not a requirement when it comes to the inverted metallographic microscope since the surface under investigation rests directly on the microscope.

- The *in-situ* method is very convenient for the preparation of samples for metallurgical observation. Furthermore, it is much less destructive than the fragment extraction method. Most armour pieces making up a suit of armour possess an edge that can be examined.

The author could benefit from a Short Term Scientific Mission (STSM) financed by the EU COST Action G8 to be trained in the *in-situ* method under the supervision of Dr Williams. This method will be adopted at the Malta Centre for Restoration with the technical support of the Department of Metallurgy and Materials Engineering, University of Malta to study the microstructure of Armour artefacts from the Palace Armoury.

Contact: Daniel Vella (DSL - MCR)
Funding: COST Action G8 & the Wallace Collection

Ongoing research projects

‘Golden altars’ in Denmark (***NMD / UoC, DACS, AH***)

Remaining in or originating from nine or ten churches in the Scandinavian region there still exist so-called ‘golden altars’ from the 12th and 13th century, most likely made in the western part of medieval Denmark, namely in different workshops in Jutland. Each altar may have consisted not only of a front section (an antependium/antemensale) for the altar table, but also of a low mounted feature at the back of the table, a reredos (retable), most often crowned by a large ‘arch of heaven’. Both antependium/antemensale and retables are made of copper plates, attached to supports of oak planks, and the plates are embossed, engraved, stamped or almost plain, covered by fire gilding, often combined with brown varnish (*vernis brun*). The first dendro-chronological examinations confirm that the oak planks for supports come from the Danish region.

Smaller remnants of copper plates reveal, along with written sources, that many other churches in Scandinavia were embellished with golden altars in the high medieval period. In total, 41 are known to have existed, of these not less than 32 were in medieval Denmark.

The fundamental study on these golden altars was published by Poul Nørlund in 1926 [1]. It is a very thorough art-historical and archaeological investigation, but now, three-quarters of a century later, it has become obvious that this work on the subject is outdated from many points of view. A project aiming for a new corpus work on the altars has consequently been established in Copenhagen as a collaboration between the University and the National Museum. This publication is going to be based on thorough investigations on both the technical and art historical/cultural field.

As will be described below, several technical analyses of different kinds have already been carried out. At the present state of these different investigations, we have decided to arrange a symposium in August 2005 for both conservators/conservation scientists and art historians to discuss *Stand der Forschung* and problems within both fields, and also within their interdisciplinarity. We are therefore keen on hearing from metal specialists who would be interested in such a meeting - especially colleagues investigating the process of gilding and ornamentation of metal plates or working with the possibilities of determining from which mines the metal originates.

Analysis of the copper, gilding and varnish was made using a low vacuum scanning electron microscope with an energy dispersion spectrometer (LV-SEM/EDS). Thirty-six small copper samples for both LV-SEM/EDS and neutron activation analyses were cut from the least noticeable areas of the different pieces, some with gilt surfaces. As expected the LV-SEM/EDS analysis proved the copper to be quite pure. Ten analysis points on each of the 36 samples amounted to an average of 99.07% copper.

Concerning the fire gilding, it has in cross-sections a typical slightly spongy appearance. When analysing this gold by EDS, it mainly contained a minor copper content from the base metal and various amounts of mercury depending on how much of this had evaporated during the heating of the gold amalgam.

Theophilus described the combined technique of applying brown varnish and gilding in his treaties from about the year 1100. There have been several examinations of the method, but they do not fully explain the variations or difficulties in the technique. In the published documentation of the altars by Nørlund and others, several questions arise in connection with the varnish on the Scandinavian pieces. Having the possibility of combining analyses and practical experiments, it was decided to focus our attention on the varnish.

The reredos and antependium/antemensale from Odder that have decorative elements with the brown varnish were chosen for the study and five small varnish samples were excised from the surfaces with a scalpel. Organic analysis was made by Fourier Transform Infrared Spectroscopy (FT-IR) and by Gas Chromatography - Mass Spectrometry (GC/MS). It has convincingly been shown that the varnish from the reredos is made of dried linseed oil. It was impossible to determine the exact nature of the dried oil used on the samples from the antependium/antemesale because beeswax (presumably from later surface maintenance) disturbed the analyses. The assumption that this oil was linseed is reasonable though, since other oils would be from rarer and exotic plants. The organic analysis showed that the raw linseed oil had changed into boiled and dried oil by heating and oxidation on the copper.

Two of the above-mentioned copper samples from the crucifix and reredos originating from the Odder church, were specifically excised where gilding and brown varnish met along an engraved border. An EDS line scan analysis indicated no signs of pigments in the varnish and only traces of copper minerals as well as phosphorous, calcium and chlorine (a minor trace of such elements was to be expected). The varnish colour is therefore a result of the oxidation process of the oil in contact with the copper base.

Both optical microscopy and SEM showed the gilding extending just to the engraved line and that the varnish on the other side also runs into the groove of the engraving. The varnish in the groove proved it had been applied after the engraving had been made, as Theophilus has explained it. These results will be discussed with other examinations. Practical experiments have also been carried out in collaboration with two goldsmiths acquainted with fire gilding on silver.

[1] Nørlund, P.: Gyldne Altre - Jysk Metalkunst fra Valdemarstiden, Copenhagen 1926 - 'English Summary', pp. 221-46; reprint with an appendix by T.E. Christiansen, Aarhus 1968 - 'Appendix - Forty Years', pp. 21*-31*

Contacts: Søren Kaspersen, (UoC), Karen Stemann-Petersen (NMD- DC) and Poul Grinder-Hansen (NMD)
Funding: The Novo Nordic Foundation.

New research project

<u>Monitoring the condition of archaeological artifacts and samples of experimental modern metals</u> (***EH***)

The early Iron Age wooden causeway at Fiskerton, Lincolnshire, UK was discovered in 1981[1]. Associated with the causeway were numerous artefacts in metal, bone and other materials, of which weapons and tools were the most prolific. The site is currently under threat due to soil erosion and also a proposed scheme of land management including flood protection. The latter would involve raising the water levels in the vicinity. Part of the archaeology lies below the water-table and is therefore probably reasonably stable, but some lies in or above the semi-saturated zone. The effects of re-watering these deposits are not known.

The current project is one of several surveys funded by English Heritage to assess the effects of the proposed new land-use on the buried archaeology, and to test monitoring and sampling systems that could be applied elsewhere in the Witham Valley. Within this project, we aim to monitor the ground water conditions and quality before and after raising the water levels and to investigate burial effects on a variety of modern experimental materials.

The ground water is being measured from dipwells for pH, redox potential, temperature, conductivity and chemical composition. In December 2003, experimental modern materials were installed in the ground and these will be analysed at 6 month, 12 month and then yearly intervals by researchers at several different institutions. The metal samples are ferritic wrought iron and pure copper, and these will be investigated at English Heritage Centre for Archaeology, Portsmouth using principally XRD to characterise the corrosion products. Additional analyses will be made on samples of corrosion products from previously excavated archaeological metalwork, to build on earlier studies[2]. The iron samples and selected Iron Age ferrous artifacts will also be investigated by metallography, and by SEM-EDXA if appropriate.

[1] Field, N., and Parker-Pearson, M., *Fiskerton:* Iron Age Timber Causeway with Iron Age and Roman Votive Offerings. 2003. Oxford: Oxbow Books.

[2] Fell, V., and Ward, M., 'Iron sulphides: corrosion products on artifacts from waterlogged deposits', in W. Mourey and L. Robbiola (eds) *Metal 98*, Proc. ICOM Metals Conservation Group Conference, 27–29 May 1998, Draguignan, France, 111–115.

Contact: Vanessa Fell (EH)
Funding: no external funding

New research project

Restoration of a 17th century tin coffin (***NMD***)

During the last six months a royal tin coffin, dating from 1624, from the crypts of Roskilde Cathedral has been restored at the National Museum of Denmark. Even though conservators wish to minimise treatments of such items, total deconstruction and reconstruction would have been the alternative. The tin of the coffin sides had deformed so much that complete collapse was an ever-present threat. The motivation to write about its treatment is to share experience in handling large tin objects and information on constructions and that of its corrosion, as done by Lihl [1]. Apart from the ethical considerations of having to open the coffin, there was the engineering task of handling a tin construction of about 200 kg. The following describes the procedures taken to ensure the best possible conditions for examining the coffin and restoring its straight angled renaissance appearance. Detailed results from the collaborated historical and technical examinations will be published in the near future with the participants mentioned in the acknowledgements.

Over time, large tin and pewter items are known to alter in shape due to their own weight. Pictures from 1907 and 1992 of this renaissance coffin and observations in 2003 showed that the bending sides of the coffin were becoming more pronounced.

The tin coffin's simple shape consisted of a three-sided lid covered with engravings, and, originally, near-vertical sides. There was a cast tin/lead crucifix on the lid and cast profiled bands edging each end of the coffin. Cast lion faces on the vertical sides had held gilt iron-rod carrying handles. Inside was a wooden coffin that had fallen apart. The Danish duke had lived in Bützow, in northern Germany, and his coffin was made in Rostock. It was ordered to be brought from Bützow to Roskilde Cathedral in Denmark by the Danish king, Christian the 4th, in 1642.

The tin has suffered from a combination of deterioration factors. Besides the strain of its own weight, the deterioration in the lower parts had been caused by damp and cold conditions – presumably mostly in its first years in a crypt in Bützow. Crater-like holes are therefore a part of the damage. On the tin of the lid the expanding formation of tin oxide (because of dust settling there) was causing the destruction of some parts of the engraving.

To restore the bending sides of the tin coffin and support it afterwards, the coffin had to be opened. The areas with fragile tin oxide layers on the lid were consolidated with surplus amounts of Paraloid B-72 in acetone. A steel support was built as a framework fastened around the top part of the coffin. The coffin was held partly in this framework while the tin bottom was cut off at the side-edges. Before this step, observations of the insides had been made with flashlights and an endoscope. The lid of the deteriorated inner wooden coffin covered the deceased and lay half way down the tin coffin. After some preparations the coffin top, consisting of lid and sides, was lifted off. At the department's workshop an inner support was built of stainless steel and honeycomb boards. The sides of the coffin were carefully straightened.

Hallmarks on the tin have been identified and traced to a workshop in Rostock. The tin contained about 5% lead. Examination showed that the 4 mm thick sheets of tin had been soldered together and built up on top of the wooden inner coffin. The preliminary examination of the grave has been published in a Danish journal[2].

[1] Lihl, F.: On the cause of tin decay in the sarcophagi of the "Kapuzinergruft". Studies in Conservation, Vol. 7, No. 3, 1962, p. 89-105.
[2] Kruse, A. and Skals, I. article in Nyt, Nationalmuseet, Copenhagen, in press.

Acknowledgments
This work is being done in close collaboration with the curators Anette Kruse, Roskilde Cathedral Museum, Roskilde, Jørgen Hein, Royal Collections Rosenborg Castle, Copenhagen and Henrik Vensild, National Museum of Denmark. Participants, also from the museum, include the conservators Ernst Jørgensen and Irene Skals and senior researcher Jens Glastrup. The architect firm, Erik Møllers Tegnestue AS, is responsible for the work for Roskilde Cathedral's administration.

Contacts: Karen Stemann-Petersen and Peter Henrichsen (NMD)
Funding/client: Administration of Roskilde Cathedral

New research project

<u>Pewter objects in decorative arts – their deterioration and restoration</u> (***SABK***)

Deterioration of pewter objects, their causes and possible conservation and restoration treatments are investigated in this diploma thesis at the State Academy of Art and Design Stuttgart (SABK). Analyses of the composition of corrosion products and alloys were carried out on 16 pewter objects from the 17th to 19th century. Only 2 of 12 objects show a higher lead content than tin. On the others, only tin corrosion products are found. The corrosion phenomena are explained by atmospheric corrosion.

The conservation/restoration section of the thesis discusses cleaning, joining, and filling of pewter objects. Common cleaning methods are evaluated according to conservation considerations like the loss or changing of original material. Cleaning with tap water or soft cloth can be recommended unreservedly, but all other cleaning methods (mechanical, electrochemical, chemical) do affect the object in varying degrees. To find a suitable adhesive for the soft tin alloys, six resins (acrylic and epoxy resins) are compared in terms of adhesive strength. The results show with statistical significance that a PMMA/MMA 2-component-acrylic resin is the strongest adhesive. Fillings can be made by epoxy resin mixed with tin powder. Corrosion tests of tin alloys with synthetic resins, as well as natural and synthetic waxes, give no visible reaction except with natural waxes. Coatings are not necessary if pewter objects are stored in a dry environment, protected against dust and at a moderate temperature. One important exception is with tin-lead alloys since the lead component reacts readily with acidic or sulphurous vapours emitted by storage or showcase materials. These findings are verified with our own experiments on alloy samples.

Contact: Britta Schmutzler (SABK)
Funding: no external funds

New research project

Protection of copper based and common metal artwork by electropolymerisation of acrylic monomers. (***USIB, UoL, StGPP, MLLN***)

Waxes, cellulosic or acrylic varnishes with added benzotriazole corrosion inhibitor are common compounds used to protect metal artefacts. Due to their low adherence, these films quite easily lose their protective effect when temperature and relative humidity fluctuate. Furthermore, thick layers (20 to 25µm) are applied and then modify the metallic appearance of artefacts.

In this project we propose an innovative electrochemical treatment where the object to be coated is polarized cathodically and immersed in a solution of:

- an organic solvent, acetonitrile (handled with specific precautions) or dimethlyformamide (handled with specific precautions)
- a conductive-salt, tetraethylammonium perchlorate
- an acrylic monomer that is going to give the polymeric protection layer

The potential is given versus a pseudo-reference electrode. A counter electrode is also necessary to receive the current flow. The cathodic potential is scanned progressively by voltammetry until the polymerisation potential is reached, which is then kept constant for a few seconds. Under the applied potential, the monomer reacts with the surface to be coated: it is chemisorbed through an organometallic bond (grafting), which simultaneously initiates the chemical polymerisation.

The maximum thickness of the electrografted coating is around 100nm. The grafting of norbornenyl-ethyl acrylate (hydrophobic monomer) gives totally waterproof coatings ($CuSO_4/H_2SO_4$ test) with a strong adhesion energy above 2400N/cm (ASTM test). Polyethyl acrylate, which has a low glass transition temperature is an elastomeric coating able to fill in accidental scratches of the metal without any additional treatment.

The grafted films are not sensitive to cycles of the temperature or humidity. The low thickness of the layers allows keeping the visual appearance of the original surface. Furthermore the treatment is reversible.

Copper, copper alloys, iron, lead, silver can be protected. The treatment of brass spoons from the 18th century shows the practical significance of this process.

This work was performed in collaboration with the University of Liege, St Gobain Performance Plastics (Petit-Rechain, Liège) and the Museum of Louvain-la Neuve[1]

[1] Guillaume, J., Martinot L., Gabriel S., Jerôme C., Weber G., Jerôme, Mertens M. and Duychaerts E., Protection d'oeuvres d'art en alliages de cuivre et de métaux usuels par électropolymérisation de monomers acryliques, « Les polymères », actes du colloque Art et Chimie, Paris, 2003, 87-90

Contact: Joseph Guillaume (ISIB)
Funding: "Politique scientifique Fédérale" within the "Pôles d'attraction Universitaires", "PAI V/03" and the National Funds for Scientific Research (FNRS)

General information

Websites

- Cost Action G8: Non destructive analysis and testing of museum objects. http://srs.dl.ac.uk/arch/cost-g8. Abstracts and booklets from previous workshops can be downloaded.

- Cost Action G7: Artwork conservation by laser
http://alpha1.infim.ro/cost

- Working Group Metals ICOM Committee for Conservation
http://icom-cc.icom.museum/WG/Metals/

- **CAMEO**: website containing chemical, physical, visual, and analytical information on over 10,000 historic and contemporary materials used in the conservation, preservation, and production of artistic, architectural, and archaeological materials
http://www.mfa.org/_cameo/frontend/

- IR and Raman for cultural heritage
http://www.irug.org/default.asp

Future seminars and conference

- Workshop "Art and Electrochemistry" (07 May 2004, Carré des Sciences, Paris, France) organised by the French Society of Chemistry, Electrochemistry Group. For more information contact André Savall (savall@chimie.ups-tlse.fr)
- The Group **Pre-industrial Metallurgical Arts and Techniques**. **Study and Conservation** organise two conferences at the Musées Royaux d'Art et d'Histoire, Brussels : «*Les bénitiers et fonts baptismaux médiévaux en fer en Lorraine*» at 11am and «*Un phénomène de la période de la fin de l'Age du Bronze et du début de l'Age du Fer dans l'Ouest européen: les haches à douille armoricaines» at 12.30am* on the 26 May 2003. For more information contact Monique de Ruette (m.de.ruette@kmkg-mrah.be)
- The Metals Conservation Summer Institute (7-18 June 2004, Worcester Mass, USA), organised by the Higgins Armory Museum and the Metals Processing Institute at WPI, Worcester Mass. USA. For more information consult www.wpi.edu/+mcsi or contact Josephine Jacobs (jjacobs@higgins.org)
- Philosophy, ethics, history and mechanics of cleaning, 2004 American Institute for Conservation (AIC) annual meeting (09-14 June 2004, Portland, Oregon, USA). Session organised by the Object Speciality Group within AIC. For more information contact Alice Paterakis (alicepaterakis@yahoo.com)
- Ancient and Historic Metals: Technology, Microstructure, and Corrosion. (July 5-16, 2004, UCLA Summer Institute, Los Angeles, USA). Course organised by Prof David A. Scott, Chair. Apply on-line at http://www.summer.ucla.edu/institutes
- Workshop on "Heritage Conservation/Corrosion of Archaeological Objects" (14 September 2004, Nice, France) within the EUROCORR 2004 congress (12-16 September 2004). Organised by Philippe Dillmann, CEA & Gérard Béranger, UTC. Papers presented at this workshop will be published in the Proceedings of EUROCORR 2004 (CD-ROM). Abstracts should be submitted electronically to CEFRACOR via: www.scifrance.org/congres/eurocorr2004. Submission deadline January 16, 2004.

- **Workshop "Bigstuff: Care of large technology object"** (29 September – 1 October 2004, Canberra, Australia), organised by the Australian War Memorial. For more information contact Alison Wain (alison.wain@awm.gov.au)
- Metal 2004, Interim meeting of the ICOM-CC Metal Working Group (04-08 October 2004, Canberra, Australia). The meeting will take place at the National Museum of Australia. For more information consult http://rsc.anu.edu.au/~hallam/metals2004.html
- **Material Issues in Art and Archaeology VII** (29 November – 03 December 2004, Hynes Convention Centre and Sheraton Boston Hotel, Boston MA), organised by the Materials Research Society. For more information consult www.mrs.org/meetings/fall2004/
- Conservation and Management of Oudoor Bronze Monuments (2-4 December 2004, Genoa, Italy). Organised by ISMAR, ICR, SPSADL, SRBACL and CG. For more information contact Paola Letardi (paola.letardi@ismar.cnr.it) or visit the following website: http://www.bmc2004.org
- 2nd Congress Latinoamerican on metal conservation (25-28 July 2005, Rio de Janeiro, Brazil), organised by the Museum of Astronomy and Sciences (MAST) and the Latinoamerican Group of Metal Conservation (GLRM). For more information contact Marcus Granato (marcus@mast.br) or Johanna M. Theile (jtheile@abello.dic.uchile.cl)

Abbreviations and acronyms

EH: English Heritage
DSL: Diagnostic Science Laboratories
EDS or EDX(A): Energy Dispersive Spectroscopy
FTIR: Fourier Transformed Infrared
GC-MS: Gas Chromatography - Mass Spectrometry
ISIB: Institut Supérieur Industriel de Bruxelles
MCR: Malta Centre for Restoration
MLLN: Musée de Louvain-la-Neuve
MND - DC: Museum National of Denmark, Department of Conservation
SABKS: Staatliche Akademie der Bildenden Künste Stuttgart
StGPP: Saint-Gobain Performance Plastics
SEM: Scanning Electron Microscope
STSM: Short Term Scientific Mission
XRD: X-ray Diffraction
UoC : University of Copenhagen
UoL: University of Liege

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