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METALConsn-info

Metal working group

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BROMEC13

Editorial

The Metal WG is going through some major changes in its organisation. To help the coordinator and assistant coordinator in their tasks a new group of close collaborators has been set up. These collaborators, called **national representatives of the Metal WG coordinator** will play the role of the previous correspondents of the BROMEC, but will have more responsibilities on a national level.

Most of the previous national correspondents of the BROMEC have accepted to extend their role, while others preferred to pass their duties to a colleague. This is the case for Alena Silhova, from the Czech Republic and Birgit Wilster Hansen from Norway. They have been respectively substituted by Dusan Perlik and Douwtje van der Meulen.

Currently there is no longer a national representative for Brazil, but countries like Finland and Egypt now have one (respectively Eero Ehanti and Wafaa Anwar Mohamed). France has lost one national representative, Jean-Bernard Memet, and the USA has gained one, John Scott.

We want to thank the colleagues who as national correspondents of the BROMEC have helped us so much to establish this bulletin in its early days. We also welcome the newcomers hoping that they will be active in contributing to this important communication tool.

All national representatives of the Metal WG coordinator are, or will soon be either ICOM-CC voting members or friends of ICOM-CC. In addition to the BROMEC, their role will also be to assist you with any query you have about ICOM-CC and the activities of the Metal WG.

In this issue we have some contributions from students at the Netherlands Institute for Cultural Heritage (ICN), Amsterdam who are in their final year and who are presenting their dissertation project. Students from both the Academy of Fine Art, Antwerpen and the ICN are regularly using the BROMEC to present their research projects. It is hoped that other training schools begin to do the same in the near future. These abstracts are often the only communal trace we keep from dissertation works. They nonetheless give an indication of the topics of interest on metal conservation in the Netherlands and Belgium.

Two French-Chilean and French-Indian cooperation research projects are also presented. They show the importance of international collaboration in the study and conservation of metal artefacts.

As usual, we hope that you will find this issue as useful and as interesting as ever.

Editor
Christian DEGRIGNY
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Ongoing research projects

 Chronopotentiometry of archaeological coins (***CD-FA, CU***)

The Supreme Council of Antiquities excavated a large number of Greco-roman and Ptolemaic coins in North Sinai in 1995. The burial environment greatly affected the mineralisation processes of copper-based archaeological coins. Furthermore, disfiguration of the shapes and details of the coins due to the formation of thick corrosion layers were observed. The preliminary analysis of the corrosion products of three coins indicated the presence of libethinite (basic copper II phosphate) as the essential major corrosion product, atacamite and malachite. The composition of the alloys will be analysed using energy dispersive spectroscopy (EDS). Electrochemical treatments will be applied to reduce corrosion products back to metals and to uncover minute details of the coins. Chronopotentiometry monitoring will be applied during the treatment (recording of the potential of the specimen versus time). This method was theoretically proposed in the past [1]. The research aims to assess the applicability of the method to measure the amounts of different corrosion products found on the object during reduction treatment. The reduction apparatus consists of a battery, a variable resistor, a micro-amperometer, a potential measuring and recording device, a saturated calomel electrode and the reduction cell.

1. Kruger, J., 1977, Some Brief Remarks on Electrochemical Reduction, Corrosion and Metal Artifacts, NBS SP 479, (1977) 59-65.

Contact: Wafaa Anwar Mohamed (CD-FA, CU)
Funding: no external funding

Ongoing research projects

The armours of the Palace Armoury under synchrotron investigation (***DSL-MCR***)

This project is part of a larger research study of the steel armours exhibited at the Palace Armoury (PA), Valletta that was presented in BROMEC 6. These armours are corroding within our harsh Maltese atmosphere and need regular conservation work to maintain them in an acceptable display state. In collaboration with Heritage Malta, a small team of researchers from the Malta Centre for Restoration (MCR) and the Department of Metallurgy and Materials Engineering (DMME - University of Malta) is currently working on a project, which aims to devise new ways of protecting these artefacts in an attempt to minimise conservation intervention.

However, no conservation treatment can be applied without first conducting a thorough study of the metal constituting the artefacts [1] and a study of the nature of the corrosion layers covering them [2]. This project entailed microscopically extracting samples of corrosion products from the corroded surface, followed by analysis using highly specialised x-ray equipment. Since the x-ray equipment is not available on the island the research group had to collaborate with specialised laboratories abroad in order to perform the work.

The group obtained a grant from the European Community - Research Infrastructure Action under the Sixth Framework "Structuring the European Research Area" Programme (through the Integrated Infrastructure Initiative “Integrating Activity on Synchrotron and Free Electron Laser Science”) allowing the possibility of performing x-ray micro-diffraction under synchrotron radiation at Daresbury Laboratory (Warrington, UK). This very powerful technique permits very fast retrieval of information on the crystalline structure of rusts obtained from the corrosion layers. During this preliminary experiment micro-samples from a selection of artefacts at the PA were investigated. A second mission planned in March should be an occasion to study a larger number of artefacts. This low-interventive sampling and non-destructive investigation of the collection should contribute greatly to the understanding of the corrosion processes developing on the artefacts, as well as help in defining a proper protective application for these objects.

This experiment has been made possible thanks to the financial support of the Council for the Central Laboratory of the Research Councils and the technical help of Dr Manolis Pantos of Daresbury Laboratory.

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2. Vella, D., Degrigny, C. and Grech, M. 2005, A preliminary study of the morphology of corrosion developing on historic steel armour artefacts from the Palace Armoury Collection, Valletta, Malta, in Proceedings of the 14th triennial meeting of ICOM-CC, The Hague (submitted for evaluation).

Contacts: Christian Degrigny and Daniel Vella (DSL-MCR)
Funding: EC-Research Infrastructure Action through the CCLRC

Ongoing research projects

 /  Atmospheric corrosion : study of long-term behaviour of iron through the analysis of archaeological artefacts. Part1: Role of phosphorus in the corrosion mechanisms (***LADIR / IIT-Kanpur***)

In the context of the corrosion of Indian irons a hypothesis has been proposed for their long-term behaviour. Indeed iron pieces like the Delhi iron pillar are famous for their high resistance to atmospheric corrosion. Balasubramaniam [1-6] explains these particular properties by the presence of phosphorous in the metallic core that drives the formation of passivating iron phosphates.

In order to validate this hypothesis, electrochemical experiments were conducted under the guidance of Prof. Balasubramaniam during a post-doctoral research project performed at the Indian Institute of Technology of Kanpur (India). Several samples of different phosphorous amounts (0.1 to 0.6 wt%) and one of iron were tested in a 0.1 M Na_2SO_4 solution. It is assumed that this neutral solution is not aggressive towards the corrosion layer formed during the experiment. Different tests have been conducted (potentiodynamic polarisation, impedance spectroscopy) on the samples immersed during three weeks. It seems that after this duration, the behaviour of iron and P-irons is similar: the polarisation resistance of these systems are of a few kOhms. No specific influence of phosphorous on the corrosion resistance is evidenced. The corrosion layer formed on these samples will be characterised to determine its composition and structure at the LADIR (UMR 7075, Thiais, France) under a CNRS post-doctoral fellowship. Furthermore an in-situ test to observe the evolution of the corrosion products formed on phosphorous irons by Raman spectroscopy on longer periods will be established.

A piece of iron sampled on the Bhavani Shankar cannon exposed to outdoor atmospheres in Jhansi fort (Madya Pradesh) has been analysed. The metallic core contains phosphorous heterogeneously distributed in the metal but no phosphates have been evidenced in the corrosion products analysed by Raman spectroscopy [7]. The detected phases were goethite (α-FeOOH), maghemite (γ-Fe_2O_3) and lepidocrocite (γ-FeOOH).

1. R. Balasubramaniam and A.V.R. Kumar, Corrosion Science **42** (2000) 2085-2101.
2. R. Balasubramaniam, Corrosion Science **42** (2000) 2103-2129.
3. R. Balasubramaniam and P. Dillmann. Corrosion resistance of the Delhi iron pillar - scale characterisation and passive film growth models. in Prediction of long term corrosion behaviour in Nuclear Wastes System, Cadarache, France, EFC series, 36, 2003.
4. R. Balasubramaniam, Current Science **82** (2002) 1357-1365.
5. R. Balasubramaniam, Delhi Iron Pillar - New Insights, ed. Aryan Book International, New Dehli, 2002
6. R. Balasubramaniam and A.V.R. Kumar, Corrosion science **45** (2003) 2451-2465.
7. D. Neff and R. Balasubramaniam, Journal of History of Science (accepted)

Acknowledgments

Special thanks to Paolo Piccardo (Metallurgy laboratory, Chemical department, Genova University, Italy) for providing the phosphorous samples.

Contacts: Delphine Neff (LADIR) and R. Balasubramaniam (IIT-Kanpur)

Funding: French Foreign Office through the EGIDE association

Ongoing research projects

Atmospheric corrosion : study of long term behaviour of iron through the analysis of archaeological artefacts. Part 2: Atmospheric corrosion mechanisms (***LADIR / LRC DSM / LRMH / LAE / LECA***)

For several years, archaeological iron artefacts have been studied as analogues for long term atmospheric corrosion [1-4]. This provides useful physico-chemical data on the structure and composition of ancient corrosion layers that can be used in both of the fields of nuclear waste storage management and preservation of cultural heritage. The first aim is to build mathematical models based on empirical data for the designing of iron nuclear waste storage containers and the forecasting of their long-term. The second aim uses simple tools to determine the deterioration degree of the metallic elements used to build the structures of ancient monuments. For both contexts it is required to understand the corrosion mechanisms and the deterioration phenomenon of iron in the atmosphere.

Stratmann [5] based his hypothesis of mechanisms on the reactivity of the lepidocrocite (γ-FeOOH). During the wet-dry cycles, which correspond to the variation of the electrolyte thickness, that occur in the atmospheric corrosion process, lepidocrocite is reduced in a conductive phase. During the wet stage, the lepidocrocite reduction reaction is the cathodic reaction of the corrosion process. Therefore, when the thickness of the electrolyte is at its maximum (during the wet stage), this conductive phase increases the cathodic surface inside the corrosion layer thereby permitting the delocalisation of the O_2 reduction. After the dried period, the reduced lepidocrocite is reoxidised and a new cycle can start again.

The validation of this model is the topic of a CNRS post-doctoral fellowship at the LADIR (UMR 7075, Thiais, France). First, it is important to characterize ancient corrosion layers (morphology, composition, structure). Therefore a corpus of archaeological samples excavated from nine dated indoor sites has been gathered. Samples are embedded in epoxy resin and cut in order to observe them on cross sections. The structure and composition of the corrosion layers are carefully characterised by EDX analysis, microRaman spectroscopy and X-ray microdiffraction [6-8]. The presence of goethite (α-FeOOH), lepidocrocite (γ-FeOOH) and maghemite (γ-Fe_2O_3) have been confirmed on the first samples studied. Moreover goethite is in most of the cases in contact with the metal at the metal/corrosion products interface. The fact that this phase is not conductive questions Stratmann's mechanisms hypothesis. As it is established that some components of the corrosion layer are reduced during the wet-dry cycles [5], it is now important to understand the reactivity of the different phases observed in the corrosion layers, their electrical conductivity and their adherence to the metal core. This will be studied through both the characterisation of archaeological artefacts and electrochemical tests on standard synthetic powders.

1. P. Dillmann, F. Mazaudier and S. Hoerle, *Corrosion Science* **46** (2004) 1401-1429.
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3. P. Dillmann, V. Vigneau, F. Mazaudier, C. Blanc and S. Hoerle. *Rust characterisation of ancient ferrous artefact exposed to indoor atmospheric corrosion*. in *Prediction of long term corrosion behaviour in Nuclear Wastes System*. 2001. Cadarache, France: European Federation of Corrosion.
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6. D. Neff, S. Reguer, L. Bellot-Gurlet, P. Dillmann and R. Bertholon, *Structural characterization of corrosion products on archaeological iron. An integrated analytical approach to establish corrosion forms*, in *Journal of Raman Spectrometry*. 2004. p. 739-745.

7. D. Neff, P. Dillmann, L. Bellot-Gurlet and G. Béranger, *Corrosion Science* **47** (2005) 515-535.
8. D. Neff, *Apport des analogues archéologiques à l'estimation des vitesses moyennes et à l'étude des mécanismes de corrosion à très long terme des aciers non alliés dans les sols*, in *Sciences Mécaniques pour l'Ingénieur*. 2003, Université de Technologie de Compiègne: Compiègne. p. 360. (Text on line: http://www-drecam.cea.fr/lps/index.htm)

Contacts: Delphine Neff and L. Bellot-Gurlet (LADIR), P. Dillmann (LRC-DSM) and F. Mirambet (LRMH)
Funding: French Ministry of Culture and Communication

New research projects

French-Chilean cooperation project for the conservation of sculptures in Santiago de Chile ***(INP, LRMH, CMN)***

Within an extensive programme of sculpture conservation in Santiago de Chile city centre, two conservators who have just graduated from the Conservation Department, Institut National du Patrimoine (INP), Paris, Ms Anne-Marie Geffroy, metal section and Ms Pascale Mauny, sculpture section, have setup research projects specific to the alteration of this group of sculptures.

The aim of this project is to establish conservation-based exchanges between French and South American institutions.

This project should start at the end of 2005 or the beginning of 2006 with the study of overpaint, its removal and the application of anti-graffiti coatings. To date, the French partners are the Research Laboratory of Historic Monuments (LRMH) and the INP. In Chile, the project is supported by the Council of National Monuments (Consejo de Monumentos Nacionales).

Contact: Anne-Marie Geffroy
Funding: Grant of the Carnot Foundation

New research projects

The influence of lead on the embrittlement of silver alloys ***(ICN)***

This project deals with the formation of cracks on silver plaques. Bennekom ascribed these cracks to the lead content in the silver alloy [1]. Scott however, published data on silver alloy containing 4% Pb having no structural problems [2]. As a consequence the influence of lead on the embrittlement of silver copper alloy is still unclear.

In this study several questions will be addressed. For example:

i) where did the lead in the silver alloy come from?
ii) what extent can the silver be deformed as a function of lead content?

1. J. Bennekom, Fine silver plaquettes made by the Flemish silversmith mathias Melin, Artmatters 1 (2002) 21-31

2. D. Scott, Technological, analytical and microstructural studies of a renaissance silver basin, Archeomaterials (1991) 21-45

Contacts: Daan Brouwer, Robert van Langh, Bart Ankersmit and Bill Wei (ICN)
Funding: ICN

New research projects

The introduction of bioreduction in conservation: the removal of iron corrosion with bacteria (***ICN***)

Current and past methods intended for the removal of iron corrosion on historic artefacts often coincide with affecting the original metal surface. Furthermore the treatment method might not be practical to perform.

Bacteria are not yet used for the removal of iron corrosion on museum objects, whereas several industrial applications already exist. With these organisms, we can make specific use of their metabolism of certain metals or metal ions. This application causes a change in valence of the metal (ion).

This research investigates the possibilities for the use of *Shewanella Putrefaciens*, *Geobacter Metallireducens* and *Shewanella Oneidenensis*, to transform insoluble iron corrosion products (found on historic artefacts) into soluble products, without damaging the original metal surface.

Contacts: Eveline Los, Robert van Langh and Bart Ankersmit (ICN)
Funding: ICN

New research projects

The influence of different cleaning agents and cleaning methods on the tarnishing rate of silver (***ICN***)

The aim of this dissertation project is to investigate the influence of tarnish removal treatments on the tarnishing rate after treatment. Parameters that will be taken into account include: rinsing and subsequent drying of the silver sheets after cleaning. The tarnished surface after treatment will not only be studied with time, but also the morphology of the formed layer will be a decisive parameter for evaluation. The overall goal of this project is to identify the best cleaning treatment for silver that has experienced a slow (re)tarnishing rate and has an evenly distributed tarnish layer.

Contacts: Wandalin van den Abeele, Robert van Langh and Bart Ankersmit (ICN)
Funding: ICN

New research projects

Patina management: the conservation and preservation of restored patina of outdoor bronze sculpture (***ICN***)

Bronze sculptures in outdoor environments are, through their way of presentation, often prone to damage. Vandalism and unintentional damage inflicted by viewers, is often reported. Another example of damage is treatments applied in the past. There are of course treatments that are no longer used because of their far-reaching consequences.

The result of these types of damage is that the appearance of the patina of the object is disturbed. Not only it is aesthetically unwanted, but also, the bare metal becomes extra sensitive to decay.

To date there is no treatment that simultaneously restores the patina aesthetically and also provides protection in the outdoor environment for an acceptable amount of time with good conditions for normal maintenance.

The aim of this project is to determine if it is possible to develop a colour coating system, which meets these requirements. The minimum requirement for maintenance is one annual treatment.

Objects from the Hepworth collection at the Kröller-Müller Museum in Otterlo, Holland all show problems concerning the conservation and management of their rich-coloured patina. As a pilot project, the sculpture “single form” or “Ikon” (Dame Barbara Hepworth 1966) will be treated.

At the same time a test series will be established to determine long-term protection and preservation of the colour coating system.

Contact: Vera Bakker, Robert van Langh and Bart Ankersmit (ICN)
Funding: ICN

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 as well as announcements of future activities (Short Term Scientific Missions deadlines, training schools…).

- 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

- LabS-TECH network
http://www.chm.unipg.it/chimgen/LabS-TECH.html

- ARTECH network
http://server.icvbc.cnr.it/progetti_futuri/progetto_artech.htm

Future seminars and conference

- Developing Technical Tendering Specifications for Large Technology and Cultural Objects (16 March 2005, London, UK), organized by the UKIC Metals Section at the Science Museum, London. For further details contact Suzanne Kitto (suzanne.kitto@armouries.org.uk).

- GMPCA (Groupe des Méthodes Pluridisciplinaires Contribuant à l'Archéologie) seminar (19-22 April 2005, Saclay, France) organised by the Pierre Süe Laboratory, CEA Saclay will take place at INSTN, Saclay 91. For more information contact Mrs Anne Morel (GMPCA2005@iscsa.cnrs.fr) and visit the following website : http://www.ladir.cnrs.fr/GMPCA2005/.

- Metallurgy – a touchstone for cross-cultural interaction (28-30 April 2005, London, UK), a conference organised by the British Museum to celebrate Paul Craddock's contribution to the study of metal through the ages. For more information contact slaniece@thebristishmuseum.ac.uk.

- ART'05 conference (15-19 May 2005, Lecce, Italy), 8th International Conference on "Non-Destructive Testing and Microanalysis for the Diagnostics and Conservation of the Cultural and Environmental Heritage organised by the Italian Society for Non-Destructive Testing Monitoring Diagnostics (AIPnD), the Central Institute of Restoration (ICR) and the Dept of Materials Science of the University of Lecce. For more information visit the following website: http://www.dsm.unile.it/art05.

-Metals Conservation Summer Institute 2005 (May 29 – June 9 2005, Worcester, Massachusetts, USA) organised by the Higgins Armory Museum, the Metal Processing Institute and the Worcester Polytechnic Institute. The Metals Conservation Summer Institute is funded in part by a major grant from the Institute of Museum and Library Services, a federal agency that fosters innovation, leadership and a lifetime of learning. For more information visit our website www.wpi.edu/+mcsi.
- 2nd Congress Latino-American 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)
- 14th Triennial Meeting of ICOM's Committee for Conservation, ICOM-CC (10-16 September 2005, The Hague, Netherlands) organised by the ICN (Netherlands Institute for Cultural Heritage in collaboration with ICOM, ICOM-Netherlands; ICOM-CC, the Netherlands Museum Association (NWV), the Dutch association of professional Conservators and Restorers (VeRes) and the Congress and Study Centre (VNG). For more information visit the following website: http://www.icom-cc2005.org
- LACONA VI (Lasers in the conservation of Artworks) (21-25 September 2005, Vienna, Austria), 6th International Congress on the Conservation of artworks by laser, organised by the Federal Office for Care and Protection of Monuments Austria-Bundesdenkmalamt and the Academy of Fine Arts Vienna. For more information visit the following website: www.lacona6.at.

Abbreviations and acronyms

CCLRC: Council for the Central Laboratory of the Research Councils
CD-FA, CU : Conservation Department – Faculty of Archaelogy, Cairo University
CMN : Consejo de Monumentos Nacionales
CNRS: Centre National de la Recherche Scientifique
DMME : Department of Metallurgy and Materials Engineering
DSL-MCR : Diagnostic Science Laboratories – Malta Centre for Restoration
EC: European Commission
EDS : Energy Dispersive Spectroscopy
ICN: Netherlands Institute for Cultural Heritage
IIT –Kanpur: Indian Institute of Technology of Kanpur
INP: Institut National du Patrimoine
LADIR : Laboratoire de Dynamique Interaction et Réactivités
LAE : Laboratoire d'Analyse et d'Environnement - CNRS/Evry University, UMR 8587, France
LECA : Laboratoire de l'Etude de la Corrosion Aqueuse, French Nuclear Agency, France
LRC DSM 01-27 : Laboratoire Métallurgies et Cultures ULR5060 CNRS et Laboratoire Pierre Süe, CEA/CNRS, bat 637, 911191 Gif/Yvette cedex
LRMH: Laboratoire de Recherche des Monuments Historiques

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