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METAL*Consn*-info

Metal working group

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Editorial

This new issue of BROMEC is rather short. METAL07 is taking all our energy!

In this issue you will find the presentation of an interesting multidisciplinary research project, ODéFA setup by French laboratories to understand why some iron archaeological artefacts remain active after their stabilisation treatments. Two stabilisation techniques are considered here: the immersion in heated NaOH solutions and cathodic polarisation in KOH.

The recent development of the eCell tool to monitor in-situ electrochemical processes while analysing metal surfaces at Synchrotron facilities continues to produce some very important information on the behaviour of chlorinated copper based alloys while being stabilised in sodium sesquicarbonate solutions. More recent studies concerning the protection of lead artefacts with sodium carboxylate are presented in this issue.

The Music Museum in Paris is initiating some research on manufacturing techniques applied specifically to materials of their collections. Two projects on piano strings and plating covering Sax instruments are also presented in this issue.

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

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Ongoing research projects

Programme ODéFA : Optimisation de la déchloruration des ferreux archéologiques (***Arc'Antique / SOLEIL / IRAMAT / LPS/ LEMMA / LRMH / LCRR-CAV***)

Stabilisation treatments of iron archaeological artefacts are usually considered as efficient although the mechanisms of extraction are still not well understood. In the long term these artefacts often show active corrosion due to residual chlorides still inserted in the material structure. A better knowledge of the mechanisms involved during the treatment of the artefacts would not only help in defining the end of the stabilisation process but would contribute to the optimisation of the treatment parameters so as to reduce its duration.

To better appreciate the whole problematic, several French laboratories have joined forces within the ODéFA project funded under the national research projects on the knowledge and conservation of cultural heritage materials (PNRC) supported by the Ministry of Culture. The multidisciplinary team is comprised of specialists in the conservation of archaeological objects (Arc'Antique, LCRR-CAV and LRMH) and researchers specialised in the understanding of corrosion mechanisms of iron artefacts through the study of their corrosion products (LEMMA, LPS (CEA) and SOLEIL). The good practice and ethics of conservation professionals is completed then by the knowledge and up-to-date analytical techniques available in research laboratories.

Within this project, two categories of samples are considered:

- terrestrial iron archaeological artefacts (nails from Glinet site (76));
- marine iron archaeological artefacts (ingots from the Saintes-Maries-de-la-Mer site (13) provided by Luc Long (DRASSM).

These artefacts are currently treated according to the protocols established at LCRR-CAV (immersion in NaOH at 50°C) and Arc'Antique laboratory (cathodic polarisation in KOH). They are characterised before, during and after treatment at Arc'Antique, LEMMA, LPS and SOLEIL with SEM-EDS, micro-XRD and Raman microspectroscopy.

The first results show that the nails from Glinet site only contain a small amount of chlorides. Therefore they are not suitable to understand properly the chlorides extraction. The first studies carried out in this project concentrated then on the marine artefacts. The monitoring of the chloride extraction in treatment solutions of the ingots shows high concentration of chlorides. The analytical results have permitted to identify the different corrosion products and to localise them. Chlorides are initially localised in the internal corrosion layer mainly as (β-$Fe_2(OH)_3Cl$) and in a smaller proportion as akaganéite (β-FeOOH) mixed with magnetite (Fe_3O_4). Chlorides are not found in the marine crust. After 15 days of cathodic polarisation chlorides are only detected in localised areas (zones of a few micrometres) at the interface between the metal and the internal corrosion layer or in corrosion products formed along the inclusions of the metal structure. At the same time the extraction curves still give high amounts of chlorides. During these electrolytic processes β-FeOOH and β-$Fe_2(OH_3Cl)$) are transformed in goethite, magnetite and iron oxyhydroxydes that are poorly crystallised. Analyses are still in progress to understand the evolution of corrosion products both during the cathodic polarisation in KOH and immersion in NaOH at 50°C.

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Funding: French Ministry of Culture

Ongoing research projects

 /  In-situ electrochemical and SR-XRD time resolved study of lead carboxylate coating for the protection of cultural heritage artefacts (***DP-UW / DAC-GU***)

European lead heritage ranges from simple ornaments to vast artistic and engineering masterpieces such as pipe organs that are all in danger of decay and loss through corrosion. Indeed, lead organ pipes are amongst the most seriously jeopardised artefacts: their sound is critically dependent on the material, as well as its shape, size, and condition [1]. Lead is seriously affected by the presence of organic acids (formic, acetic...) in the environment, and both original and restored wood (e.g. in the wind chest) are a source of these.

Coating lead artefacts to protect them is fraught with difficulty; badly researched coatings can lead to irreversible changes in appearance and function, and even more rapid degradation. Only after meticulous investigation can a coating be applied with relative confidence. An attractive feature of the carboxylate coating process is simplicity. The lead is soaked in a non-toxic solution of sodium decanoate [2]. The result is a dark coating, similar to aged lead in appearance. In this study, we set out to measure the growth and protective characteristics of such layers using time resolved X-ray diffraction (XRD) [3], electrochemical impedance spectroscopy (EIS), scanning electron microscopy (SEM) and mass gain measurements.

An innovative environmental cell (the eCell [4]), developed especially for the characterisation of rough heterogeneous metal surfaces and the characterisation of conservation methods using synchrotron-based techniques was used. The cell can be used for electrochemistry (i.e. in a fluid electrolyte) and also for exposure to environments such as acetic acid vapour, both with simultaneous spectroscopy measurements of the lead surface using X-ray techniques.

Initial results [3,5] obtained at the European Synchrotron Radiation Facility, show that the growth of the carboxylate coating can be monitored in real time using SR-XRD, in parallel with EIS and OCP measurements. The first two techniques give quantitative information on the coating quality, whilst the third may be useful for monitoring coating deposition in a simple way appropriate for use in conservation laboratories and on-site. Studies of high level acetic acid exposure (~3% w/w acetic acid/air at 70% RH) have lead to an improved method for producing the sodium decanoate solution which results in more corrosion resistant coatings.

References:

[1] For more information see the website of the FP5 project COLLAPSE at www.goart.gu.se/collapse/
[2] E. Rocca, C. Rapin and F. Mirambet, Inhibition treatment of the corrosion of lead artefacts in atmospheric conditions and by acetic acid vapour: use of sodium decanoate, Corrosion Science 46 (2004) 653-665
[3] www.esrf.eu/files/Highlights/HL2006.pdf (pages 75-76)
[4] M. G. Dowsett and A. Adriaens, Cell for Simultaneous Synchrotron Radiation X-ray diffraction and electrochemical corrosion measurements on cultural heritage metals and other materials, Analytical Chemistry 78 (2006) 3360-3365
[5] To be presented at Metal 07 (Amsterdam, the Netherlands, September 2007), and to appear in the proceedings

Contacts: Mark Dowsett (DP-UW), Annemie Adriaens & Bart Schotte (DAC-GU)
Funding: A new version of the eCell with innovative analytical capability (X-ray excited optical luminescence) will be developed with support from the Paul Instrument Fund (Royal Society, UK), and Fonds voor Wetenschappelijk Onderzoek (FWO) (Belgium). The work will be further supported by an ESRF long-term project in collaboration with Robert van Langh (Rijksmuseum, Amsterdam) and Carl Johan Bergsten (GOArt, Göthenburg)

Ongoing research projects

 /  In-situ time resolved monitoring of copper corrosion using an automated electrochemical cell (***DAC-GU / DP-UW***)

As with many other metals, copper corrodes once it comes into contact with an aggressive environment. Corroded archaeological cupreous artefacts recovered from wet saline environments, in particular, are very susceptible to further corrosion. The objects are usually stored in tap water or a sodium sesquicarbonate solution to remove the chlorides. Nevertheless, this treatment often results in instability of the artefacts, such as the chemical transformation of the natural patina and the development of active corrosion. The occurrence of these side effects means that monitoring of the treatments remains necessary.

Here the authors report on an ongoing study of the basic processes involved using artificially corroded copper coupons that feature various corrosion products, soaked in sodium sesquicarbonate (BROMEC 4). The study was carried out using an automatic electrochemical cell (eCell) designed for time resolved synchrotron spectroelectrochemistry.

The cell consists of three main modules and is based on its predecessor which has been used in a number of cultural heritage projects [1-3]: the hardware, a custom interface and potentiostat, and a data system written in Visual Basic™ 2005. In addition it consists of a webcam that images the surface of the working electrode (sample) and is used extensively for setting up and progress checking. The set-up was tested for the first time at station MPW 6.2 (Daresbury, UK). In operation, the data system is programmed to raise and lower the working electrode periodically (typically every few minutes). The latter is ultimately important to be able to collect the electrochemical data in a mode where the reaction takes place in the bulk of the electrolyte (i.e. allowing the optimization of transport processes in the electrochemical cell), and to raise the working electrode to the cell window for a short time for analysis. Otherwise, the restricted fluid volume between the working electrode and the window distorts the reactions.

The new design was tested by means of a corrosion monitoring experiment which involved the simultaneous acquisition of XRD data and corrosion potential (Ecorr) values from a copper substrate covered with a layer of nantokite (CuCl), which was immersed in a 1wt% sodium sesquicarbonate solution, and this as a function of time. Results have demonstrated that nantokite disappears as a function of time, while the cuprite signal increases. When it comes to the Ecorr data, a steady increase is seen for the first hour. Ecorr values provide useful information on the surface reactivity as they are based on the composition of the sample and its interface with the solution. The variation of these values as a function of time therefore confirms the change in surface chemistry.

References:

[1] K. Leyssens, A. Adriaens, M. Dowsett, B. Schotte, I. Oloff, E. Pantos, A. Bell and S. Thompson, Simultaneous In-situ time Resolved SR-XRD and Corrosion Potential Analyses to Monitor the Corrosion on Copper, Electrochemistry Communications 7 (2005) 1265-1270

[2] M. Dowsett, A. Adriaens, Cell for Simultaneous Synchrotron Radiation X-ray and Electrochemical Corrosion Measurements on Cultural Heritage Metals and Other Materials, Analytical Chemistry 78(10) (2006) 3360-3365

[3] A. Adriaens, M. Dowsett, K. Leyssens, B. Van Gasse, Insights into electrolytic stabilization with weak polarization as treatment for archaeological copper objects, Analytical Bioanalytical Chemistry 387(3) (2007) 861

Contacts: Annemie Adriaens (DAC-GU) and Mark Dowsett (DP-UW)
Funding: no external funding

New research projects

 Characterization of plating materials and techniques used in instruments of Sax factory (***Conservare - IRRAP***)

The conservation of a cultural object implies a deep comprehension of the details concerning its nature. Gathering information about its constituting materials and the employed manufacture processes is of fundamental importance for choosing the adequate conservation conditions as well as the most suitable restoration treatments. Regarding brass musical instruments, they often present a surface finish consisting in a more or less thin layer of a 'white' metal, usually silver or nickel. Whatever the reason for such practice, aesthetical or protective, the close contact between different metals represents an additional challenge for the conservation of the instruments.

Aiming to gain insights in technological aspects related to plating procedures used in brass instruments factory in the late 19th century, a comprehensive study is carried out.
It deals with both, a documentary survey on plating techniques used at that period and a structural characterization of some components of instruments made by the Sax factory belonging to the collection of the Musée de la Musique, Paris. Examination is performed in a non-invasive way and results show details indicating the use of a given procedure to obtain the coating, as well as some features of its deterioration.

Contact: Virginia Costa (Conservare – IRRAP pour le muse de la musique)
Funding: Musée de la Musique, Paris

New research projects


Characterisation of Erard piano strings (***Conservare – IRRAP***)

The choice of materials and techniques used in the manufacture of strings is one of the most important issue craftsmen are faced with and are currently the subject of a research project dedicated to the choice of appropriate materials and manufacturing techniques to produce replica in the conservation field. A lot of manufacturing processes are given in historical records but the information given cannot be used within the current metallurgical processes that produce strings having very different characteristics than those obtained in the past. The best approach would be first to precisely study the string considered and to manufacture then a new one having similar mechanical and vibratory properties.

The objective of this project is then to determine the properties of Erard piano strings in order to assess the possibility to define a particular typology associated to these instruments. A certain number of fundamental characteristics such as the structure, the composition and the mechanical resistance of strings from instruments dated from years 1791, 1801 and 1812 have been obtained by optical microscopy, SEM-EDS and microhardness tests. The results confirm the diversity of the alloys used for the manufacturing of red, yellow and white strings and allow estimating the original working conditions from predetermined mechanical characteristics. Further development of our study to other strings should allow establishing a database that could be used for the restoration of artefacts and the realisation of replicas.

Contact : Virginia Costa (Conservare-IRRAP pour le musée de la musique)
Funding : Musée de la Musique, Paris

General information

Websites

- **ARTECH network**: http://server.icvbc.cnr.it/progetti_futuri/progetto_artech.htm. Network facilitating the access of conservation professionals to different investigation techniques of Cultural Heritage artefacts

- **BIGSTUFF (Care of Large Technology Objects) 2004:**
http://www.awm.gov.au/events/conference/bigstuff/index.asp

- **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/

- **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 past activities (Short Term Scientific Missions deadlines, training schools…).

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

- **Cost Action D42: ENVIART** (Chemical Interactions between Cultural Artefacts and Indoor Environment): www.enviart.org. You have to register (free access) to get access to all information.

- **e-Preservation Science**: http://www.e-preservationscience.org. Online publication of papers in conservation science.

- **European Cultural Heritage Network**: http://www.echn.net/. European network of professionals interested in the conservation of Cultural Heritage.

- **ICOMAM**: International Committee of Museums and Collections of Arms and Military History: http://www.klm-mra.be/icomam

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

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

- **Laboratoire Pierre Sue**: LPS PhD thesis related to the alteration of archaeological artefacts can be downloaded from http://www-drecam.cea.fr/lps/ (in French) and go to "Archéomateriaux et prévision de l'altération."

- **METAL*Consn*-info** homepage: http://rsc.anu.edu.au/~hallam/METALConsn-info.html

- **M2ADL** - Microchemistry and Microscopy Art Diagnostic Laboratory is now available at the following website: http://www.tecore.unibo.it/html/Lab_Microscopia/M2ADL/

- **New York Conservation Foundation** website: http://www.nycf.org/

- **PROMET** website: http://www.promet.org.gr

- **RESTAURACION METAL SUR AMERICA**: www.restauraciondemetales.cl

- **TEL (PhDs on line):** http://tel.ccsd.cnrs.fr/

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

- **Online publications of Surface Engineering Journal.** Issue addressing specifically to Metal issues: **Surface Modification Issues in Art**, Volume 17, Issue 3, June 2001. Can be downloaded
from:(http://www.ingentaconnect.com/content/maney/se/2001/00000017/00000003;jsessionid=1xpmlw91522a3.victoria)

- **ANDRA** (Agence Nationale pour la Gestion des Déchets RadioActifs)
http://www.andra.fr/interne.php3?publi=publication&id_rubrique=82&p=produit&id=5. The following documents can be ordered for free from this website : *Analogues archéologiques et corrosion* (in French only) and *Prediction of Long Term Corrosion Behaviour in Nuclear Waste Systems* (in English).

Future seminars and conference

- **COST Interdisciplinary Training School On Cultural Heritage** (10-15 September 2007, Genova, I). For more information visit the following website: www.cost.esf.org/2007-ts-genova
- **METAL07**, triennial meeting of the ICOM-CC Metal WG (17-21 September 2007, Amsterdam, NL). For the whole programme, list of papers and posters and abstracts visit the following website: www.metal07.org
- **Conference on Surface Modification Technologies (SMT 21)**, Session on "Arts and Surfaces" (24-26 September 2007, Paris, F). The session on "Arts and Surfaces" will be coordinated by Dr Alessandra Giumlia-Mair. For more information please contact Alessandra Giumlia-Mair (giumlia@yahoo.it)
- **Symposium 2007** – Preserving Aboriginal Heritage: Technical and traditional approaches (24-28 September 2007, Ottawa, Canada) organised by the Canadian Conservation Institute. For more information visit the following website: http://www.cci-icc.gc.ca/symposium/index_e.aspx
- **Les techniques électrochimiques en conservation d'objets métalliques : principes et applications** (08-09 November 2007, INP Conservation Dept, St Denis, Paris). For more information visit the following website:
http://www.inp.fr/professionnel/formation_permanente/fp_restaurateurs/fpr_le_programme/fpr_calendrier_annuel/
- **Metallkonservierung – Metallrestaurierung Geschichte, Methode und Praxis** (07-08 December 2007, University of Applied Art - Vienna, Conservation Department), conference organised by the University of Applied Art as a celebration of its 140 years jubilee. For more information visit the following website: http://www.dieangewandte.at/restaurierung/
- **Training Seminars on Research Planning, Characterisation, Conservation and Management in Archaeological Sites – ARCHAIA** (28-30 January 2008, Copenhagen, DK and 15-17 May 2008, Bologna, I) addressed to 90 post-graduate students, scholars and professionals of different backgrounds. The results of some funded EU research

projects and COST actions will be presented. For more information visit the following website: www.archaia.eu

- **Holding it all together; ancient and modern approaches to joining, repair and consolidation** (21-22 February 2008, London, UK) organised by the British Museum. For more information contact Janet Ambers: science@thebritishmuseum.ac.uk

- **Art2008**, 9th International Conference (25-30 May 2008, Jerusalem, Israel) on the non-destructive testing, microanalysis and preservation in the conservation of cultural and environmental heritage, organised by the Israel National Society for NFT. For more information visit the following website: www.isas.co.il/art2008

- **MetalEspaña '08** Conference on Conservation and Restoration of Metallic Cultural Heritage (Madrid, 10-13 April 2008) organized by the National Center for Metallurgical Research (CENIM), CSIC and Autonomous University of Madrid (UAM). For more information, contact congreso.metalespana08@cenim.csic.es or visit www.cenim.csic.es/metalespana08

- **Ancient mining in Turkey and the Eastern Mediterranean** - AMITEM (15-21 June 2008, Ankara, Turkey) organised by the Institute of Archaeometallurgical Studies, Bogazici University, Istanbul (Turkey), the Deutsches Bergbau – Museum Bochum (D), the Institute of Archaeology, London (UK) and the Atilim University, Ankara (Turkey). For more information visit the following website: http://amitem.atilim.edu.tr

Abbreviations and acronyms

DAC-GU: Department of Analytical Chemistry, Ghent University
DP-UW : Departments of Physics, University of Warwick
DRASSM : Département de Recherche en Archéologie Subquatique et Sous-Marine
EDS: Energy dispersive X spectroscopy
EIS: Electrochemical Impedance Spectroscopy
ESRF: European Synchrotron Radiation Facility
IRRAP: Institut de Restaurations et de Recherches Archéologiques et Paléométallurgiques
LCRR-CAV: Laboratoire de Conservation Restauration et Recherches, Centre Archéologique du Var
LEMMA: Laboratoire d'Etude des Matériaux en Milieux Agressifs
LPS - CEA: Laboratoire Pierre Süe – Centre d'Etudes Atomiques
LRMH : Laboratoire de Recherche des Monuments Historiques
OCP: Open Corrosion Potential
SEM: Scanning Electron Microscopy
SR: Synchrotron Radiation
XRD: X-Ray Diffractometry

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