



Cultural Heritage Agency
Ministry of Education, Culture and Science

**“New strategies for diagnostics of conservation treatments”
7, 8 February 2019, Amsterdam, the Netherlands**

Dear colleague,

The Cultural Heritage Agency of the Netherlands organises an international two-day conference on new diagnostic strategies for assessing the cleaning in conservation treatments. This conference will be held on the 7th and 8th of February 2019 in the centre of Amsterdam.

The conference is the result of IPERION CH activities in the work-package 7, investigating diagnostics of cleaning of paintings, the coating of metal and the consolidation of stone. Several case studies, techniques and general strategies will be presented.

The conference is held under the auspices of, and sponsored by, IPERION CH and is open to everybody working or studying in the field of conservation, restoration, science and conservation science.

In the current document we present all abstracts of the oral presentations as well as the abstracts of the posters. This document will be distributed as pdf only. We hope that you will enjoy this conferences and are looking forward to fruitful discussions.

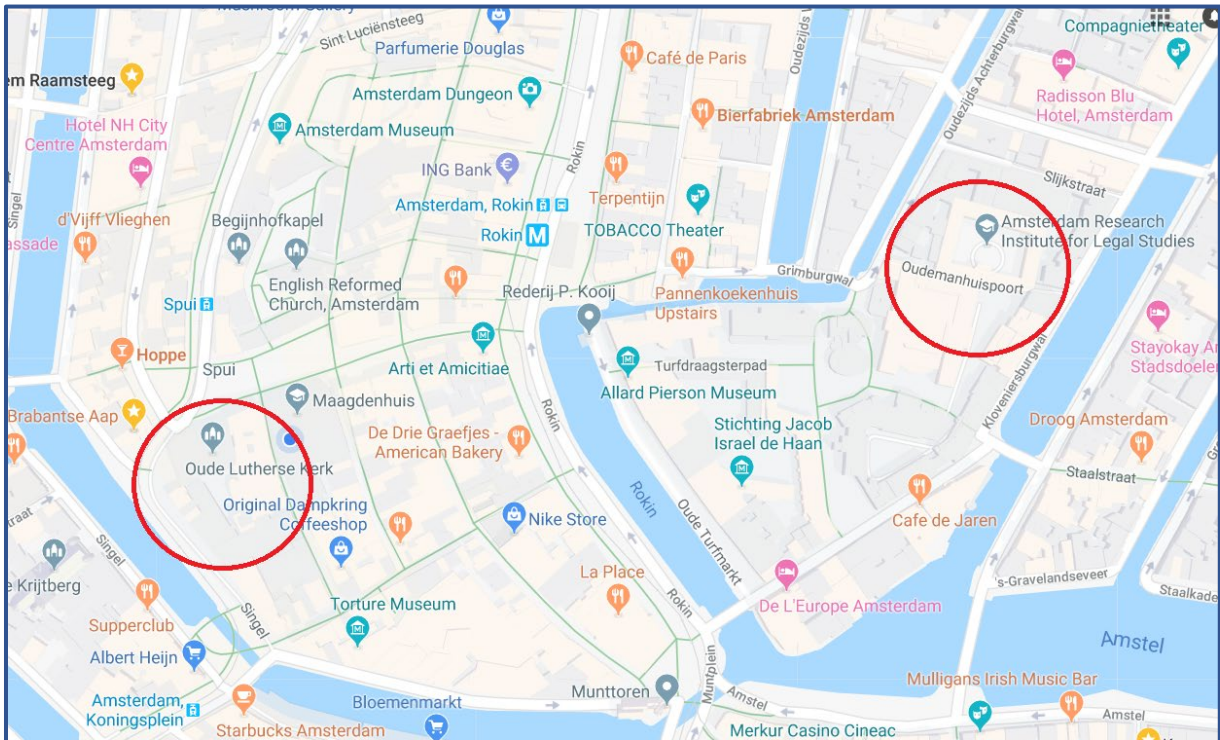
The program committee

Melissa Daugherty
Ineke Joosten
Klaas Jan van den Berg
Maarten van Bommel



IPERION CH

Locations



February 7, 2019

Oude Lutherse kerk

Singel 411, Aula

February 8, 2019

Oudemanhuispoort 4-6

Room D 1.09

February 7, 2019
Aula, University of Amsterdam
Singel 411, Amsterdam

- 09:00 Registration and coffee
- 10:00 Maarten van Bommel, Welcome and general introduction
- 10:20 Klaas-Jan van den Berg, Research approach to monitor the removal of varnishes and over-paints
Monica Galeotti, Research approach to monitor the application of coatings on metal objects
Jadwiga Łukaszewicz, Research approach to monitor the consolidation methods of stone
- 11:20 Discussion
- 11:45 Poster pitches
- 12:30 Lunch and poster presentation
- 14:00 Parallel sessions
Cleaning of Paintings (Aula)
Coatings of Metals (Doelenzaal)
Consolidation of Stone (Belle van Zuylenzaal)
- 17:00 Drinks

Research approaches to monitor the removal of varnishes and over-paints

Klaas Jan van den Berg and Melissa Daugherty, on behalf of all contributors to WP7 Task 1a

Cultural Heritage Agency of the Netherlands, Hobbemastraat 22, 1071 ZC, Amsterdam, The Netherlands

Abstract

WP7 Task 1a focused on diagnostics using high-tech analytical possibilities, especially non-invasive technology, in support of questions arising before and during the removal of unwanted surface layers such as varnishes and overpaints from works of painted art. The aim was to integrate research on the application of the non-invasive microscopic and analytical techniques available in the MOLAB consortium to questions arising during conservation treatment of paintings, especially cleaning. Concerted efforts were made to optimise their use and consider methodological aspects such as their place within the range of scientific techniques currently being used routinely for conservation diagnostics.

Research was carried out aiming at a) providing information on Painting structure and stratigraphy, b) Surface characterization, c) Chemical analysis in support of cleaning questions and d) Swelling studies. Experiments were carried out on a range of multi-layered paint-outs as well as actual 19th Century paintings with a range of varnish and overpaint. The paintings travelled across Europe to the different partners, or were analysed in the presence of instruments from different partners together. In this way, the benefits, limitations and results of each technique were explored, individually and in concert with other techniques.

This talk will give a general introduction to IPERION CH WP7 Task 1a, its philosophy, the members, the materials and works that were studied. It will outline the aims of the project based on practicing conservator's wish-lists, sketch some outcomes, and discuss some of the aspects of the collaboration.

Research approach to monitor the application of coatings on metal objects

Monica Galeotti, on behalf of all contributors to WP7 Task 1b

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Abstract

The application of a protective coating on the surface is the most common strategy to protect metallic heritage against interaction with environmental agents. Restorers apply these protective layers with different methods and use several cleaning treatments to remove old ones. Controlling these operations (removal and application) is critical to achieve either the required extent of cleaning or a good protection effectiveness. In this joint study, our aim was to test the suitability of methods available within IPERION CH partnership for investigating the key features of protective layers on metals, like thickness, evenness and adhesion. We also aimed at getting to know to what extent application parameters (concentration, type of solvent, multiple coats) affect the coating characteristics.

We chose three metal substrates – iron, silver and bronze – and prepared three sets of samples, with a curved shape to mimic folds and protrusions encountered on metal artworks. Traditional protective coatings were applied on the surface, varying some application parameters, like concentration and superimposition of coats. A bunch of analytical techniques – punctual and imaging ones – based on different principles and with different degree of ease of use and of interpretation was applied, and the results were combined together. The bronze set was subjected to artificial ageing to confirm predictions on the protective performance based on their physical and chemical characteristics. The same overall approach was used to study specimens more representative of corroded objects, like slabs of a naturally weathered copper roofs.

Diagnostic strategies for assessment of stone consolidation

Jadwiga W. Łukaszewicz, on behalf of all contributors to WP7 Task 1c

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Abstract

In the current paper the main goal of task 7.1.c. is presented, which is to develop innovative and effective methods for in situ evaluation of consolidation action. We present the general concept of stone consolidation and criteria for the assessment of stone consolidation efficiency.

In the first phase of the work plan, specific mock-up samples were collected and fully characterized. These were two types of stone: a light limestone Pińczów and a porous sandstone Zerkowice. The consolidation was performed by means of an acrylic resin (Paraloid B-72), an epoxy resin (Epidian 5), TEOS (Funcosil 300) and an acrylic-silicone resin (Funcosil AS). Treated and untreated samples were characterized again using the research infrastructure of the IPERION CH Consortium.

The following physical properties i.e. water absorption, colour, wettability of the surface of treated stones were determined first (NCU). In the Stone Department of CNRS-LRMH ultrasonic velocity and capillary absorption investigations were done. In the second stage micro NMR (RWTH Aachen) and IR Thermogravimetric methods were applied for in depth characterization of the samples under investigation. Subsequently, the samples were passed to Tera-Hertz characterization aiming at the measuring of consolidant distribution in the treated samples (CNRS-LRMH). After the completion of the non-invasive tests the samples were subjected to some invasive methods like drill-system and etching in 2MHCl (only in treated limestone) to determine the depth of penetration of consolidants. The protocol of methodology for the assessment of stone consolidation efficiency will summarize the achieved results.

Parallel Session: Cleaning of paintings

Chair: Klaas-Jan van den Berg

Monitoring laser-assisted removal of aged varnish layers from paintings with laser-induced fluorescence spectroscopy

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Abstract

One of the tasks of the EU project IPERION CH (GA 654028) is to investigate diagnostic strategies for assessing the cleaning of paintings and in this respect laser-based methodologies have been considered both for cleaning as well as for monitoring purposes. Ultraviolet (UV) laser ablation at 248 nm has been tested as it offers significant advantages in varnish removal, such as selectivity, high resolution and controllability. Within the project a number of tests using different laser cleaning methodologies have been undertaken. These tests included the application of various laser energy density values and number of repetitions in order to investigate the cleaning depth and the ablation rates.

In parallel, Laser-Induced Fluorescence (LIF) spectroscopy has been also considered as regards its potential in the evaluation and monitoring of the cleaning procedures. LIF being a non-invasive laser spectroscopic analytical technique provides information on the molecular fingerprint of the analysed surfaces and thus it allows for direct comparison of the fluorescence signals recorded on the painted surfaces before and after the cleaning. Therefore, its role as regards the reliable assessment and the on-line monitoring of the cleaning interventions is critically examined.

In this work, the preliminary tests to define the selected laser ablation methodology for different cleaning challenges (i.e. ultra-thin varnish layers, over-paintings etc.) as well as the tests to investigate the role of LIF as an assessment and monitoring tool, will be critically discussed. Work on technical (fresh and aged) varnish samples as well as on the three paintings selected from the study collections of the Cultural Heritage Agency of the Netherlands and the Rijksmuseum for the research purposes of Task 7.1 of IPERION CH, will be presented.

Monitoring of the painting surface morphological and stratigraphic changes by microprofilometry and OCT measurements

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Abstract

The cleaning of paintings is one of the most important and controversial process bringing irreversible changes in the artwork: decisions have to be made regarding partial or complete removal of varnish, which is a very delicate procedure. Proper documentation of both the morphology of the upper surface and the stratigraphy of the superimposed materials is then mandatory. Traditional cleaning methods include mechanical or chemical removal, and the work of restorers and conservators would be considerably helped by the knowledge of the varnish/upper layer thickness.

Two methods for the non-invasive and non-contact documentation and monitoring of the cleaning process are presented: micro-profilometry, for the high-resolution surface survey, and Optical Coherence Tomography (OCT), for cross-sectional imaging of paint layers. Integration of the results allows to characterize the cleaning in terms of both material removed/left and morphological surface changes.

Evaluation of microscopic and macroscopic changes of paintings caused by cleaning treatments by means of the integrated use of solid phase micro extractions and Nuclear Magnetic Relaxometry

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[b] Institut für Technische und Makromolekulare Chemie, RWTH Aachen University, Worringer Weg 2, 52074 Aachen, Germany

Abstract

Cleaning a painting is an invasive procedure and should therefore be examined and monitored with all available diagnostic methods able to study the physical and mechanical changes that occur to the painting during and after the treatment. We propose the novel integrated use of solid phase micro extraction and nuclear magnetic resonance relaxometry to evaluate the solvent entry and retention in the paint layer, as well as the microscopic and macroscopic changes caused by the cleaning treatment.

Unilateral NMR is increasingly being used as a tool for monitoring the state of tangible cultural heritage [1] and has already been employed to quantify the diffusion of water into paint layers resulting from cleaning agents [2]. SPME has been tested to evaluate solvent retention after cleaning the painting layer [3-4].

The two techniques have been employed to study mock-ups and a genuine painting cleaned with different solvents applied by swabs and with gels. Similar results were obtained with both techniques when swabs were used, identifying the same solvent retention scale. Moreover, NMR reveals variations in the relaxation time distributions of the paint layer, which can be considered a fingerprint of its elasticity. When gels were applied, the low mass transfer of the solvent did not allow the simultaneous determination of retention in all layers. For this reason a single slice in the paint layer is selected and monitored. Only the complementary use of the two techniques allows to describe both the retention and the variation of the elasticity of the painting layer. The integrated use of the two techniques could thus be particularly useful to optimise the cleaning procedure.

References:

1. Rehorn, C., Blümich, B., *Angew. Chem. Int. Ed.* 2018, 57,7304-7312.
2. Angelova, L. V., Ormsby, B.; Richardson, E., *Microchem. J.* 2016, 124, 311-320.
3. Samorì, C.; Galletti, P.; Giorgini, L.; Mazzeo, R.; Mazzocchetti, L.; Prati, S.; Tagliavini E., *Chem. Sel.* 2016, 1(15), 4502–4508.
4. Prati, S., Volpi, F., Fontana, R., Galletti, P., Giorgini, L., Mazzeo, R., Mazzocchetti, L., Samorì, C., Sciotto, G., Tagliavini, *Pure Appl. Chem.* 2018, 90 (2), 239-251.

Detection of cleaning system residues on polychrome surfaces by reflection FT-IR

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Abstract

In this research, reflection FT-IR spectroscopy has been evaluated for in situ non-invasive detection of non-volatile cleaning residues during conservation treatments of paintings. As a matter of fact, over the last fifteen years, several new formulations and procedures have been developed in order to respond to the specific needs of painting cleaning. These innovative methodologies are based on the use of active substances like surfactants, chelating agents and enzymes (mostly non-volatile) able to solubilize in a targeted way the components to be removed. The total control of the cleaning action is extremely difficult and residues of these cleaning products can remain on the painted surface with adverse effects on surface stability over time.

In this study more than ten non-volatile compounds, frequently reported in cleaning formulations, have been selected and analysed by FT-IR in transmission and reflection mode in order to elaborate a complete infrared database for their recognition. Then, the selected cleaning agents were applied on painting mock-ups and analysed by reflection FT-IR spectroscopy in order to test sensitivity and specificity of the technique for their non-invasive detection in consideration of the possible spectral overlapping with the painting components as well as of the spectral distortions arising from reflection phenomena at the surface. The analytical approach was extended to the examination of more complex painting systems, such as aged paint mock-ups and an historical oil painting, where the same non-volatile components analysed in laboratory were applied as ingredient of gel formulations commonly in use for cleaning gels. The presence of cleaning residues was, then, investigated at two common steps of the gel cleaning practice: after dry removal of the gel and after clearance with solvent or water.

The results here presented and discussed clearly show limits and potentials of reflection FT-IR spectroscopy for in situ on-line monitoring of cleaning treatments of painting surfaces, demonstrating a clear advance in the detection of cleaning residues in terms of sensitivity and of provided chemical information.

Step-by-step monitoring of gradual varnish removal from easel paintings by combined use of optical coherence tomography (OCT) and reflection FTIR spectroscopy

Magdalena Iwanicka [a], Patrizia Moretti [b,c], Saskia van Oudheusden [d], Marcin Sylwestrzak [e], Laura Cartechini [b], Melissa Daugherty [d], Klaas Jan van den Berg [d], Piotr Targowski [e] and Costanza Miliani [b]

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Abstract

The solvent removal of unwanted layers (e.g. aged varnishes, overpaints, oxalate patinas, dirt) from easel paintings is one of the most frequently performed restoration treatments. However, questions rise regarding its controllability and safety to the artwork. Since there is a need for developing diagnostic methodologies able to inform restores on the chemical, optical and morphological effects of cleaning, we have explored the possibility of complementary use of optical coherence tomography (OCT) and reflection mid-FTIR spectroscopy. The OCT technique non-invasively provides cross-sectional images with varnish layers directly visible and thus permits for direct measurement of their thicknesses. Combining many cross-sections into 3D data provides insight into the varnish thickness distribution over a given area of the paintings. Reflection FTIR allows for a chemical characterization of the surface compounds revealing the nature of the varnish and monitoring its removal. Artificially aged model samples and two historical paintings were investigated at different steps of solvent cleaning tests and the results of the two techniques cross compared highlighting the benefits of their combined exploitation.

Synthesis and integration of the results

Melissa Daugherty

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Abstract

The research in work package 7.1a 'Diagnostic Strategies for Assessing the Cleaning of Paintings' was carried out on a range of mock-ups and three paintings dating between the 18th and 19th century, from the study collections of the Cultural Heritage Agency of the Netherlands and the Rijksmuseum: Interior scene (fig. 1a), Portrait (fig. 1b) and Flowers (fig. 1c). The paintings were selected for the range of cleaning issues that they present, in particular the presence of degraded varnishes and overpaints.

The measurements were performed before, (if possible) during and after cleaning, combining multiple techniques and when available supported by micro-invasive analysis. The paintings travelled to four different countries, collaborating with eight different partners and performing fifteen different analysis techniques, most of which were discussed in the previous lectures. This resulted in a vast amount of data that have been published/presented in several papers, talks, a summer camp and Ph.D. theses.

This talk will present aspects related to the logistics and collaboration between the partners. Furthermore it will discuss a synthesis of the general outcomes of the work package. Lastly, the repository that is currently being made to ensure internet access to all research data in the future will be presented.



Figure 1a: Interior, oil on canvas, 44.6 x 55.8 cm.



Figure 1b: Portrait, oil on canvas, 48.7 x 59 cm.



Figure 1c: Flowers, oil on panel, 20.8 x 27.4 cm.

Parallel Session: Coating of Metal

Chair: Maarten van Bommel

Assessment of protective properties of metal coatings by Electrochemical Impedance Spectroscopy (EIS)

Emilio Cano and Blanca Ramírez Barat

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Abstract

Corrosion, i.e., the chemical reaction of the metal with the environment, is probably the main degradation factor for metallic cultural heritage, and different strategies are used by conservators-restorers to arrest it. One of those strategies is the application of coatings, such as varnishes or waxes, to form a barrier between the metal and the environment. Measuring the protective properties of these coatings, comparing the performance of different materials or application procedures and assessing the evolution of their behaviour over time is therefore crucial to ensure a proper conservation of metallic heritage.

While other characterization techniques can provide information on the elemental or molecular composition of the coating (for instance, FTIR, Raman, XRF, ect.) or physical structure or properties (OCT, Eddy Current, etc.), electrochemical techniques directly probe the corrosion process, providing quantitative information of corrosion rates and information about corrosion and protection mechanisms.

One of these techniques, the Electrochemical Impedance Spectroscopy (EIS) is the main technique used for corrosion and protection studies since the 80s. EIS is based on the application of a small AC voltage (E) signal (usually 10 mV) to the system under study, and measuring the AC current (i) response. Nevertheless, EIS is not widespread in heritage science since conventional electrochemical cells are not suitable for large objects or in situ measurements on the irregular surfaces of sculptures and monuments. As an alternative, we have designed a gel polymer electrolyte (G-PE) electrochemical cell specifically tailored for metallic heritage studies.

Under IPERION CH, this G-PE EIS has been used to measure and compare the performance of different coatings and application procedures on bronze, silver and steel samples. Results have shown significant differences in the performance of different coatings, not always related with the thickness of the coating. In the case of bronze, coatings have been subjected to artificial ageing, and the loss of protection of the coatings has also been assessed.

Non-invasive FT-IR reflection to assess and monitor coatings on metals

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Abstract

Uniformity, thickness and long-term behaviour of the coating are among the main questions that conservators need to address when they apply a protective treatment. An attractive aspect of FT-IR spectroscopy is that not only chemical characterization but also quantitative information can be obtained within a single spectrum, enabling an overall assessment of the entire coating-substrate system. Non-invasive FT-IR reflection spectroscopy is particularly advantageous when applied on metals, because it suffers less than on non-reflective surfaces of diffusion and refraction phenomena that cause distortion of the spectra. On reflective surfaces coated with optically thin layers adherent to the surface, a double transmission of the radiation may occur through the coating itself, providing high sensitivity and spectra quite similar to those in transmission. This process is termed as “transflection” or “reflectance-absorption” depending on the thickness of the film. Based on the Beer’s Law, a linear relationship exists between absorbance and thickness, providing information on the uniformity of the coating.

In this framework, the reliability of FT-IR reflection measurements was explored on bronze and silver coupons coated with several protective films (acrylic polymers, nitrocellulose, microcrystalline waxes), in single and multiple layers and with different concentrations. The absorbance of marker bands of each chemical composition and of alteration products of the organic coatings (e.g. carbonyl and methyl/methylene stretch bands) was calculated as area and compared, showing a correlation between band intensities and number of coats. Eddy current and Optical Coherence Tomography were used as reference methods to measure the coating thickness in several spots. The FT-IR reflection spectroscopy proved effective in checking the uniformity of the investigated coatings, showing that no spectral distortions occur up to a thickness of at least 12 μm on average. In addition, the technique has proven its high sensitivity, as it is able to detect very thin layers (<1 μm) of organic coating. This feature is of great importance when reflectance FT-IR is used to monitor the removal of old coatings.

Ultrasound (Acoustic) μ Tomography applied to metallic objects coatings evaluation

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Abstract

In this work high-frequency ultrasounds are utilized for the evaluation of the stratigraphy of metallic samples that are coated via a class of varnishes. Acoustic (ultrasonic) microscopy is a well-established non-destructive technique which is based on the efficient emission-reception (pulse - echo) of high frequency ultrasonic waves. It constitutes a powerful non-destructive testing technique that provides quantitative structural information of an object of the order of micro-meters. The basic advantage of the acoustic microscopy, compared to equivalent electromagnetic techniques, is the ability of the acoustic waves to penetrate at almost any liquid and solid material, thus providing information of the inner layers. The interfaces between the various materials with different acoustic properties produce echoes that result in the a-scan signal. A series of consecutive a-scans result in the b-scan and offers a tomographic two-dimensional cross-sectional view of the object that is under investigation. Furthermore, a raster scanning of a-scan measurements in a region of interest (ROI) results in a 3D model – tomographic imaging of the internal "anatomy" of the object. From the 3D model and the tomographic images various post-processing data can be acquired such as roughness measurements, stratigraphic information and structural defects identification at the scale of micrometres. Additionally, advanced signal processing techniques, such as wavelet analysis and Hilbert transformation, can be applied to the raw data of each a-scan in order to enhance the acquired images. Data are acquired with a modular acoustic microscope able to work in the lab as well as in situ. The frequency range of the utilized ultrasonic transducer is 110-260 MHz with central frequency on 175 MHz providing spatial and thickness resolution of a few microns. Several promising results on metal alloy (silver alloy) samples with protective coatings are indicated based on the tomographic images. The aim is to gather information on the features that affect the performances of coatings on metal surfaces, like thickness in a very low range down to few micro-meters, evenness and discontinuities of the film as well as to compare different application methodologies.

Assessment of coatings on iron

Ineke Joosten [a], Janneke van der Stok [b] and Teresa Bruni [c]

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Abstract

The surface of iron metal objects is in most cases covered by a natural patina and/or by a coating for aesthetic and protective purposes. The assessment of the efficiency of protective coatings on the iron metal surface is fundamental to set the appropriate preservation strategies and maintenance plans, as well as to make decisions for the best practice of applying the coating. To test a selection of most used coatings a set of mock-ups of low carbon steel with a specific shape and dimensions were produced. Five different coatings, Paraloid B44, Paraloid B72, Tecero wax, Zaponlac and Soter-Fe, and combinations of these coatings were applied with a brush. The coated surfaces were characterized by microscopy and scanning electron microscopy (SEM). For comparison, four iron nails dating from the Roman period different in shape, corrosion products and state of conservation are cleaned and coated. The thickness of the coatings on the mock-ups was assessed by Optical Coherence Tomography (OCT) and found to be extremely thin, often below 2 μm . The thickness and uniformity of the coating over the surface was tested by analysing cross sections of the mock-up with SEM.

Application of OCT to monitor the coatings of metals

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[b] Faculty of Physics, Astronomy and Informatics - Nicolaus Copernicus University - Grudziadzka 5, 87-100 Torun, Poland

Abstract

Optical Coherence Tomography (OCT), a broadband interferometric technique that uses infrared radiation in the 800–2000 nm range, has been widely applied for examination of cultural heritage objects as it allows a cross-sectional visualization of the internal structure of multilayer systems. Recently, it has been applied to objects other than paintings, such as made of metal or stone, to measure the thickness of transparent coatings.

We present the results of OCT examinations of a set of metal samples (made of iron, brass and silver) and deliberately developed in the framework of IPERION CH project as well as naturally aged copper roof sheets, covered with various combinations of coatings. The main purpose of the utilisation of OCT was to cross-reference the measurements of thicknesses of coatings performed with the other methods. The results obtained with OCT were broadly consistent with others, showing a considerable heterogeneity in the thickness of the hand-applied coatings.

OCT may be considered as the method of choice, if a combination of layers is under investigation (transparent over opaque or transparent over the rusted surface) and the thickness of the transparent one is to be determined. In such cases, standard methods such as magnetic eddy current will provide rather the cumulative thickness of all non-metallic coatings.

Protective coatings on metal artworks: a multi-technique approach to the characterization of key parameters for performance assessment

Monica Galeotti

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Abstract

In the restoration of metal artworks, either steel or silver- or copper-based alloys, a fundamental goal is to leave the surface in the most stable condition for future preservation. To this end, the metal items which are to be exhibited either outdoors or indoors need protection against environmental agents and pollutants. By far the most common option chosen is the application of an organic protective coating on the surface after the cleaning phase. Notwithstanding the knowledge acquired over time on the performance of these coatings, many questions are still open about the key requirements of protective layers. They have to match several conservation expectations, from visual impact to uniform protection of the whole surface treated, whether just after application or after ageing. Among the key parameters are, of course, coating thickness and its homogeneity.

The questions of whether a proper thickness is achieved through various application methods and of how homogenous the coating is are very challenging for those facing metal artefacts protection. Practitioners often work in a "blind" way, either when the application is done by brush (most common) or by spraying. A further open question about protective coatings of metals is whether suitable methods to investigate their characteristics are available. Industry offers a wide variety of monitoring methods, but only few of them meet the specific needs of the diagnosis in the field of historical metals. In addition to the obvious requirements of no invasiveness and portability, the appropriate techniques for investigating coatings on metal artefacts must fit complex, three dimensional surfaces, with chiselled and punched details. Of course, different levels of analysis are available, varying in terms of time required, sophistication, and expense.

The easy-to-use and practical methods are the most suitable to control and monitor the application of the coating and its long-term behaviour, even at the expense of the degree of characterization. Fast methods must be tested against the most reliable methods for safe use in the field. Among the available techniques, imaging techniques are preferred to point-wise ones because they provide a clearer view of the spatial homogeneity of the surface, provided that their resolution is sufficient with respect to the scale of thickness variations.

In this project, we use a combination of different techniques to investigate the parameters that regulate the corrosion protection ability of varnishes on silver and bronze artefacts. These techniques include Optical Coherence Tomography, Electrochemical Impedance Spectroscopy, Reflectance FTIR, Ultrasound Tomography, Electron Microscopy, Eddy Currents and others. The goal of the work is twofold. On the one hand, we want to help improve the knowledge of the characteristics and properties of coatings. On the other hand, we want to test suitable methods for in situ control of conservation processes and their reproducibility. In particular, our aim is to identify suitable methods to control the gradual removal of old coatings and the uniform application of new ones.

Parallel Session: Consolidation of Stone

Chair: Véronique Vergès-Belmin

IR thermal analysis to monitor stone consolidation

Jadwiga Łukaszewicz

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Abstract

IR thermography is accounted to non-invasive methods, which in addition is fast and simple in use. At first, the method found an application to the determination of thermal loses in buildings of different kind. The awareness of the method principles and advantages lead to first attempts towards its application to the conservation of historic monuments. The first practical research focused on the analysis of the surface of wall paintings. In this case, by the determination of surface temperature distribution one tried to discover the structure and the state of preservation of wall paintings and mortars. In case of architectonic and stone monuments the method was successfully used for the determination of humidity distribution in the surface layer. In frames of the current project another application field was investigated i.e. several attempts were performed to determine the consolidant distribution in the surface layer of stone monuments. Mock-up limestone and sandstone samples were saturated with acrylic resins, epoxy resins and a consolidant agent treatment based on TEOS. After the consolidants got hardened, such treated samples were subjected to investigations by means of a thermal imaging camera. Passive analysis mode was applied first. Such obtained results were not interpretable. Therefore, a next series of mock-up samples was prepared in which the concentration of consolidating agent was higher in the surface and additionally the samples were saturated with water. Particularly, the last measure let to distinguish surface spots where the consolidants were present. After the completion of passive tests active mode was applied i.e. the samples were heated-up shortly prior the thermographic analysis.

Comparative results of micro-drilling technique used to evaluate consolidation action on limestone and sandstone

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Abstract

Consolidation treatments are necessary when stone materials show loss of cohesion and disaggregation. Their use in practice shall be done only after the assessment of their behaviour on the substrates to be consolidated. In this laboratory work, the objective was to evaluate three important requirements of a consolidant product when applied to get a mass consolidant effect; i) the increase of stone resistance (hardness by drilling); ii) the depth of penetration of the consolidant; iii) the presence of interfaces due to the treatment.

In this research, several consolidants available on the market and commonly used in the past (ParaloidB72, an epoxy resin, TEOS and acryl-siliconic resin) were applied (by brush and by capillary raise) in two type of stones (one limestone and one sandstone) used in cultural heritage, in Poland.

The DRMS equipment is basically a power drill with constant feed and a force transducer that measures the thrust force as a function of the drilling depth. During the test (micro-destructive) a hole of about 5 mm diameter is produced. The method (Tiano & et al., 2000) has been used for several other purposes, besides the evaluation of stone consolidation treatments applied on soft materials that justified the research in this field at that time. Several aspects have been point out and justify care, experience or the use of a different protocol to test and interpret correctly the data, namely the wear effect of the drill bit (on abrasive materials) or dust accumulation (indicated as examples).

Using these data in a comparative basis, it was not only possible to identify the treated zones and the depth of penetration but also to evaluate the distribution of product to promote an homogenous (or heterogeneous) strength areas after consolidation. A very clear illustration of the different behaviour of the products was also possible, in both lithotypes, regardless the abrasively of the stone material.

Reference

Tiano, P., Filareto, C., Ponticelli, S., Ferrari, M., and Valentini, E. – Drilling force measurement system, a new standardisable methodology to determine the “superficial hardness” of monument stones: prototype design and validation. *Int. Journal for the Restoration of Buildings and Monuments*, 2000, vol. 6, No.2, 115-132.

Perspectives of THz method for the determination of the depth of penetration of consolidating products

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Abstract

In the framework of the IPERION CH project, Task 1-c: stone consolidation, the LRMH investigated the terahertz technique (THz) and the ultrasound velocity measurement for the evaluation of the penetration depth of stone consolidating products. The project was realized in partnership with different teams of the project, each one contributing with a technique in order to compare non-destructive and destructive analyses in the laboratory for future investigations in-situ.

At the present only destructive techniques are available and efficient for the evaluation of such a parameter in-situ. This project intended to find a non-destructive technique as reliable as destructive ones. The idea is to share the same samples (cubes of stone) and test different techniques: the samples went from one laboratory to another according to a predefined characterization protocol. Thus, besides ultrasound velocity measurements and terahertz technique as well as dithizone test, differential HCl attack and micro drop absorption time achieved by the LRMH in France, nuclear magnetic resonance was performed by RWTH Aachen ITMC in Germany, infrared thermography was achieved by NCU in Poland, drilling resistance measurements were realized by LNEC in Portugal, and SEM observations were performed by ICVBC-CNR, Italy.

Two series of samples were studied. The first one comprises about 20 limestone and sandstone cubes from Poland consolidated with different products and not contaminated in salt. The second series of sample considered is about 30 limestone cubes from France which have been either just consolidated with ethyl silicate, or just contaminated with sodium chloride, or both contaminated and consolidated.

Terahertz measurements performed on the first series were not conclusive. Possible reasons are: a product penetration deeper than the depth investigated by the THz technique, and/or an interface between consolidated and non-consolidated part not well defined, and thus not detectable with the THz technique. The analysis on the second series of samples is still ongoing, but the first THz scans show promising results.

NMR-Relaxometry and depth profiling for the analysis of historic mortars and hydrophobic coating in masonry

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[b] School of Engineering and Computing, University of the West of Scotland, High St, PA12BE Paisley, Scotland

Abstract

Sensors for stray-field nuclear magnetic resonance (NMR) have been employed to measure tangible cultural heritage since they were first conceived. Although NMR is a method with inherently low sensitivity and requires larger amounts of time than many other techniques, it is non-destructive and grants access to spin densities and relaxation times, physical quantities which are exclusive to the method. This paper describes theory, instrumentation, and applications of unilateral NMR in the field of cultural heritage.

One of the most prominent fields of application for unilateral NMR is the investigation of porosity in stone and soil. In a study of ancient mortars in Herculaneum, a city buried by the ashes of Mount Vesuvius during the famous eruption of 79 AD, the profiles of over 60 sites, fragments and mock-ups were compared for the first time with methods known from statistics and pattern recognition. The effects of high temperatures up to 900 °C on low and high-density sandstone were determined in terms of their transverse relaxation times. Mock-ups were cross referenced with actual walls and fragments to assess the damage of the western wall of the burnt down Mackintosh Library in Glasgow.

Due to the capability of unilateral NMR sensors to non-destructively detect fluids in porous media, the technique can be employed to evaluate the efficacy of various hydrophobic surface treatments. This talk will show selected examples and provide an outline of how NMR-relaxometry and depth profiling can be applied to such samples.

Synthesis and integration of the results.

Jadwiga Łukaszewicz

Nicolaus Copernicus University, 87-100 Toruń, Poland

Abstract

Structural reinforcement (often called as consolidation) of stone monuments is one of key stages in the whole conservation process. Consolidation is still a problem under discussion and a target of numerous investigations. Corrosion and erosion which are continuously running in stone objects are resulting in stone disintegration and diminishing of its mechanical strength. Such a decay has several typical symptoms like cracking, falling apart and creation of voids. Generally, two intervention scenarios are possible: an exchanges of a decayed element to a new one which is not permitted by conservation doctrine or the preservation of an original matter which is strongly advised by the same doctrine. Over the centuries, mainly the last two, attempts have been made to use various strengthening substances for this purpose, i.e. natural resins, waxes, drying oils and in the 20th century, artificial resins. Works were also carried out on the methods of introducing consolidators into the structure (pores) of stone materials.

Efficacy and, at the same time, the possibility of using individual materials were evaluated on the basis of laboratory tests of reference samples. A completely open problem is to determine the effectiveness of the in situ consolidation process. This requires the use of non-invasive methods. The IPERION project attempts to integrate research methods to determine the effectiveness of consolidation of historic buildings. Two types of methods were applied: (1) invasive methods such as physical properties testing, i.e. water absorption, porosity, wettability of the surface, strength and penetration depth by measuring the drilling resistance measurements, (2) non-invasive methods, i.e. IR thermography, terahertz technique (THz) or NMR.

Invasive methods show much greater accuracy and usefulness. Non-invasive methods require further research. Although ultrasound velocity measurements and nuclear magnetic resonance seem very promising.

February 8, 2019
University of Amsterdam
Oudemanhuispoort 4-6
room D1.09

- 08:30 Coffee
- 09:00 Coating of metal plenary session
Monica Galeotti, summary of the parallel session
Stefania Agnoletti, Case study: Facing a huge conservation project: the three bronze doors of the Baptistery of Florence
Tonny Beentjes, future perspectives in metal conservation
- 10:00 Consolidation of stone session
Jadwiga Lukaszewicz, summary of the parallel session
Andreas Furche, Case study: Surface analyses and cleaning of blackened Italian marble sculptures burnt during the Second World War from the collection of the Bode-Museum in Berlin
David Giovannacci, Future perspective: On the relevance of non-destructive methods for measuring water content in masonry
- 11:00 Cleaning of paintings session
Klaas-Jan van den Berg, summary of the parallel session
Bronwyn Ormsby, Case study, The conservation of modern and contemporary art: collaborative cleaning research – methodologies, materials and progress
Aviva Burnstock, future perspectives
- 12:00 Concluding remarks and closure

Case study

Facing a huge conservation project: the three bronze doors of the Baptistery of Florence

Stefania Agnoletti

Opificio delle Pietre Dure- department of metal conservation, viale Strozzi, 1 - 50129 Firenze, Italy

Abstract

Since 1979, the Opificio delle Pietre Dure has been facing a huge conservation project of the three monumental bronze doors of the Baptistery of Florence. The first door restored has been the Door of Paradise, made by Lorenzo Ghiberti between 1425 and 1452, which features large part of the surface covered with gilding. The intervention carried out on this door marked a turning point in the history of conservation of gilded bronzes because of the use of the laser cleaning technique (owing to a collaboration with IFAC-CNR).

The next conservation intervention was done on the North Door, made again by Ghiberti between 1404 and 1424. The restoration of this door took advantage of the know-out and the techniques implemented for the Door of Paradise, which were adapted to the specific needs of the North Door. Currently, the restoration of the door by Andrea Pisano, made between 1330 and 1336, is ongoing.

In this contribution, an overview of the restoration process of the doors will be given, with a particular focus on the decision making process about preventive conservation measures: the doors, in fact, were not put back to the Baptistery and they are now conserved indoors. The strategy adopted involve the exhibition in controlled showcases with any protective coating. On the other way round, cases where the application of a coating was chosen, for bronzes either exhibited outdoors or indoors, will be shown.

Future perspective

Future perspectives in metal conservation, a conservators view

Tonny Beentjes

University of Amsterdam, Conservation and Restoration of Cultural Heritage, Johannes Vermeerplein 1, 1071 DV Amsterdam, the Netherlands

Abstract

The profession of metal conservator has developed a great deal over the years. The older generation of often autodidacts has now largely been replaced by formally trained conservators. Over the years the level of conservation training in most countries has been brought up to university level to provide a good understanding of materials and their deterioration and an increased level of science required for complex conservation treatments. Today's metal conservator is also more and more involved with the art-technological aspects of objects, characterising the used materials and techniques. The increased knowledge of the material aspects of objects also makes the modern conservator more aware of the impact of past and current treatments on these material aspects. As a result of this, conservators are increasingly careful and hesitant to use interventive treatments. Even mild surface cleaning and applying protective surface coatings such waxes and lacquers are now first critically evaluated and not so readily applied as in the past. Treatment innovations can be roughly divided in two groups: low tech such as gels and high tech such as laser, plasma and dry-ice cleaning. The successful application of both these techniques, low tech as well as high tech requires a thorough understanding of the mechanisms involved and their effect on an historic object. Another innovation in the field is the use of 3D technology, such as 3D imaging and printing. 3D printing has been used for some years now successfully to make up missing parts or supportive elements in conservation treatments. 3D imaging for documentation is gradually finding its way in conservation, especially since the hardware and software is becoming more affordable and user friendly.

Case study

Surface analyses and cleaning of blackened Italian marble sculptures burnt during the Second World War from the collection of the Bode-Museum in Berlin

Andreas Furche [a], Thomas Wirth [a], Ilaria Federici [a], Elke Cwiernia [a], Cristina Aibéo [a], Paul Hofmann [b], Julien Chapuis [b] and Ina Reiche [a]

[a] Rathgen-Forschungslabor

[b] Skulpturensammlung und Museum für Byzantinische Kunst, Staatliche Museen zu Berlin - Stiftung Preußischer Kulturbesitz

Abstract

The cleaning of black carbon covered Italian marble and limestone sculptures is a challenging task for restorers and scientists, especially when black soot is penetrated into the porous surface of a stone. Numerous sculptures of the Bode-Museum underwent two domestic fires at the repository in Berlin in May 1945, where soot damaged the surface of these artworks. Promising results in removing black layers were achieved by the application of modified gels to artificially weathered and sooted Carrara marble mock-ups. However, transferring the cleaning method to original samples requires a cautious proceeding with respect to short-term and long-term implications. To understand physico-chemical reactions, the analytics of gel-treated marble surfaces is of essential importance.

Time-of-Flight Secondary Ion Mass Spectroscopy (ToF-SIMS) and Auger electron spectroscopy (AES) were used to gain insights into surface chemistry and the distribution of chemical groups or fragments and enable a deeper understanding of the functionality of the applied gels and of the efficiency of tested cleaning processes. Depth profiling by ToF-SIMS is introduced as a tool to study the penetration behaviour of applied cleaning chemicals.

The contribution presents the analytical methods, their possibilities and challenges, and summarizes initial results of this study.

Future perspective

On the relevance of non-destructive methods for measuring water content in masonry

David Giovannacci [a], Laura Normand [a], Véronique Vergès -Belmin [a], Markus Küppers [b], Christian Rehorn [b] and Bernhard Blümich [b]

[a] CRC-LRMH, USR 3224, French Ministry of Culture/CNRS, France

[b] ITMC RWTH Aachen University, Germany

Abstract

The water content in stone is of primary relevance for the preservation of cultural heritage. On one hand, high water content promotes the development of microorganisms and causes mechanical or physico-chemical alterations by dissolution/recrystallization of salt, on the other hand, low water content promotes physical damages by salt crystallization or shrinkage. Therefore it is important to determine and control this variable to assess the risk of damage and take preventive conservation measures.

The most accurate methods to measure water content require destructive sampling and can only exceptionally be used. Only a few methods are non-destructive but their accuracy may be limited.

In this presentation, the authors describe and compare the different non-destructive techniques or prototypes like the thermography, the evanescent-field dielectrometry, the electrical tomography or the nuclear magnetic resonance (with the help of RWTH Aachen) used by the LRMH in France to evaluate the water content in masonry, .

It is addressed to whoever is responsible for the preservation and maintenance of immovable cultural heritage (heritage buildings).

Case study

The conservation of modern and contemporary art: collaborative cleaning research – methodologies, materials and progress

Bronwyn Ormsby

Scientific Dept., Tate, Millbank, London, UK

Abstract

This presentation outlines the approach, methodology, key results and challenges encountered as part of Tate's ongoing, highly collaborative research into the cleaning of modern and contemporary paints and painted works of art. Recent projects (2015-2018) such as the Cleaning of Modern Oil Paints (CMOP) and Nanorestart, as well as Tate's ongoing work with the Dow Chemical Company (Dow) have offered invaluable opportunities to explore several aspects of cleaning science pertaining to modern paints - with an emphasis on paint surfaces, to co-design and/or rigorously evaluate newly developed materials for conservation, and to carry out case study treatments on painted works of art. This activity has been supported through a collaborative, practice-based, methodological approach, involving many contributors. Key stages involve the characterisation of paints and paint surfaces, the preparation of test samples, extensive comparative evaluation studies involving established and novel soiling removal methods, and cleaning material residue studies; all of which have enabled the completion of soiling removal treatments on hitherto challenging modern and contemporary works from Tate's collection, including Roy Lichtenstein's *Whaam!* (1963, T00897) and Eva Hesse's *Addendum* (1967, T02394).

Future perspective

The conservator's future perspectives on the surface cleaning of painted art and the diagnostics supporting conservation treatment

Aviva Burnstock

Department of Conservation & Technology, Courtauld Institute of Art, Strand, London, UK

Abstract

Dr. Bronwyn Ormsby and Prof. Aviva Burnstock were present at the onset of the 'diagnostics for surface cleaning of paintings' task in IPERION CH. Their 'questions from conservators were instrumental in setting up the project. Among other areas for research, the importance of developing appropriate methodology for predicting and evaluating the painting's surface was suggested, the use of appropriate reagents and methods for cleaning and the importance of evaluating the impact of residual cleaning reagents on the painting. Documentation, interdisciplinary discussion and a willingness to explore the implications of the changes induced by cleaning were also discussed. This discussion will reflect on the outcomes of research and their implications for conservation practice.

February 7, 2019
Aula, University of Amsterdam
Singel 411, Amsterdam
Poster session
12:30 – 14:00

Posters to be presented

Reflection FT-IR spectroscopy as versatile tool for non-invasive monitoring of cleaning treatments of painting surfaces

Patrizia Moretti , Laura Cartechini, Francesca Rosi and Costanza Miliani

The micro-drilling technique used to evaluate consolidation action- some examples in detail

Dória Costa, Susanna Bracci and Donata Magrini

The application of HIROX microscopy and RTI for the characterization of the paint surface

Melissa Daugherty

Micro-profilometric and RBS analysis of coatings for metals

Róbert Huszánk, Attila Csík, Anikó Angyal and Ákos Csepregi

Non-destructive assessment of protective coatings for heritage metals by means of Electrochemical Impedance Spectroscopy (EIS)

Blanca Ramírez Barat and Emilio Cano

Microprofilometry and OCT: 3D survey and tomographic analysis for the non-invasive monitoring of the cleaning procedure

Raffaella Fontana, Marco Barucci, Alice Dal Fovo, Enrico Pampaloni, Marco Raffaelli and Jana Striova

Optical coherence tomography (OCT)

Magdalena Iwanicka, Marcin Sylwestrzak, Piotr Targowski

Non-invasive FT-IR reflection on metals

Barbara Salvadori, Monica Galeotti, Simone Porcinai, Andrea Cagnini

Drop wettability tests: an evaluation of conservation treatments efficiency applied to metal

Aurélia Azema and Annick Texier

NMR-Relaxometry and Depth Profiling

Christian Rehorn, Markus Küppers and Bernhard Blümich

Terahertz time-domain reflectometry system

David Giovannacci

Reflection FT-IR spectroscopy as versatile tool for non-invasive monitoring of cleaning treatments of painting surfaces

Patrizia Moretti [a,b], Laura Cartechini [b], Francesca Rosi [b] and Costanza Miliani [b]

[a] Dipartimento di Chimica, Biologia e Biotecnologie, Università degli Studi di Perugia, via Elce di sotto 8, 06123 Perugia, Italy

[b] Istituto CNR di Scienze e Tecnologie Molecolari (CNR-ISTM), via Elce di sotto 8, 06123 Perugia, Italy

Abstract

The remarked versatility, selectivity and sensitivity of reflection FT-IR spectroscopy make this technique very promising for in situ real-time monitoring of painting cleaning. The technique is, in fact, highly specific for the identification of organic, inorganic and organometallic compounds and sensitive to the surface molecular properties. Furthermore, the considered instrumentation is non-invasive, portable, fast, reliable and, therefore, recommended to perform in situ investigations. For these reasons reflection FT-IR spectroscopy demonstrated to be a valid tool in support of researchers developing new methods for conservation, as well as conservators in defining the most appropriate cleaning procedures to solve specific issues. In particular, reflection FTIR spectroscopy was used to develop analytical methodologies to be exploited in situ for: i) the detection of cleaning system residues (i.e. non-volatile compounds such as surfactants, chelating agents and thickeners) on treated painting surfaces; ii) the assessment of the removal of overlaid materials (e.g. natural and synthetic varnishes, waxes and overpaints) from painted surfaces in combination with optical coherence tomography (OCT) [1]; iii) the chemical characterization of the removed materials extracted from the cotton swabs used by conservators/restorers. In the poster, an overview of these different analytical approaches based on the use of reflection FT-IR spectroscopy is given.

[1] Iwanicka M, Moretti P, van Oudheusden S, Sylwestrzak M, Cartechini L, van den Berg KJ, Targowski P., Miliani C. "Complementary use of Optical Coherence Tomography (OCT) and Reflection FTIR spectroscopy for in-situ non-invasive monitoring of varnish removal from easel paintings." *Microchemical Journal*, 2018, 138, 7 – 18.

The micro-drilling technique used to evaluate consolidation action- some examples in detail

Dória Costa [a], Susanna Bracci [b] and Donata Magrini [b]

[a] National Laboratory for Civil Engineering (LNEC), Lisbon 1700-066, Portugal; drcosta@lnec.pt

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Abstract

The results obtained with DRMS technique allow the characterization of stone hardness in depth. As a result, not only the evaluation of stone consolidation treatments effect but also the characteristics of the interfaces produced by the treatments can be investigated with this approach.

However, for the selection of the parameters several variables related with stone material (original hardness, composition), the treatment (consolidation effect) and the direct use of this technique on the material (wear effect and abrasively, dust accumulation) must be considered. The interpretation of the profiles obtained also need a global overview of the results and the integration of the information.

In the poster, several practical examples of profiles are presented and commented aiming to illustrate the advantage and the drawbacks of the method, in particular when used to evaluate stone consolidating effects promoted by the products (Paraloid B72, an epoxy resin, TEOS and acryl-siliconic resin) on the two types of stones, namely the limestone and the sandstone, considered in the project. To complement this information, some other cases are included aiming the global discussion of the method.

Reference

Tiano, P., Filareto, C., Ponticelli, S., Ferrari, M., and Valentini, E. – Drilling force measurement system, a new standardisable methodology to determine the “superficial hardness” of monument stones: prototype design and validation. *Int. Journal for the Restoration of Buildings and Monuments*, 2000, vol. 6, No.2, 115-132.

Delgado Rodrigues, J. and Costa, D. - A new method for data correction in drill resistance tests for the effect of drill bit wear. *International Journal for Restoration, Internationale Zeitschrift für Bauinstandsetzen*, 2004, Vol. 10, No 3, 1–18.

The application of HIROX microscopy and RTI for the characterization of the paint surface

Melissa Daugherty

Cultural Heritage Agency of the Netherlands (RCE), Hobbemastraat 22, 1071 ZC Amsterdam, The Netherlands

Abstract

In WP7 Task 1a of IPERION CH the focus was on the diagnostics of the removal of unwanted surface layers. In this part of the research HIROX microscopy and Reflectance Transformation Imaging (RTI) were compared. The measurements were done on the Flower painting; a painting from the reference collection of the Rijksmuseum of Amsterdam. The Flower painting is characterized by multiple thin layers of wax, varnish and overpaint. This makes it a very suitable case study to monitor the controlled removal of the various layers by providing information on structure and stratigraphy and surface characterization. The areas measured with HIROX and RTI were cleaned with a KrF Excimer laser.

The HIROX microscope and RTI are non-invasive techniques commonly used in museum conservation practice, but not yet as a standard tool helping monitoring the removal of layers. RTI offers a detailed photograph of a two-and-a-half dimensional representation of the surface. The process requires a fixed camera, a light that can be moved for each image, and a small, fixed shiny ball to record the light's direction as it changes. [1] The images were captured by moving the light source, without moving the camera, the object or the shiny ball. The Hirox microscope is non-invasive and provides information on the surface texture/roughness with high quality/magnification images. The Hirox produces a real colour 3D image and it gives information on surface roughness, layer structure and a comparison in relative local heights.

The difference between cleaned and not cleaned is visible with HIROX and RTI. The HIROX images illustrate to what extent/level the varnish was removed resulting in a clearly visible difference in appearance. In addition, the amount of material (thickness of the removed layer(s)) could be measured. When trying to distinguish between where the varnish was removed partially, a difference in height was not detectable: the difference in pulses must be bigger for the HIROX microscope to be able to detect a variation in height.

The difference between areas cleaned with a varying pulse or/and fluence is not always visible. The RTI image illustrates the surface texture better than HIROX, but it is difficult to read without additional information on the layer built-up. It is a technique that benefits by combining it with other techniques.

References

[1] Miles, J. et al. 'New applications of photogrammetry and reflectance transformation imaging to an Eastern Island statue' Antiquity Publications Ltd, 2014, Volume 88, Issue 340, 596-605.

Micro-profilometric and RBS analysis of coatings for metals

Róbert Huszánk [a], Attila Csík [b], Anikó Angyal [a] and Ákos Csepregi [a]

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[b] Laboratory of Electron Spectroscopy and Materials Science, Institute for Nuclear Research, Hungarian Academy of Sciences (MTA Atomki), H-4026 Debrecen, Bem tér 18/c, Hungary

Abstract

In this study, the thickness of different coatings on silver and bronze samples was measured. The mock-up samples were about 10×5 cm, with a flat part (5.5×5 cm) and a curved part to simulate a 3D surface. We aimed to check the homogeneity of the coating when it is laid on a non-flat surface, and to check how the chiselled or cleaved details of real surfaces affect the application of the coating, as well as whether these points are preferential points for the onset of corrosion.

Altogether 9 silver and 14 bronze sheets with various coatings were investigated by contact profilometer which was carried out at the Laboratory of Electron Spectroscopy and Materials Science, MTA Atomki. The measuring procedure was the following: a stylus was moved vertically in contact with the sample and then moved laterally across the sample for a specified distance and a specified contact force. A profilometer can measure small surface variations in vertical stylus displacement as a function of position. The height position of the diamond stylus generates an analogue signal which is converted into a digital signal, stored, analysed, and displayed. The method was favoured for its insensitivity for surface reflectance, colour and contaminants. The procedure of the thickness measurement was the same in all cases.

Besides the contact profilometry measurements, RBS analysis is going to be performed at the Laboratory of Ion beam Physics, MTA Atomki. Rutherford backscattering spectrometry (RBS) is an analytical technique used in materials science. RBS allows the determination of the structure, the composition and the depth profile of materials by measuring the backscattering of a beam of high energy ions (typically protons or alpha particles) impacting on a sample.

Non-destructive assessment of protective coatings for heritage metals by means of Electrochemical Impedance Spectroscopy (EIS)

Blanca Ramírez Barat and Emilio Cano

Centro Nacional de Investigaciones Metalúrgicas, Consejo Superior de Investigaciones Científicas (CENIM-CSIC). Avda. Gregorio del Amo 8, 28040 Madrid (Spain)

Abstract

In the past few decades, development of new analytical techniques applied in the field of cultural heritage has been focused on the development of non-invasive techniques and portable instrumentation. Focusing in the field of metallic cultural heritage, besides general analytical techniques, electrochemical methods are of particular interest for conservation assessment. Electrochemical techniques such as Electrochemical Impedance Spectroscopy (EIS) can give information on corrosion processes and/or corrosion rates, on the protective properties of different coatings and inhibitors used in conservation treatments, and their evolution over time.

In situ application of electrochemical techniques to metallic heritage has to deal with some practical difficulties, being one of the main challenges is how to mount an electrochemical cell filled with a liquid electrolyte on the irregular surface of an artefact. To overcome this problem we have developed an gel polymer electrolyte (G-PE) electrochemical cell specifically designed for metallic heritage studies. The G-PE cell is based in a classical three-electrode design, in which the liquid electrolyte has been gelled with agar.

The G-PE cell can be tailored to the specific needs of each study, changing pseudo and reference electrodes, electrolyte composition (to reproduce different environmental conditions), etc. For outdoor sculpture studies, it is typically based on artificial rain solutions, to imitate the environment in which sculptures corrode, to which 3% w/v of agar has been added. The agar concentration has been selected after evaluating the performance of the electrolyte at different concentrations on electrochemical measurements on bronze coupons. The cell has been tested in different materials (bronze, corten steel) on laboratory coupons and in field measurements, and used for monitoring the evolution of patinas and coatings with time. Some of these results are presented, showing the applicability of the gel polymer electrolyte (G-PE) cell for the conservation assessment of protective coatings and treatments for conservation of metallic heritage.

Microprofilometry and OCT: 3D survey and tomographic analysis for the non-invasive monitoring of the cleaning procedure

Raffaella Fontana [a], Marco Barucci [a], Alice Dal Fovo [a,b], Enrico Pampaloni [a], Marco Raffaelli [a] and Jana Striova [a]

[a] CNR-INO National Institute of Optics, Largo E. Fermi 6, 50125 Florence, Italy

[b] Chemistry Dept., University of Florence, Via della Lastruccia 3, 50019 Sesto Fiorentino, Florence, Italy

Abstract

The three-dimensional (3D) survey of an object, besides providing the digital model of the surface, can also be profitably used to investigate its less apparent and sometimes more significant characteristics: this is the case of the 3D relief aimed at documenting and monitoring the restoration intervention.

The proposed instrument for 3D measurements is a micro-profilometer based on a conoscopic distance meter mounted on two orthogonal mounted translational stages. It allows for the scanning of surfaces up to $30 \times 30 \text{ cm}^2$, with lateral and axial resolution of ≈ 20 micron and ≈ 1 micron, respectively. The working distance is 4 cm with a dynamic range of 8 mm.

In order to gain an insight in the structure of the painting, Optical Coherence Tomography (OCT), a non-invasive optical technique for cross-sectional imaging of multi-layered objects, was also applied. The instrument used, a prototype of confocal time-domain OCT, allows the acquisition of tomographic images with lateral and axial resolution of 2.5 micron and 10 micron (in air), respectively. The maximum scanning length is 2.5 cm with no constraints on the axial dynamics.

Optical coherence tomography (OCT)

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Abstract

Optical Coherence Tomography (OCT) is an interferometric non-invasive technique of structural imaging within media scattering and/or absorbing near-infrared light moderately. It originates from diagnostic medicine [1] and has been used for examination of artworks since 2004. [2] The technique's ability of scanning centimetre-wide areas in order to obtain information about sequence, continuity and thickness of the subsurface layers makes it especially suitable for examination of easel paintings as well as other objects of art.[2-4] For the work within IPERION CH project we have been using the system designed and built under FP7 Charisma project at N. Copernicus University in Torun, Poland especially for the examination of cultural heritage objects. It is a Fourier domain OCT instrument utilising near infrared (770–970 nm) radiation and a spectrographic detection. The power of a probing beam at the object is less than 0.8 mW and a given spot of the object is exposed to the light for 40 μ s only. This irradiation level is considered non-invasive even for light-sensitive materials. In practice, the ability to differentiate thin layers is denoted by the axial (in-depth) resolution, which equals ca 2.2 μ m (in material of refractive index equal to about 1.5). Lateral resolution (the ability to differentiate between structures occurring close to each other in lateral plane) is about 13 μ m. These features make our instrument especially well-suited for the examination of thin-layered planar structures, such as easel paintings. The axial imaging range is 1.7 mm and sensitivity 98 dB. The distance to the examined object from the most protruding element of the device is 43 mm and the structural information from the area up to 17 x 17 mm² may be acquired in one measurement. Data collection time for one 2D cross-section (B-scan) is 0.15 s. Two standard video cameras enable the precise documentation of the position of examined area at the object. OCT tomograms (virtual cross-sections) are presented in false colours. Warm colours correspond to high scatter/reflection of the probing light, whereas cold colours mark areas with low scatter. Transparent media (e.g. clear varnishes, glass or air above the surface of the examined object) or areas located beyond the range of penetration are shown as dark.

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3. Targowski P, Iwanicka M Optical coherence tomography: its role in the non-invasive structural examination and conservation of cultural heritage objects - a review. *Appl Phys A* 106:265–277(2012)
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Non-invasive FT-IR reflection on metals

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Abstract

Fourier transform infrared spectroscopy is suitable to provide information on the chemical composition and stability of materials and it is widely used for the characterization of conservation products. In recent years, significant advancements in the use of this technique have been obtained with the development of portable instruments operating in reflectance mode, enabling the non-invasive characterization of superficial inorganic and organic materials. Non-invasive FT-IR reflection spectroscopy is particularly advantageous when applied on metals, because it suffers less than on non-reflective surfaces of diffusion and refraction phenomena that cause distortion of the spectra. On reflective surfaces coated with optically thin layers adherent to the surface, a double transmission of the radiation may occur through the coating itself, providing high sensitivity and spectra quite similar to those in transmission. This process is termed as “transflection” or “reflectance-absorption” depending on the thickness of the film. An attractive aspect of FT-IR spectroscopy is that not only chemical characterization but also quantitative information can be obtained within a single spectrum. Based on the Beer’s Law, a linear relationship exists between absorbance and thickness, providing information on the amount of analyte. In this framework, the potentiality of FT-IR reflection measurements was explored on plastic sheets with standard thickness on reflective metal coupons. The absorbance of marker bands of each chemical composition and of alteration products of the organic coatings (e.g. carbonyl and methyl/methylene stretch bands) was calculated as area and compared, showing a correlation between band intensities and thickness. Further applications include the monitoring of cleaning steps and assessment of limit of detection, which is of great importance when reflectance FT-IR is used to monitor the removal of old coatings.

Drop wettability tests: an evaluation of conservation treatments efficiency applied to metal

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Abstract

In the frame of the WP 7 “Advancing diagnostics for conservation”, task 7.1 “New strategies for diagnostics of conservation treatments”), subtask 7.1.b dedicated to the survey of common metal coating strategies and selection of coating, the CRC-LRMH laboratory (Metal scientific department) proposes to assess wettability tests in using two methods by adapting usual and simple tools;

The first method is based on the contact angle measurement. It needs to be implemented in laboratory by using a microscope which front lens angle can be controlled. A handheld digital microscope (USB) is employed for the second method. In this case, the diameter of the drop is determined. Three sets of threated samples (bronze, silver and iron) were studied before their artificial ageing. These sets were realized according to the work of the group members and are the same in order to be analysed in the different laboratories.

This study aims to test the availability and the limits of drop test in using these two ways. Even if the first seems to provide more precise data, the correlation is always obtained with the second. Both methods give qualitative data concerning the hydrophobic level induced by a coating on a metallic surface. Consequently, it is possible to compare this property according to the different treatments.

NMR-Relaxometry and Depth Profiling

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Abstract

Nuclear magnetic resonance has established itself as an indispensable technique that is used in various scientific fields, including chemical structure analysis and medical imaging. Nowadays, its advantages find increasing use in non-destructive material testing where studies of tangible cultural heritage stand out especially [1]. Many of these studies are performed in the inhomogeneous stray field of small and portable sensors based on permanent magnets, where chemical information is inaccessible. Instead, contrast is gained from relaxation or diffusion processes, which are otherwise either inaccessible, or associated with invasive measurements.

The NMR-MOUSE is the most widely adapted stray-field sensor, as it combines the perks of portable NMR with a disk-shaped detection volume located at a distance of 5-25 mm from the sensor surface. As the sensor is retracted, spin-spin relaxation decay curves can be measured throughout the stratigraphy of any object or surface. The amplitude of these curves can be plotted at each increment to yield the depth profile - a one-dimensional image with high resolution.

Since its conception, the NMR-MOUSE has been applied to measure a wide variety of non-metallic and layered objects, most prominently paintings on wood, mortar and canvas. Textiles, paper, parchment, wooden instruments and even human remains such as bone or the skin of mummies have proven to be equally suitable for the technique [2].

[1] C. Rehorn, B. Blümich, Cultural Heritage Studies with Mobile NMR, *Angewandte Chemie International Edition* 57 (2018) 7304–7312.

[2] B. Blümich, S. Haber-Pohlmeier, W. Zia, *Compact NMR*, Walter de Gruyter (2014).

Terahertz time-domain reflectometry system

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Abstract

Compared with conventional spectroscopic and imaging techniques such as X-ray, ultraviolet, infrared, and laser spectroscopy, terahertz time-domain imaging (THz-TDI) is an innovative, non-invasive, and safe technique, which provides good penetration depth and broad spectral bandwidth (0.1–10 THz). This poster sets out the application of THz-TDI to immovable cultural heritage. The case studies demonstrate the efficacy of the technique in providing structural and material information for conservators.