Surface Analysis of Thin Films for Cultural Heritage

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Surface Analysis of Thin Films for Cultural Heritage

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- Thin Films in CH
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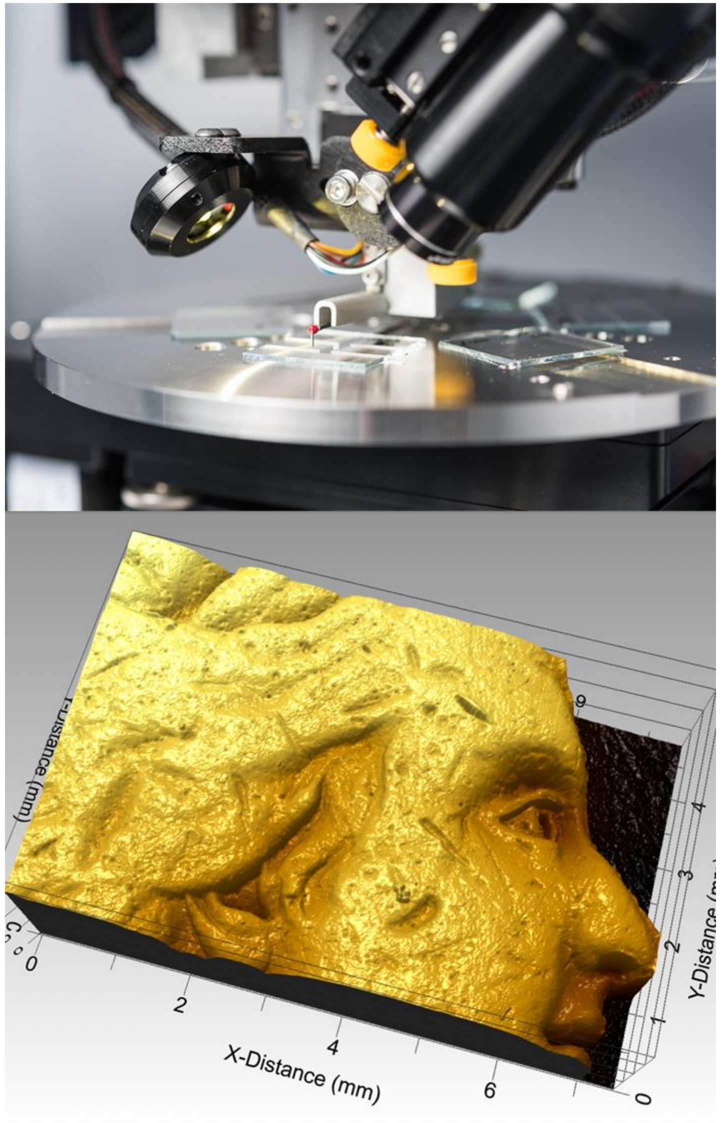

https://www.physik.uni-konstanz.de/fileadmin/physik/nanolab/profilometer/DSC_7939.jpg

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Surface Analysis Methods

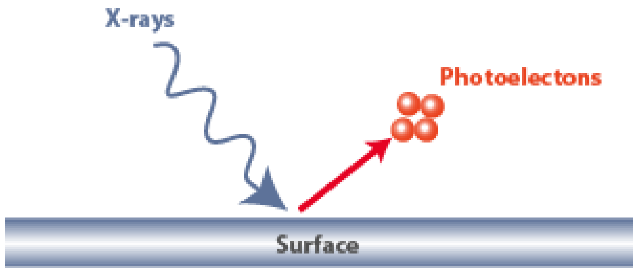
Surface Analysis Methods

Thin Films in CH

Applications in CH

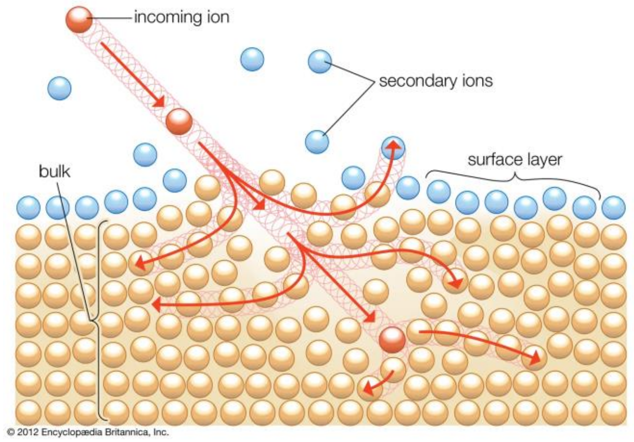
→ Electron detection

- X-Ray Photoelectron Spectroscopy (XPS)
- Auger Electron Spectroscopy (AES)
- Electron Energy-Loss Spectroscopy (EELS)



→ Ion detection

- Secondary Ion Mass Spectrometry (SIMS)
- Low-Energy Ion Scattering (LEIS)
- Nuclear Reaction Analysis (NRA)
- Atom Probe (AP) and Field Ion Microscopy (FIM)



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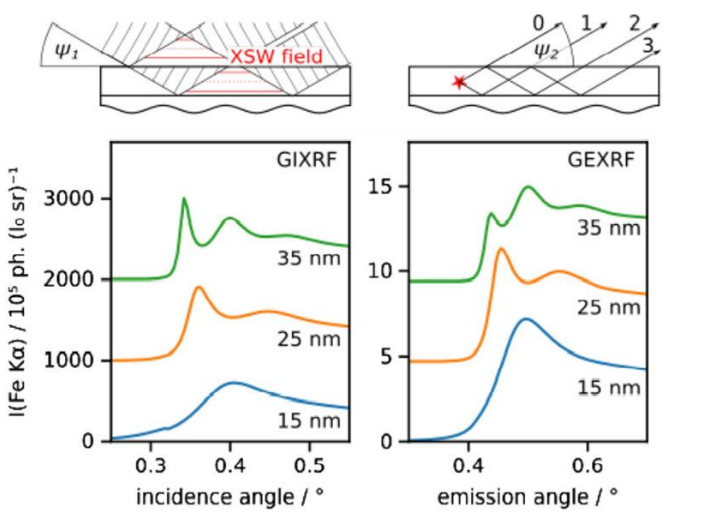
Surface Analysis Methods

Surface Analysis Methods

Thin Films in CH

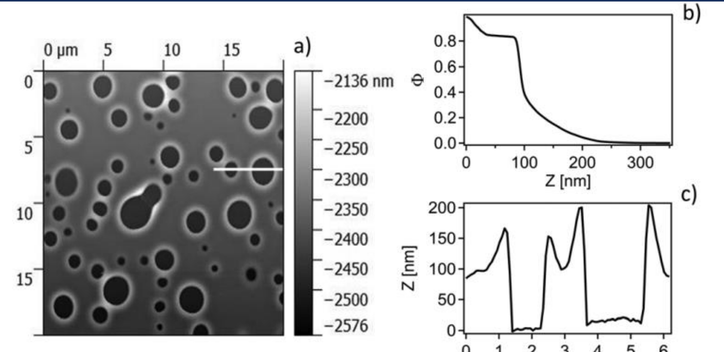
Applications in CH

→ Photon detection
- Energy-Dispersive X-Ray Spectroscopy (EDXS)
- Total-Reflection X-Ray Fluorescence (TXRF)
- Grazing Incidence/Exit X-Ray Fluorescence (GI/GE-XRF)
- Grazing Incidence Small Angle X-Ray Spectroscopy (GI-SAXS)



Phys. Status Solidi B 2020, 2000471

→ Microscopy
- Atomic Force Microscopy (AFM)
- Scanning Probe Microscopy (SPM)
- Scanning Tunneling Microscopy (STM)



Soft Matter, 2020, 16, 1485–1497

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Thin Films in Cultural Heritage

Surface Analysis Methods

Thin Films in CH

Applications in CH

→ Common thin films in CH
→ Common CH characteristics that are challenging for surface analysis

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Thin Films in Cultural Heritage

→ Common thin films in CH

Coating / Varnish
Paint layer(s)
Binder
Support

Corrosion layer
Alloy basemetal

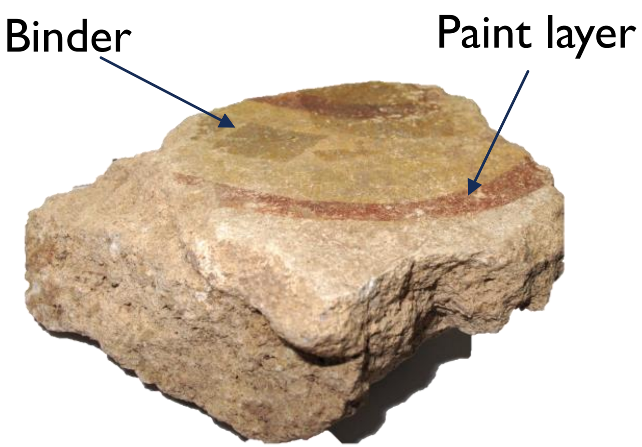

Photo by Maram Naes 2013

Coatings/Varnishes on paintings

Polymers 2021, 13, 2651

Corroded Alloys

Scientifc Reports (2022) 12:6125

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Thin Films in Cultural Heritage

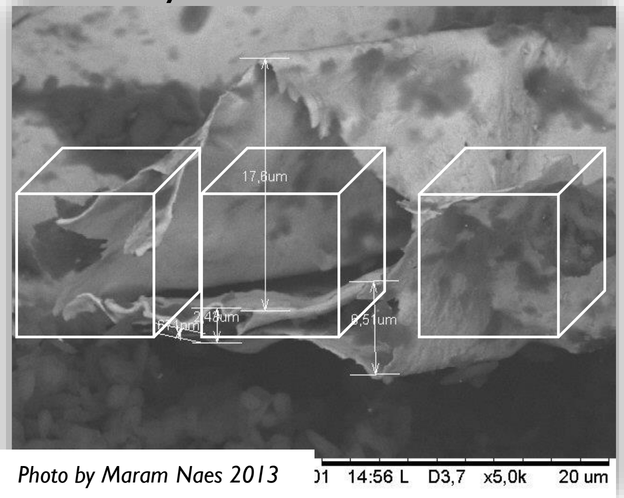
→ **Common CH characteristics that are challenging for surface analysis**

ND and NI priority

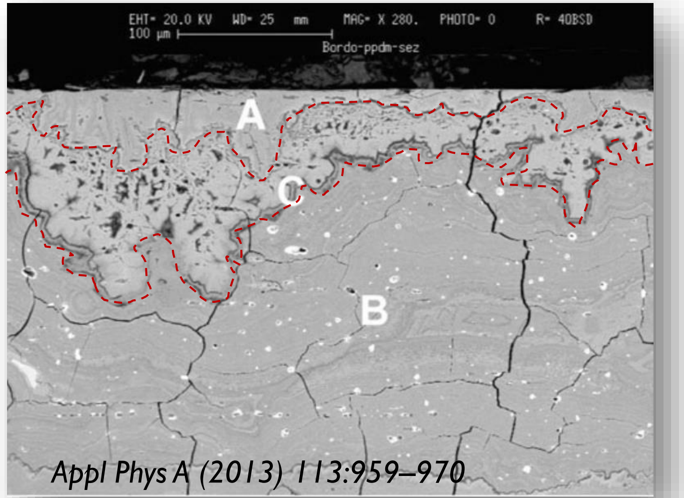
Experimental compatibility to CH integrity

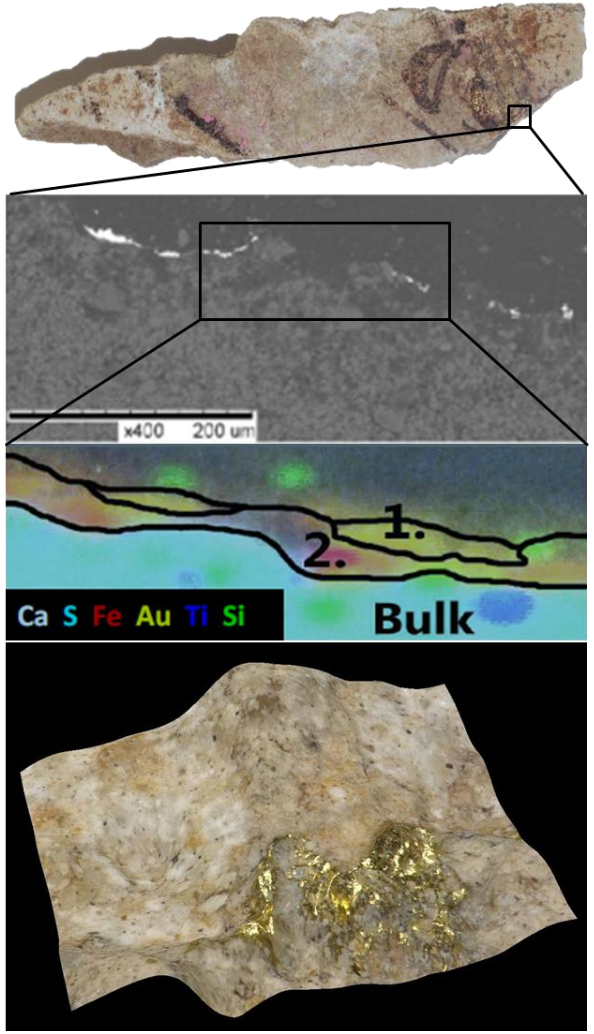
Thermal and vacuum sensitivity

Density variation



Thickness variation





Surface discontinuity

Composition heterogeneity

Surface roughness

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Thin Films Analysis in Cultural Heritage

Surface Analysis Methods

Thin Films in CH

Applications in CH

→ **Common questions for surface analysis of thin films in CH**

- Composition of historical materials and alterated materials
- Assessment of conservation materials and conservation interventions

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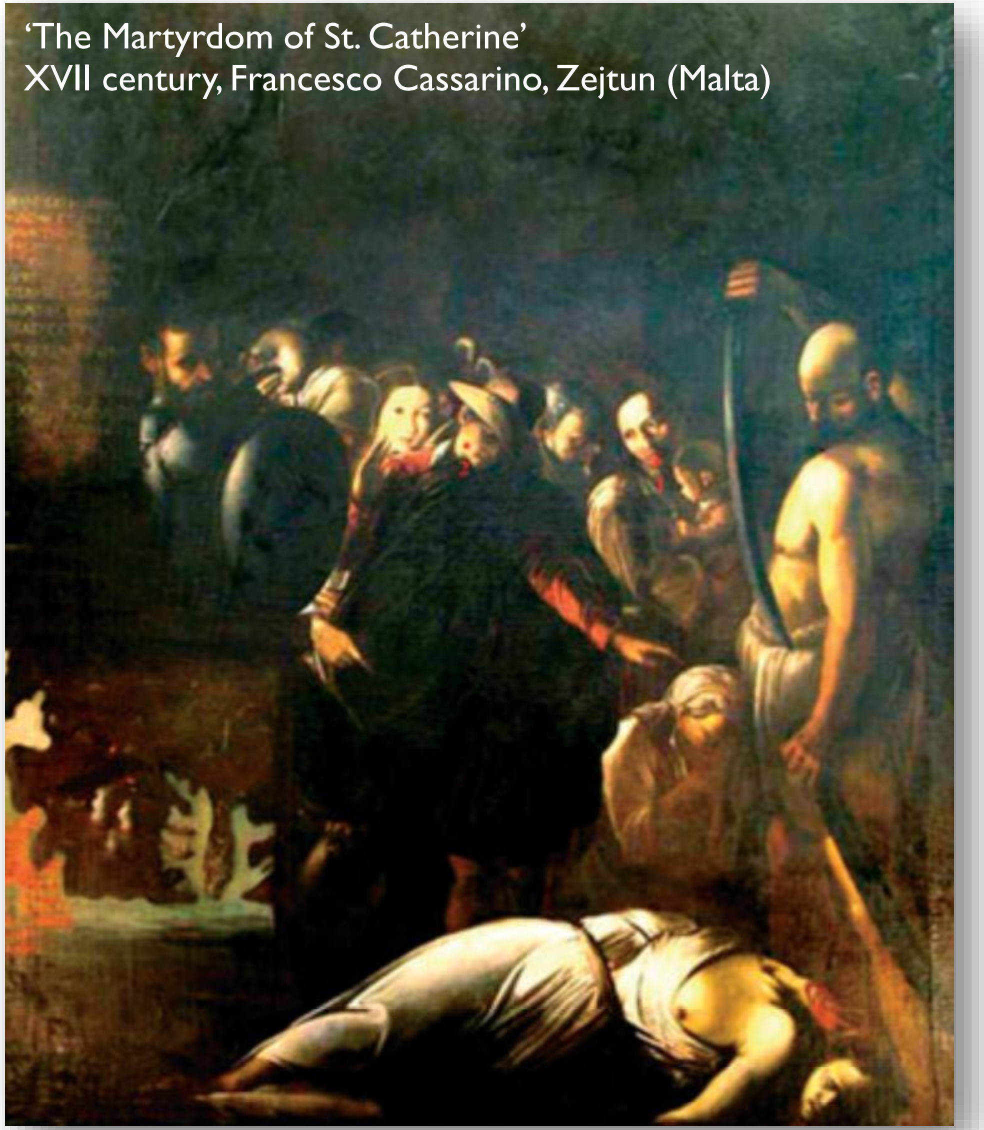
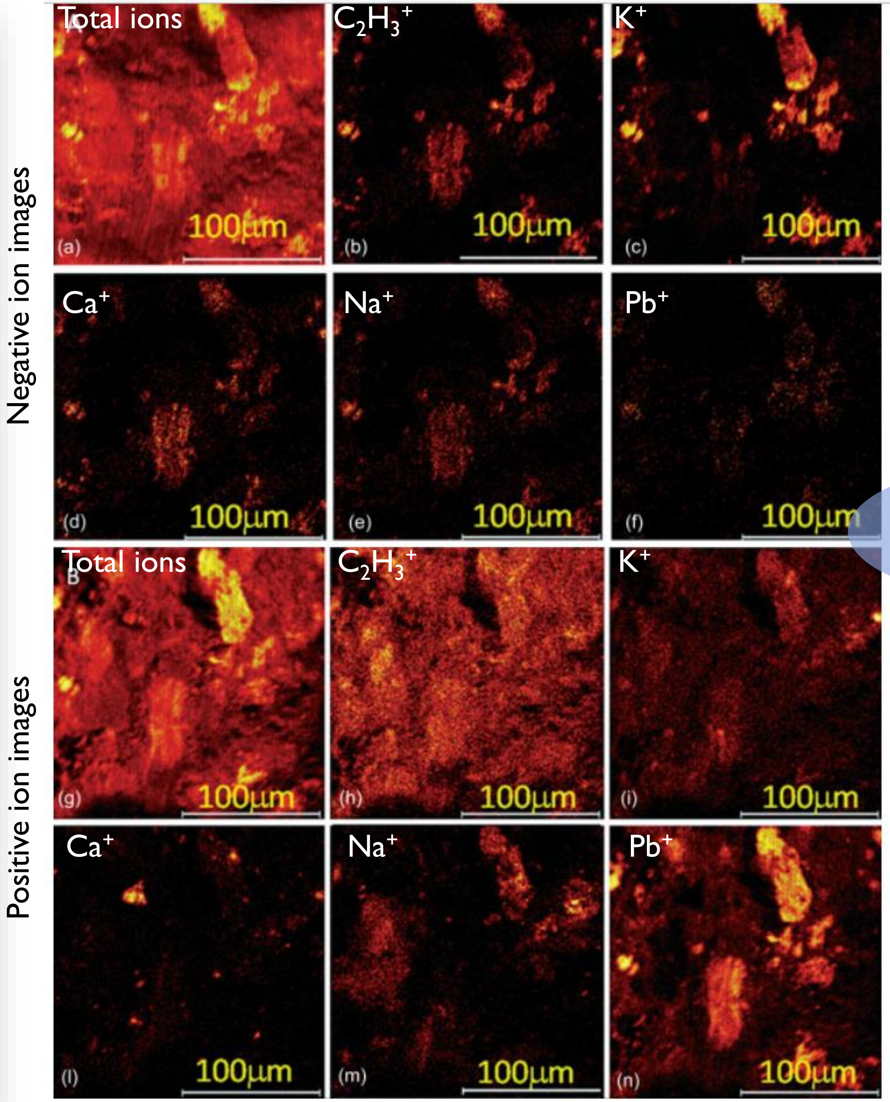
Thin Films Analysis in Cultural Heritage

→ **Common questions for surface analysis of thin films in CH**

Composition of historical materials





Surf. Interface Anal. 2011, 43, 1152–1159

ToF-SIMS images over a 200 × 200 µm² area

ToF-SIMS

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Thin Films Analysis in Cultural Heritage

→ Common questions for surface analysis of thin films in CH

Alteration of historical materials

ECASIA special issue paper

SURFACE and INTERFACE ANALYSIS

Received: 22 August 2011 Revised: 8 October 2011 Accepted: 20 December 2011 Published online in Wiley Online Library: 25 January 2012

(wileyonlinelibrary.com) DOI 10.1002/sia.4845

Micro-chemical surface investigation of brittle carthaginian and roman silver artefacts†

A. Mezzi,[a]* T. De Caro,[a] C. Riccucci,[a] E. Angelini,[b] F. Faraldi[b] and S. Grassini[b]

Brittle Carthaginian and Roman silver artefacts, such as bracelets, coins, small jewels and cups, were found in extremely brittle condition during archaeological excavations in different Italian archaeological sites. Some of these silver objects are accidentally easily broken with little applied force and with only small deformation. In order to identify the origin of brittleness, fresh fractured surfaces have been investigated by means of the combined use of surface and micro analytical techniques such as X-ray photoelectron spectroscopy, scanning electron microscopy (SEM) and field-emission SEM (FESEM) equipped with an energy dispersive X-ray spectrometer, optical microscopy and X-ray diffraction. The overall experimental findings show that the main external brittleness agents are chloride ions that come from the soil of the excavation sites and attack the silver artefacts along the grain boundaries forming a thin layer or islands of silver chloride. Copyright © 2012 John Wiley & Sons, Ltd.

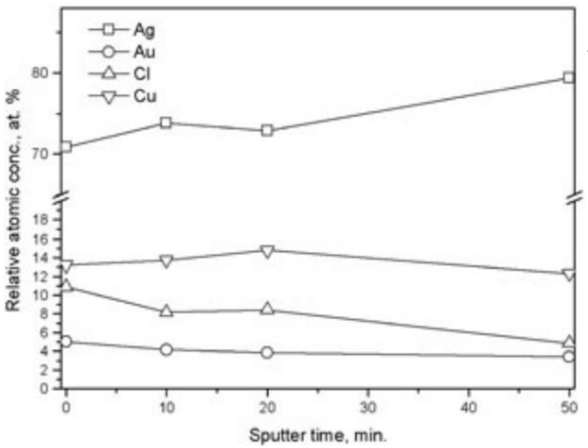
Keywords: silver artefacts; brittleness; FESEM; XPS; XRD

Introduction

Brittle Carthaginian and Roman silver artefacts, such as bracelets, coins, small jewels and cups, were found in extremely brittle condition during archaeological excavations in different Italian archaeological sites. These silver artefacts have been subjected, in ancient times, to mechanical shaping processes resulting ductile during manufacturing, but nowadays, they often break after a minimum stress. Therefore, these artefacts became brittle as a long-term

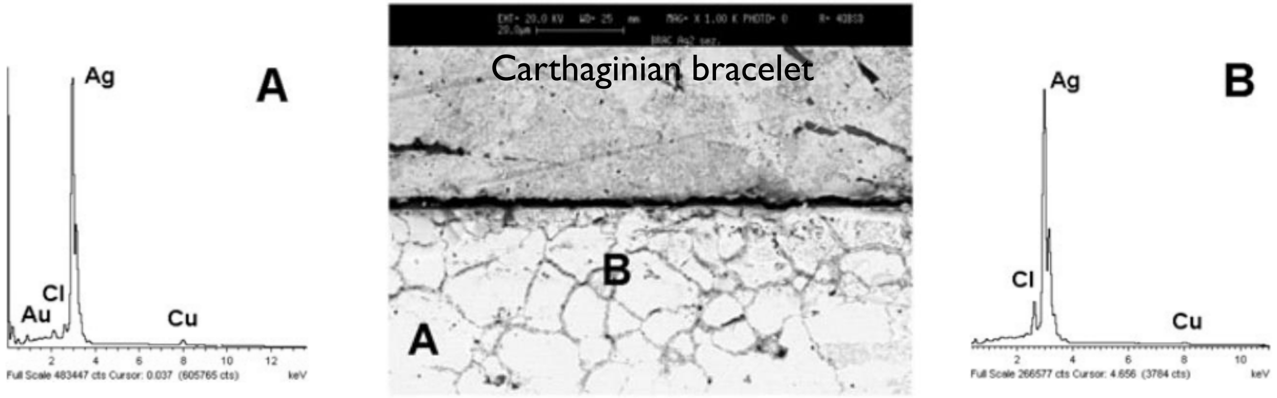
microscope (SEM), equipped with a LaB_6 filament, and a high brilliance LEO 1530 FESEM apparatus, equipped with an EDS INCA 250 and INCA 450, respectively, and a four sectors backscattered electron detector (BSD). SEM images were recorded both in the secondary electron image (SEI) and backscattered image (BSD) mode at an acceleration voltage of 20 kV. FESEM images were recorded both in SEI and BSD mode at different acceleration voltage ranging from 3 kV to 20 kV.

XRD patterns were recorded directly on the samples by a



XPS elemental concentration depth profiles

XPS, SEM-EDX



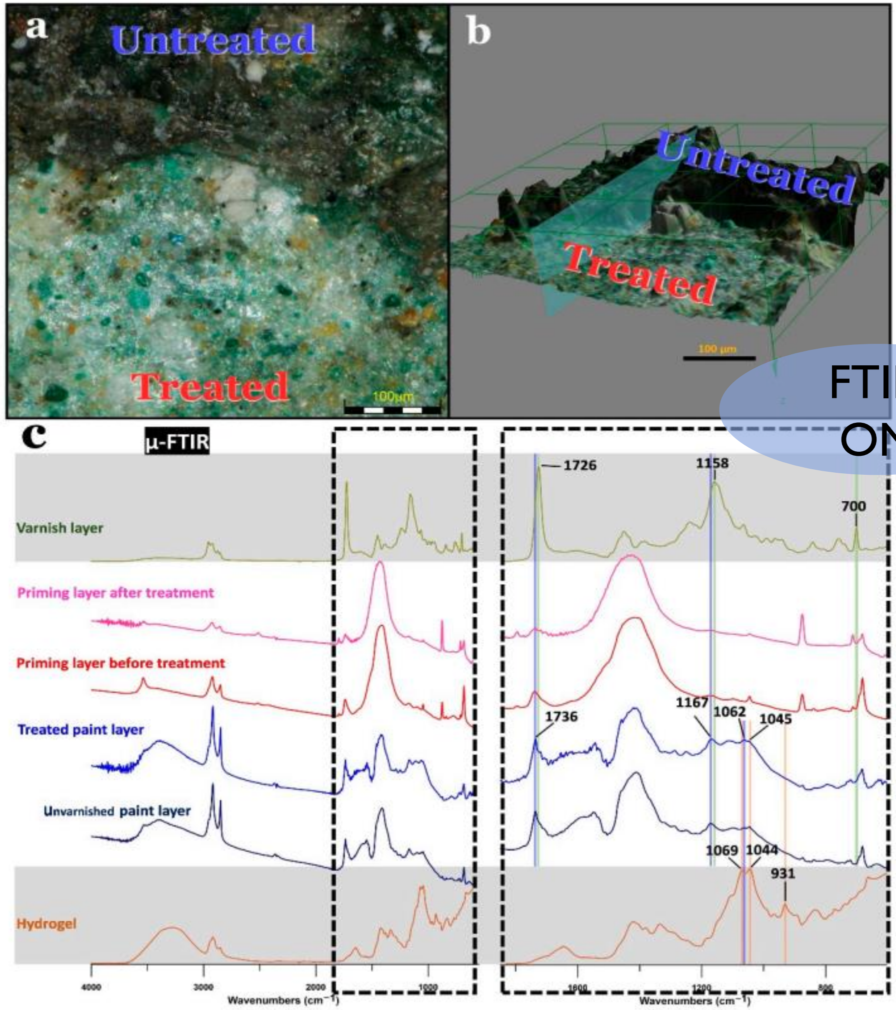
EDS analysis of area A and B on a cross section

Surf. Interface Anal. 2012, 44, 972–976

Thin Films Analysis in Cultural Heritage

→ **Common questions for surface analysis of thin films in CH**

Assessment of conservation materials and conservation interventions



FTIR, OM

Polymers 2021, 13, 2651

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Thin Films Analysis in Cultural Heritage

Willneff et al. Heritage Science 2014, 2:25
http://www.heritagesciencejournal.com/content/2/1/25

HERITAGE SCIENCE

RESEARCH ARTICLE Open Access

Spectroscopic techniques and the conservation of artists' acrylic emulsion paints

Elizabeth A Willneff[1*], Sven LM Schroeder[2] and Bronwyn A Ormsby[3]

Abstract

Introduction: Artists' acrylic emulsion paints are used in many contexts such as paintings, murals, sculptures, works on paper and mixed media; and are forming increasing proportions of modern and contemporary art collections. Although acrylic emulsion paints have been the focus of museum-led research over the past decade, the impact of artists' technique and conservation treatment on the upper-most surface of these paints remains essentially unexplored.

Results: This paper summarises previous studies using vibrational (FTIR) spectroscopy and presents initial assessments of paint surfaces using X-ray spectroscopies (XPS and NEXAFS) aimed at characterising artists' acrylic paint film surfaces after natural ageing and wet surface cleaning treatment. Both techniques were found to be well suited for surface-sensitive investigations of the organic materials associated with artists' acrylic paints, including explorations into: (A) cleaning system residues, (B) surfactant extraction from paint surfaces, (C) the identification of migrated surfactant, and (D) monitoring pigment changes at the paint/air interface of paint films.

XPS, NEXAFS

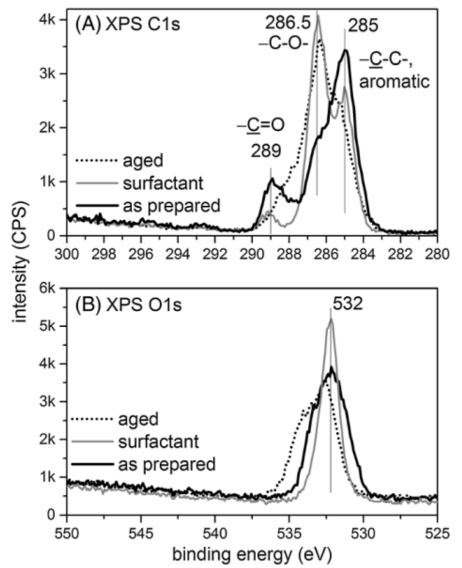

Figure 4 XPS of Surfactant Migration. XP C1s (A) and O1s (B) spectra of Talens PY3 yellow films before ('as prepared') and after ('aged') two years of natural ageing in the dark under ambient conditions compared to a spectrum of Triton™ X-405.

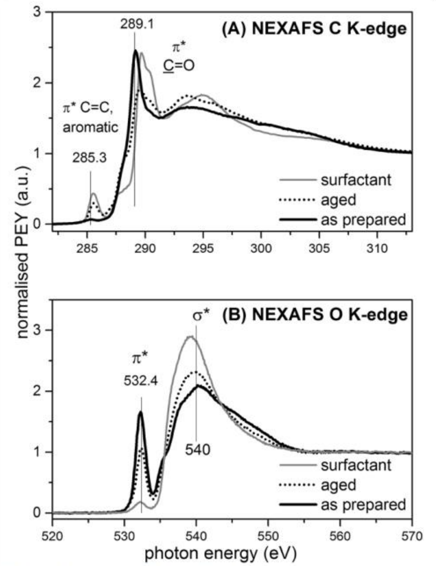

Figure 5 NEXAFS of Migrated Surfactant. NEXAFS C (A) and O (B) K-edge spectra of Talens PY3 yellow films before ('as prepared') and after ('aged') two years of natural ageing in the dark under ambient conditions compared to a spectrum of Triton™ X-405.

SURFACE and INTERFACE ANALYSIS

Research article

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(wileyonlinelibrary.com) DOI 10.1002/sia.3796

The surface behavior of gilding layer imitations on polychrome artefacts of cultural heritage

I. C. A Sandu,[a*] T. Busani[b] and M. H. de Sá[c]

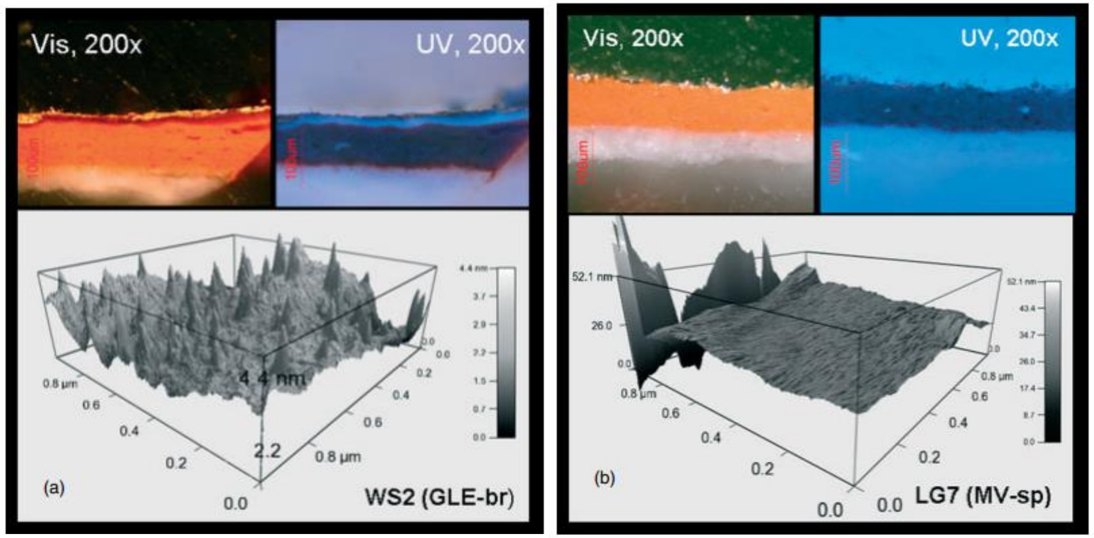
This paper proposes the first results of a larger study on the behavior of gilded surfaces of polychrome heritage artefacts with the aim to understanding the surface patterns and roughness variations before and after specific treatments (burnishing and varnishing). This study can be useful to trace correlations between the compositional features, manufacturing and applications/elaboration processes of gilding layers and the pattern of the surface topography, and also some insights into the degradation/corrosion mechanisms. The results obtained on imitations of gilded surfaces can be further useful for recognizing fake surfaces in the authentication of artefacts from the antiquity markets.

Two types of commercial imitations of gilded surfaces on a wooden support were considered: liquid 'gold' (Cu-Zn powder in a solvent) and 'gold' leaf (Cu-Zn leaf) applied over bole and gesso layers.

A combined analytical approach using atomic force microscopy (AFM), optical microscopy (OM) and colorimetry (CIE L*a*b* system) was applied in order to better understand the behavior of the gilded-varnished surface and of the interface between the metal surface and the other preparative layers. Copyright © 2011 John Wiley & Sons, Ltd.

Keywords: gilded surfaces; imitations; atomic force microscopy; colorimetry; stratigraphical structure; cultural heritage

AFM, OM



Why HESEB?

- Compatible experimental conditions (‚near‘ ambient pressure, He-atmosphere)
- Probing near-surface layer with higher resolution
- Possibility for non-destructive and non-invasive analysis
- Reduced mobility of regional CH objects

SESAME

HESEB Helmholtz-SESAME Soft X-Ray Beamline for SESAME

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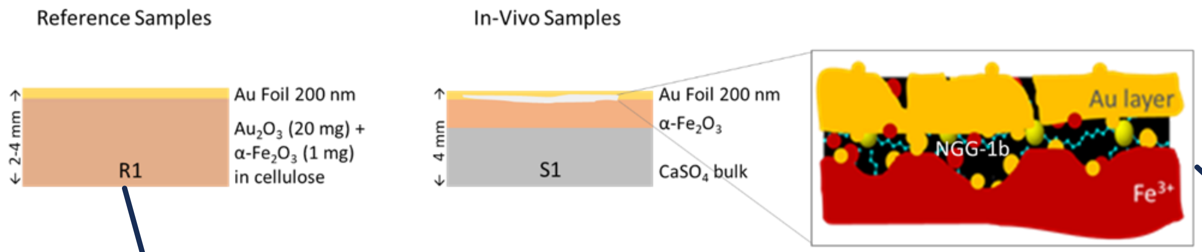
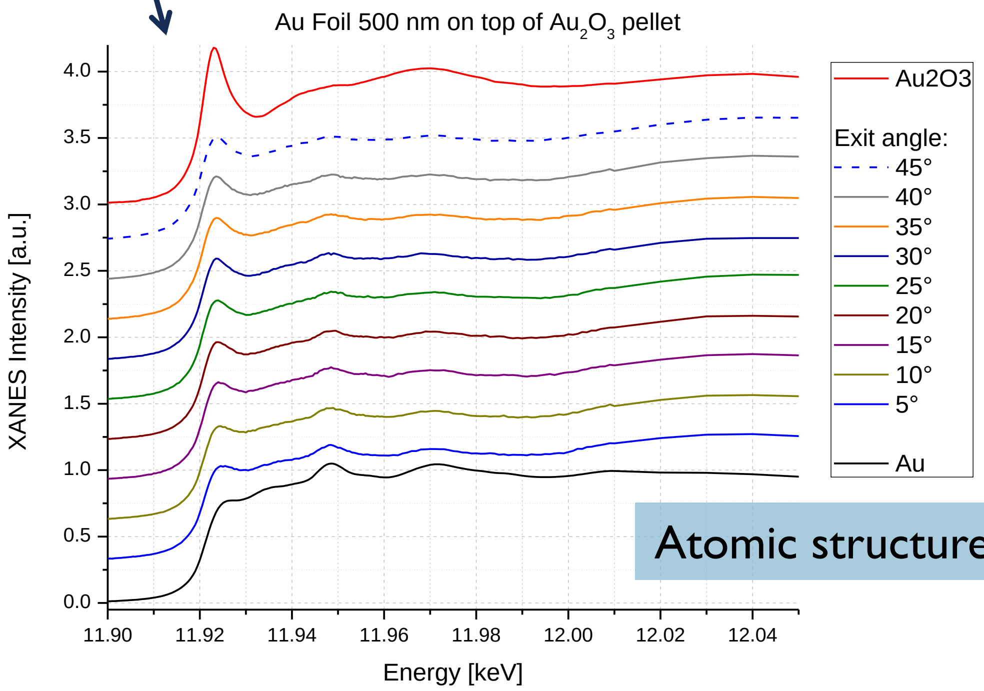
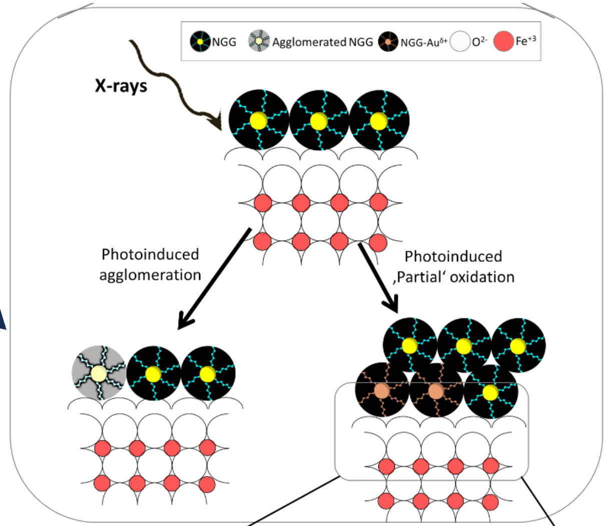
Chemical mechanisms at interfaces for painting conservation

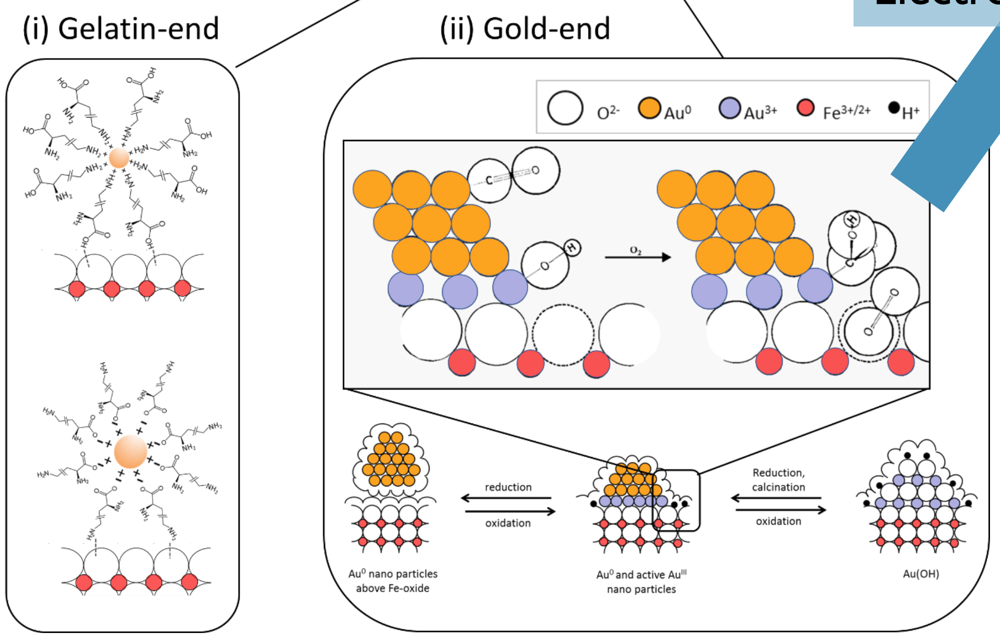




Atomic structure

"GE-XANES for chemical speciation at interfaces"
Maram Naes 2014, EXRS conference, Bologna





HESEB Helmholtz-SESAME Soft X-Ray Beamline for SESAME

Electronic structure

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Thank you for your attention

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