Gilded Nabataean Wall Paintings: Characterisation and Conservation

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Motivation

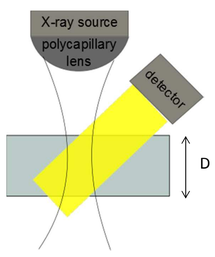
The few surviving Nabataean gilded wall paintings represent a highly significant yet understudied case in which gold layer is under constant loss. Developing a new conservation material for gilded multi-layered structures is our main aim. This requires clear understanding of strong and weak bonds between gold and its support. Our methodology includes investigating structure-property relationship of gold in multi-layered structures at various levels of interaction: macro, micro and nano for their elemental and molecular components in various layers and their contribution on structure's properties.

Instrumentation

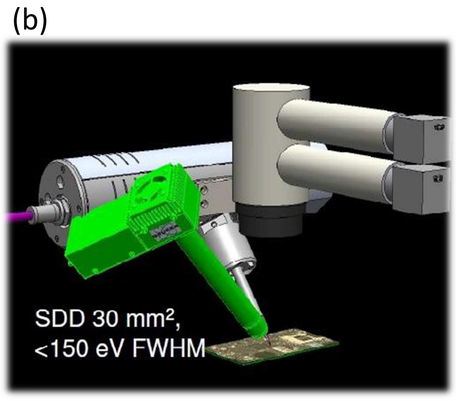
2D,3D-µXRF (M4 Tornado, Nano Bruker Analytics GmbH)

Schematic presentation of (a) 2D-µXRF principle, (b) Bruker M4 Tornado µXRF setup which is shown in (c). Setup components and parameters are described in Table (1).

(a)



(b)



(c)

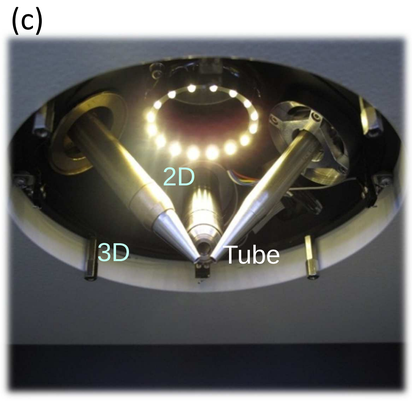


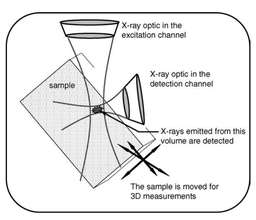
Table (1):

Source	Rh tube, 50 keV, 600 µA
Energy range	2 - 20 keV
Detector	SDD, 30 mm² Zr aperture, Resolution ~25 ± 1 µm
Energy resolution	<150 eV FWHM @Mn Kα
Optics (excitation channel)	Polycapillary full lens, < 25 µm for Mo Kα
Optics (detection channel)	3D: Polycapillary half lens, < 15 µm for Mo Kα
Geometry	45° between sample surface and excitation axis 90° between excitation and detection axes

2D,3D-µXRF (BLiX setup)

Setup schematic presentation of (a) 3D-µXRF principle[1], (b) BLiX prototype 2D,3D-µXRF spectrometer at TU-Berlin. Main components are numbered where 1) polycapillary lenses, 2) X-Ray tube, 3) SDD, 4) microscope, 5) motorised stage. In photo (c) a close up of the spectrometer's head showing the polycapillaries at both excitation and detection channels. Setup components and parameters are described in Table (2).

(a)



(b)

(c)

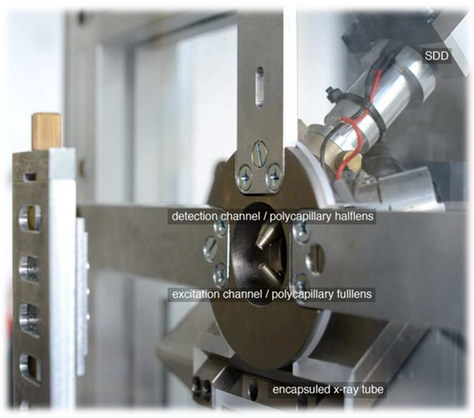


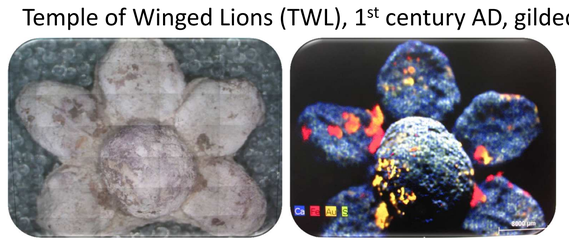
Table (2):

Source	Mo tube, 50 keV, 600 µA
Energy range	2 - 20 keV
Energy resolution	<145 eV FWHM @Mn Kα
Detector	SDD, 30 mm²
Optics (excitation channel)	Polycapillary full lens, Spot size 19.9 ± 0.7 µm @Cu Kα
Optics (detection channel)	Polycapillary half lens
Geometry	45° between sample surface and excitation axis 90° between excitation and detection axes

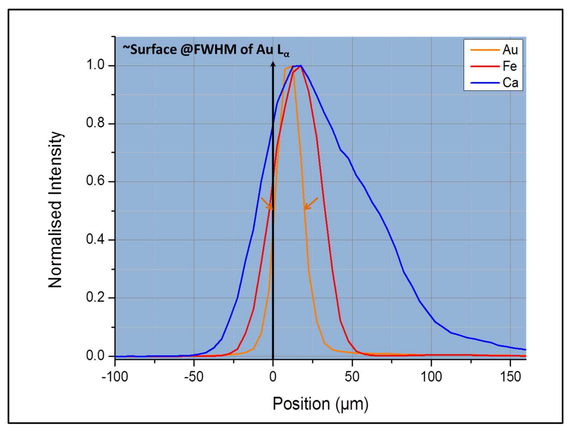
Results – *Work in progress*

Thirty four historic Nabataean samples (1st century AD) with gold layer remains are under investigation. Non-destructive and non-invasive qualitative and quantitative[2] 3D-µXRF analysis is being performed and one of the results is presented here. Reconstruction of original multi-layered structure (elemental composition, density and thickness) is work in progress.

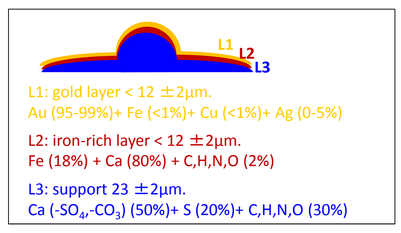
Temple of Winged Lions (TWL), 1st century AD, gilded gesso rosette



Left, Microscopic mosaic image of TWL7 sample as seen in M4 Tornado
Right, 2D-µXRF elemental mapping of TWL7 sample analysed with M4 Tornado. Mapped elements are represented in various colours. Scale is the same for both images. Measurement settings are 10 ms/pixel, 720 pixels, 25 µm spot size.

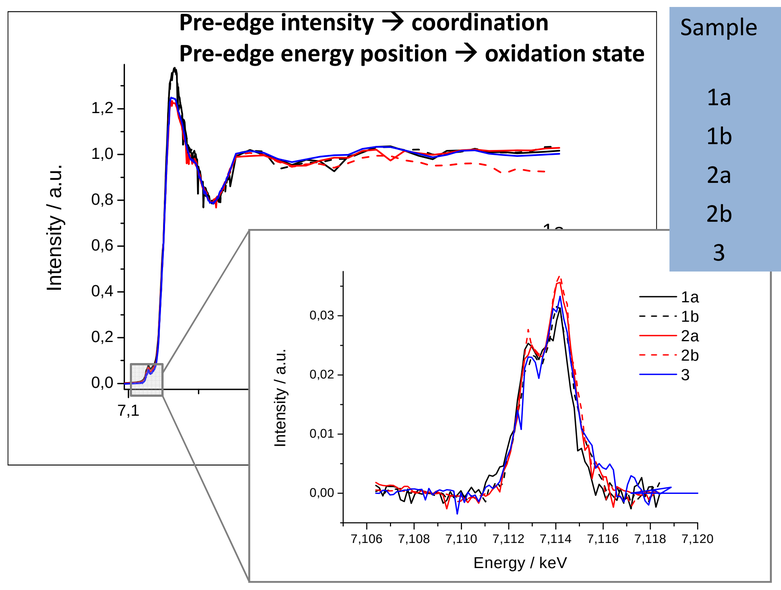


Left, Example of a depth profile analysis of the same sample above using BLiX 3D-µXRF setup. Measurement settings are 60 seconds/spot, 5 µm step size, 40 steps, ~20 µm spot size



After quantitative analysis of experimental data, reconstruction of layers thickness, composition and density became feasible. This however is a challenging appraoch and developing our methodology is work in progress. A successful reconstruction of the same sample TWL7 is schematised to the ***left***.

Then, chemical speciation of main elements per layer is considered. µXANES investigations at Fe Kα were performed in order to clarify the dependency of gold on iron and possible chemical bonding between both.



Sample	Integrated area / a.u. ± 0.01	Centroid Position / eV ± 0.05 eV
1a	0.07	7113.51
1b	0.07	7113.76
2a	0.08	7113.75
2b	0.08	7113.77
3	0.07	7113.81

1a,1b: Fe+Au, center
2a,2b: Fe alone, Leaf
3: Fe under Au, Leaf

Above, 2D-µXANES spectra of five points representing three Fe-Au visual states. From Fe-Kα pre-edge region, integrated area and centroid positions were obtained after fitting two Gaussian distributions to the background subtracted pre-edges.

Summary and Outlook

- The multi-layered structure of gilded Nabataean wall paintings was examined, characterised and reconstructed with non-destructive and non-invasive methods.
- Chemical speciation of iron in gilded samples was performed.
- No dependancy of Fe oxidation state on Au → Indirect or physical bonding
- Samples heterogenity and surface unevennes is a big challenge for real samples.
- Analysis of organic components is planned.
- Chemical speciation of Au is planned.
- Artificially destabilised replica are being prepared and its examination is envisioned.
- Development of a nano-based conservation material to regenerate the lost bond is work in Progress.

Contact

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[1] Kanngießer *et al.* 2003. A new 3D micro X-ray fluorescence analysis set-up - First archaeometric applications. *Nuclear Instruments and Methods in Physics Research* **B** 211, 259-264.
[2] I. Mantouvalou. Quantitative 3D Micro x-Ray Fluorescence Spectroscopy. PhD dissertation, Technische Universität Berlin, 2009.

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