ADVANCING X-RAY ANALYSES FOR PROBING BENEATH THE SURFACE OF ANCIENT ARTEFACTS

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INTRODUCTION

Cutting-edge instrumentation & methodologies to examine and model emerging "frontier objects" *— application to ancient pigments and decorative materials.*

Cultural heritage artifacts are complex : heterogeneous, fragile, unique, and often immobile. We offer innovative, non-destructive x-ray techniques, ranging from in situ measurements with portable instruments to high-res 2D mapping and tomography using synchrotron radiation.

The resulting large-scale datasets are processed through advanced statistical and machine learning tools for robust, quantitative insight.

Dejoie C., Martinetto P., Tamura N. 'Synchrotron Radiation, Cultural Heritage, Biomineralization' book 2025

Case Study 1 : Depicting Tin-relief Brocades

Polychrome decorations on wooden statues from the Duchy of Savoy (France), dating to the close of the Middle Ages.

Each microsample comprises multiple layers of varying thickness and composition — organic, inorganic, and metallic — exhibiting microstructures that span from amorphous to crystalline. Unlike ideal, lab-prepared materials, archaeological fragments are aged, heterogeneous, and often degraded, presenting significant challenges for accurate characterization.

Tracking variations in the stratigraphy of applied brocades reveals the distinctive signatures of multiple artists or workshops active in the Duchy of Savoy, thereby illustrating the diffusion of know-how and techniques during the late Middle Ages

P. Martinetto et al. J. Cultural Heritage 47, 89 (2021)
Exhibition at the Museum of Annecy (France), 2021-22

Case Study 2 : Illustrating Medieval Parchments

Inks and pigments in manuscripts produced at the Grande Chartreuse monastery during the 12th century, now preserved by the Municipal Library of Grenoble (France).

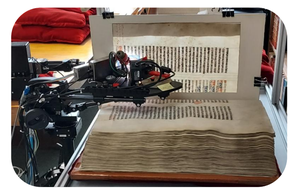
Objective physico-chemical analyses are added to traditional codicological, historical, and paleographic studies.

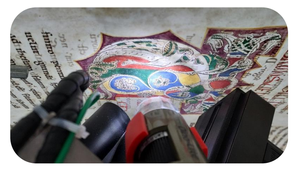
Using primarily diffraction and fluorescence, we examine the composition of writing materials and trace their evolution over time

→ Findings are contextualized within the work practices of the monastery and possible exchanges with nearby notaries and other monastic centers.

We demonstrate that scribes and illuminators, working individually in their cells, shared a common supply of materials — despite the absence of a centralized scriptorium.

H. Pasco postdoctoral research



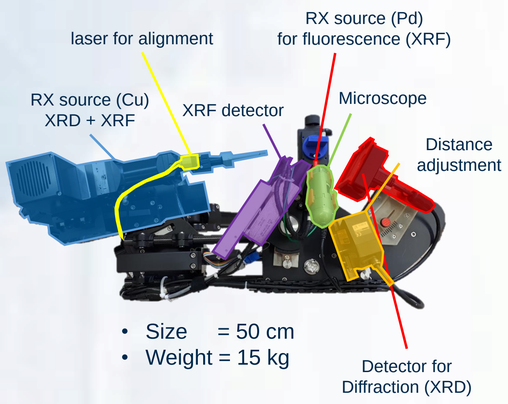


Instrumentation : Direct Examination of Artwork

We designed and built two mobile instruments that enable non-invasive characterization of objects (sculptures, paintings, documents, frescoes) using x-ray diffraction (XRD) and fluorescence (XRF).

Complementary studies (XRD/XRF-CT) on micro-fragments are performed at the BM02-D2AM CRG-ESRF beamline. Other techniques are used on layers that are too thin or non-crystalline (adhesives, varnishes).

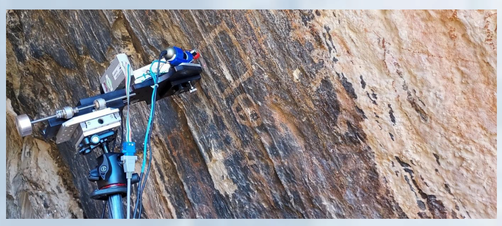
V. Poline *et al*. The European Physical Journal Plus, 138, 239 (2023)



- Size = 50 cm
- Weight = 15 kg

MobiDiff in front of a coffin preserved at the Grenoble Museum of Paintings (Ancient Egypt, Third Intermediate Period)

Thesis by C. Montembault, coll. ESRF



MobiFlu in front of the Neolithic parietal paintings of the Otello cave (Saint-Rémy-de-Provence, France)

Thesis by C. Théron, coll. EDYTEM

Analysis : Diffraction-based Imaging

1) Microsamples are complex mixtures of constituents at the micron scale

→ x-ray diffraction (XRD) is combined with computed tomography (CT) to generate large 3D datasets

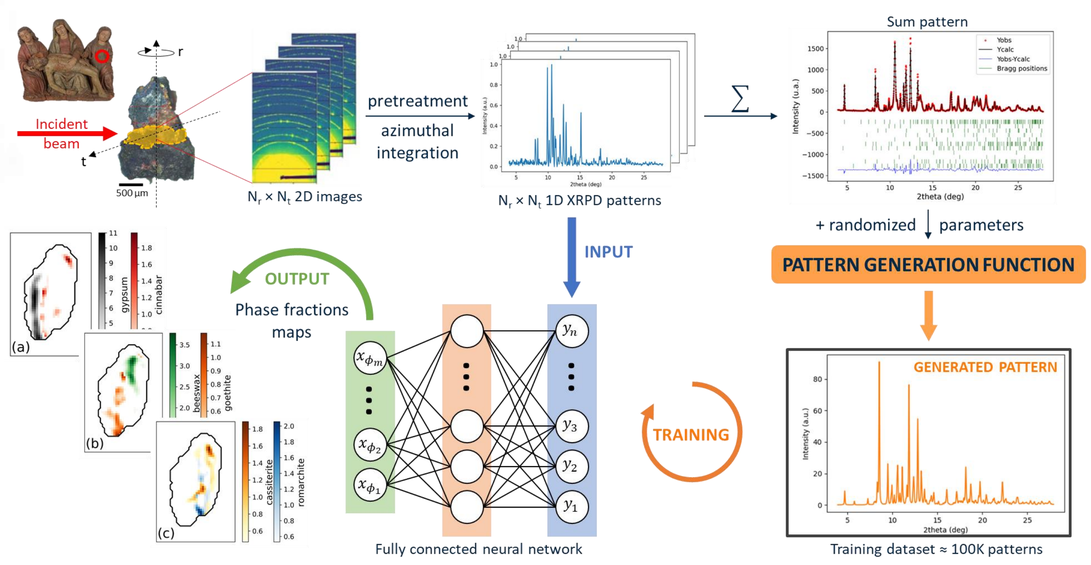
→ advanced statistical techniques, such as Multivariate Curve Resolution–Alternating Least Squares (MCR-ALS) and Non-Negative Matrix Factorization (NMF), help decompose the data into a limited number of physically meaningful components

→ supports phase identification through search-match algorithms, enables refinement of structural parameters, and allows determination of the mass proportions within the analyzed sample volume.

P. Bordet *et al*. Journal of Analytical Atomic Spectrometry 36, 1724 (2021)

2) Full-pattern fitting via the Rietveld method is enhanced by a Dense Neural Network trained on extensive experimental and simulated datasets, to ID the pigments and reveal their 3D distribution in paint coupons.

V. Poline Ph.D. dissertation, Grenoble University and J. of App. Crystal. 57, 831 (2024)



LOOKING FORWARD

The degradation of painting materials poses a significant threat to European cultural heritage. To safeguard irreplaceable masterpieces, museums and experts — chemists, physicists, conservators, restorers, and historians — collaborate to understand these works and develop long-term preservation strategies.

Our team contributes to several high-profile investigations by implementing an interdisciplinary, dual-pronged strategy :

a) Direct, in situ observation of full-scale heritage objects (manuscripts, statues, frescoes) using portable instrumentation.

b) High-resolution 2D/3D imaging of extracted microsamples using synchrotron X-ray probes.

We acquire a broad and diverse body of physico-chemical data through advanced analytical techniques, complemented by collaborative campaigns that integrate Raman spectroscopy, FTIR, and hyperspectral imaging. The resulting elemental, molecular, and structural data span multiple length scales — from millimeters to microns — and extend down to atomic-level modeling.

X-ray microscopy and spectroscopy techniques are effectively leveraged across a wide array of artefacts. To ensure a reliable interpretation, vast datasets are generated, that require robust workflows and data pipelines, built on advanced statistical methods and Machine Learning algorithms.