ESRF	**Experiment title:** COMBINATION OF MICRO-X-RAY DIFFRACTION AND MICRO-INFRARED SPECTROSCOPY FOR THE STUDY OF MULTI-LAYERED BUDDHIST MURAL	**Experiment number**: EC101
Beamline: ID21/FTIR	**Date of experiment**: from: 02/12/06 to: 05/12/06	**Date of report**: 17/04/07
Shifts: 12	**Local contact(s)**: M. Cotte	*Received at ESRF:*

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Report:

Purpose

The subject of this research was the analysis of a set of ancient Buddhist paintings, taken from Bamiyan, Foladi and Kakrak (highlands of Afghanistan), dating between 5 and 9th centuries AD. The purpose was to identify painting techniques through the characterisation of both pigments and binders, by combining µ-FTIR on ID21 and µ-XRD/ µ XRF on ID18F. Paintings usually have a complex stratigraphy consisting of both inorganic and organic compounds, therefore 2D-mapping was essential for both diffraction and FTIR microscopy. The micrometer beam was necessary to selectively probe the different layers. The interest of performing the experiments on ID21 and ID18F was the possibility of simultaneously mapping several compounds, even at low concentration, with a high spatial resolution. The combination of the two techniques was very useful for a complete characterization of inorganic/mineral and organic compounds.

Experiment

FTIR measurements:

The analyses were performed in transmission, with a beam spot of ~8×8µm^2. Samples were prepared in two ways:

i) Some fragments were embedded in resin for the preparation of cross-sections. Most of the fragments shown a remarkable capacity to be cut to produce thin sections, with few damage. Several thicknesses were obtained (from 5 to 50µm) using a microtome. Thinnest sections were more appropriate for FTIR study (Figure1, right).

ii) Some fragments of paintings were put on side and pressed in a micro-compression cell. This technique is rather "destructive" as the structure is not completely preserved but it has the advantage to completely avoid interferences from the embedding resin (Figure1, left).
Measurements were performed on both types of prepared samples, with the total of 21 different studied fragments.

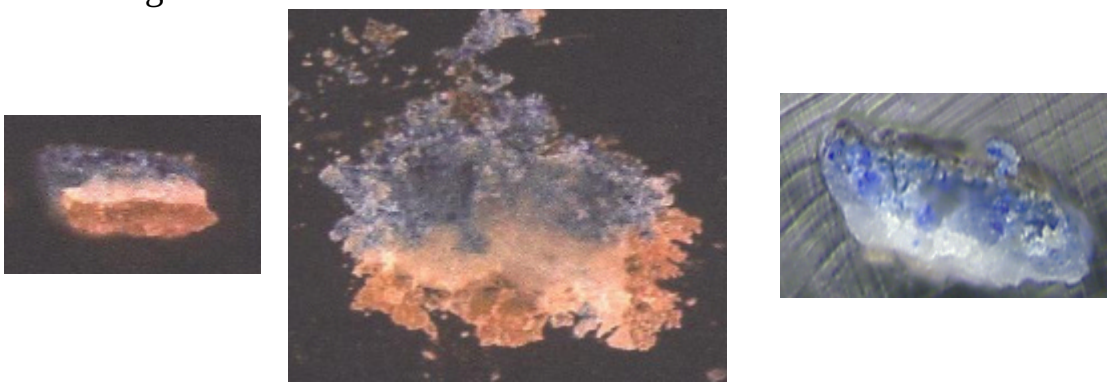

Figure1: Example of sample (BMM039), prepared by microtomy (right) or by simple pressure into the micro-compression cell, before compression (left), after compression (middle).

XRD measurements:
Micro X-ray diffraction and fluorescence were acquired simultaneously with a beam spot of ~18(hor.)×2.(ver.)µm^2, at an energy of 28keV. Diffraction was measured in transmission. Main of the analysis was conducted on cross-sections, over the whole stratigraphy of the samples (33 samples). Analyses were carried out on sections of 30 to 50µm-thick, trade-off between signal quality and stratigraphy resolution.
A tens of fragments was analysed directly, on powder particles without any preparation.

Results
FTIR measurements have revealed unexpected compositions with complex mixtures of organic substances (gums, resins, oils, proteins). In particular, a surprising preparation of oils and metal soaps was evidenced, whereas such practices are commonly said to have been used in Europe several centuries later.

The study of minerals by µXRD/µXRF is as interesting. A more delicate methodological approach was needed to treat data. First, fluorescence 2D-maps are fitted with the software PyMCA. In most of the cases, a good correlation is found between visible images of the cross-sections and elemental distribution. Then, such maps enable to define geographical regions of interest which are considered to calculate average diffraction patterns. Analyses are then conducted on these averages, to identify the main mineral composition of each painting layer. This 2-steps strategy is useful for the better understanding of diffraction pattern. A wide variety of pigments was identified, with the additional presence of alteration products. Compilation of all the results is still on going.

It was of high interest to image the distribution of compounds (atoms, molecular groups, phases) in transversal cross-sections. This way, contrary to macroscopic techniques which may mean the data on the whole sample, X-ray and FTIR microscopy enables to differentiate the successive painting layers as well as superficial pollution. Unique information could be obtained this way, and should be published soon.