EUROPEAN SYNCHROTRON RADIATION FACILITY
INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

Experiment Report Form

	Experiment title: Study of mordant degradation processes in gilding decorations by combination of micro-X-ray diffraction and micro-infrared spectroscopy	**Experiment number**: EC375
Beamline: ID21	**Date of experiment**: from: 21/09/08 to: 26/09/08	**Date of report**: 01/02/09
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Report:

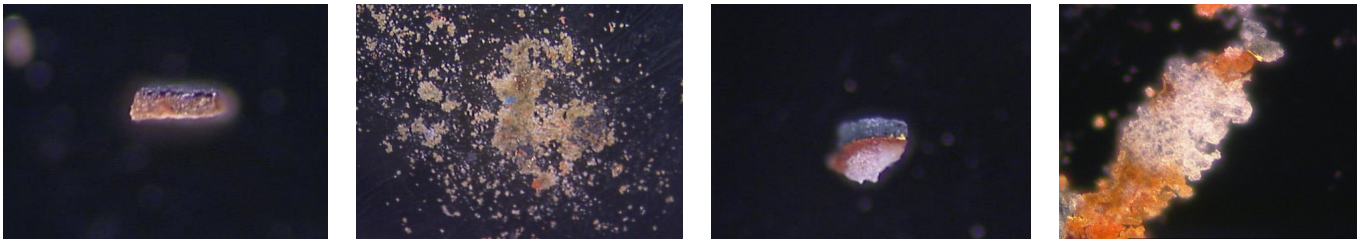
Purpose

This project arises from the collaboration between the university of Bologna and the Italian Superior Institute for Conservation ans Restoration (ISCR). The aim is the study of gilding decorations on mural paintings and the implications of metal soaps formation in mordant degradation processes using μFTIR available on ID21. The samples under study come from Giotto's frescoes of the Scrovegni Chapel in Padua (It) from 1305, which is considered one of the most important masterpieces of Western art and from the Serra Chapel in Rome (It, 1440) realised by Pellegrino Munari, a Raphael collaborator. Four different types of gilding have been distinguished according to their appearance: one has completely turned black, the other is still shining but as a discontinued layer, the third one is remained unaltered, the fourth contains a different mordant compound. Due to their complex stratigraphy and the thinness of the mordant layers, the combination of synchrotron beam with 2D-mapping has been interessant for identifying the nature of the inorganic compounds and organic substances present in low concentration, with a high spatial resolution.

Experiment

An first attempt was achieved to perform the analyses in attenuated total reflectance (ATR) mode on the samples embedded either in polyester resin or in potassium bromide (KBr) and prepared in cross-sections. The use of KBr as embedding material has been developed in our

laboratory in order to avoid the contamination of the embedding resin. However, after adjustement of the beam alignment with the ATR objective, the measurements did not succeded due to an energy's loss as soon as a contact was established with the sample's surface. In fact, the pressure achieved, even is low, may slightly change the position of the ATR objective of few micrometers, resulting in a beam out of focus.
Therefore, the analyses were performed in transmission, with beam spots from ~8×8μm^2 to ~5×5μm^2, on some selected fragments. After careful positioning on a diamond window following by pressing into a micro-compression cell, the stratigraphy is obviously damaged dur to the hardness of materials present in frescoes (figures 1a-b). Tests were made with three different samples with rather poor results. However, this preparation succedded with five samples collected from oil paintings (figures 1c-d). A gilt leather fragment has been also analysed in reflection mode.



a *b* *c* *d*

Figure 1: Mural paintings sample (14L), prepared by simple pressure into the micro-compression cell, a) before compression and b) after compression. Oil paintings sample (Fe2), prepared by simple pressure into the micro-compression cell, c) before compression and d) after compression.

Results

Regarding the samples 18L, 3L and 14L collected from Giotto's frescoes of the Scrovegni Chapel, FTIR measurements have confirmed the results obtained from previous conventional laboratory techniques, such as optical microscopy, FTIR (Fourier transform infrared spectroscopy) and SEM (scanning electron microscope) with a coupled energy dispersive X-ray spectroscopy (EDS) facility. In particular, the use of the anvil diamond cell has modified the paint structure and did not allow evaluating the distribution of the organic subtances within the different layers.
Regarding the samples MCR13, MCR8, Fe2, Rond13, ICOGblu, the higher spatial resolution and brightness of SR sources available at ERSF have allowed us to perform the measurements on small areas with a good signal-to-noise ratio. Either inorganic compounds and organic subtances, such as proteins and oils, have been characterised and localised within the stratigraphy through the use of 2D-mapping. The reflectance measurements achieved on the gilt leather's surface has permitted the characterisation of the varnish as well as the alteration products. The results will be compared with those obtained with conventional techniques, in a PhD dissertation on the application of FTIR microspectroscopy to cultural heritage.
From our point of view, further efforts should be made in collaboration with company dealing with FTIR instruments in order to develop ATR accessories adapted for the synchrotron beam. Once this improvement will be achieved, we have not the slightless doubt that the studies of brittle samples, which require to be embedded and analysed in cross-sections, will be greatly enhanced using SR sources.