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The new ID21 XANES full-field end-station at ESRF

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Abstract. A new X-ray absorption near-edge spectroscopy (XANES) full-field imaging station has been developed, installed and tested on beamline ID21 at the European Synchrotron Radiation Facility (ESRF). The set-up operates in the 2-9 keV energy range and allows for the simultaneous acquisition of up to $4 \cdot 10^6$ XANES spectra over large sample areas with preserved sub-micron spatial resolution. The versatile set-up is compatible with various types of cameras and magnifying objectives. It accommodates spatial resolutions ranging from 0.3 μm to 1.4 μm and fields of view from 600 μm up to 2 mm. The range of potential applications is broad: from geology, cultural heritage, environmental sciences to medicine.

1. Introduction

The ID21 X-ray microscopy beamline at the ESRF is dedicated to scanning X-ray fluorescence microscopy and micro-XANES applications in the 2-9 keV energy range, using focussing optics like Fresnel Zone Plates (FZP) or KB mirrors to produce a sub-micron X-ray probe [1]. It is optimized for submicron XANES analysis performed in fluorescence mode on traces or minor elements of small samples. Though it can also be used to analyse larger and more concentrated samples, it turns out that in these cases the micro-XANES scanning strategy is not efficient enough to provide reliable 2D speciation mapping within a reasonable amount of time: even with a spectrum acquisition time of 10s per pixel, it would take more than 100 days to record a 1000 x 1000 pixels XANES map.

Actually, for heterogeneous materials, the redox information can be obtained from three kinds of XANES-based mapping techniques: (1) the most widespread consists in scanning mapping mode with a microbeam for an energy corresponding to a resonance peak of the probed element [2]. The contrast variations are correlated in first approximation to the redox ones. Nevertheless, this method does not provide spectral data and the field of view is limited by the dwell time ($> 0.1\text{s/pixel}$); (2) energy dispersive EXAFS [3] allows for the acquisition of a full EXAFS transmission spectrum at once and per pixel but it is still not fast enough to get large fields of view (dwell time $> 1\text{s/pixel}$) and the achievable resolution is by nature of a few microns; (3) the method used in Transmission X-ray Microscopy involves 2D detector able to record in one shot an entire absorption image. Soft X-ray

full-field microscopes based on FZP are commonly used for high resolution imaging, but their combination with spectroscopy is challenging and 2D chemical mapping still difficult. For harder X-rays, lensless X-ray microscope based on simple propagation geometry is easier and can be readily used for 2D spectromicroscopy. However, these instruments are principally dedicated to tomography whereas XANES applications are scarce [4].

The new XANES full-field imaging station at ID21 is based on the latter approach. It is designed to perform submicronic XANES analysis in transmission mode on major elements of large samples. For each energy point across a given K- or L-edge, a magnified 2D transmission image of the sample is acquired by a camera coupled to an X-ray scintillator and magnifying visible light optics. Then, a “flat field image” recorded with sample out of the x-ray beam is used for normalization. The vacuum-air interface is realised through a viewport inserted between the scintillator and the visible light objectives. A XANES stack consists of a series of normalized images that characterize the sample absorption across the absorption-edge of interest. The XANES spectra can then be extracted for each pixel of the image.

A first “in air” prototype was successfully tested and used to derive quantitative mapping of the ratio of Fe^{3+} over total iron content in complex metamorphic rocks [5]. The new improved set-up is vacuum compatible to extend the range of available K-edges. The data collection is more efficient and the accessible data processing capabilities have been enlarged. It makes ID21 full-field end-station an analytical tool fully complementary of the ID21 scanning x-ray microscope (SXM) [1].

2. Experimental set-up

2.1. Technical design

The full-field chamber is connected to the ID21 SXM chamber through a vacuum valve. Sample changing is fast and only requires venting and pumping of the full-field chamber.

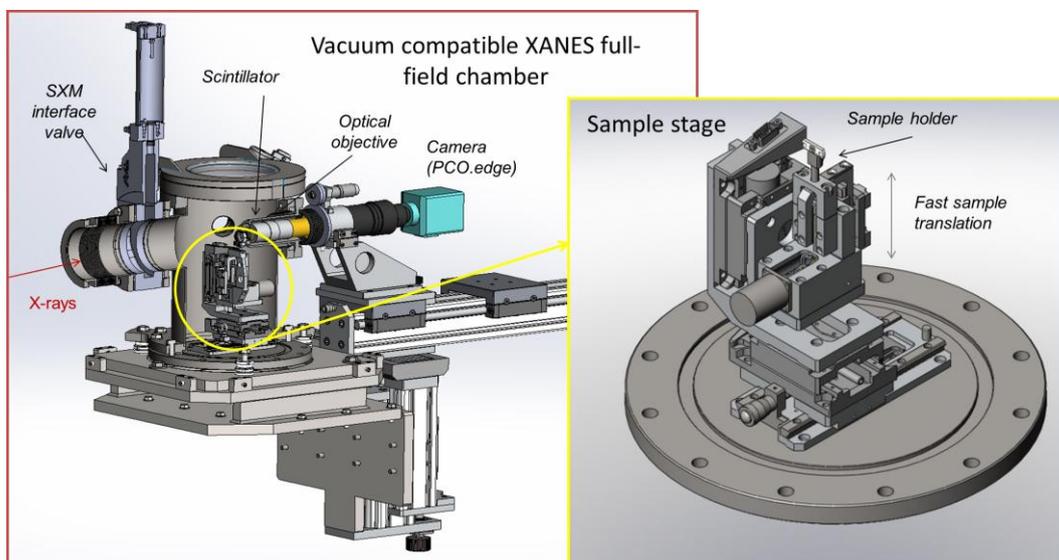


Figure 2. Schematic of ID21 full-field XANES chamber (left) and sample stage (right).

The experimental set-up uses ID21 fixed-exit double-crystal monochromator equipped with both Si(111) and Si(220) crystals to tune the beam energy, an X-ray decoheror to blend the X-ray incoming beam spatial features, a sample holder and a set of optics and cameras to detect transmitted X-rays, as described in [5]. In the new set-up, the decoheror used to smooth the spatial structures of the beam is located 1 meter upstream from the sample position. It consists of a 125- μm thick graphite foil (10 μm

grading) whose speed of rotation can be adjusted from 1 to 50 Hz. The sample stage consists of two translation stages (26 mm range in the horizontal and vertical directions) on top of which a fast vertical translation is mounted. The fast vertical sample motion is used to move out the sample from the x-ray beam for flat field acquisitions, and then to move it back in front of the beam (the escape range is ~ 10 mm). The scintillator ($\text{Lu}_2\text{SiO}_5\text{:Tb}$, 10 μm thick) is located less than 2 mm from the sample to limit phase contrast features generated by the sample heterogeneities. The support of the scintillator is directly mounted on the viewport holder that ensures the air-vacuum interface. The viewport profile has been calculated to minimize the degradation of the Modulation Transfer Function that is found to be nearly negligible. A 2D detector coupled to a long working distance optical objective (Mitutoyo) records the magnified transmission image of the sample. The set-up is compatible with both scientific CMOS PCO.edge camera and in-house developed CCD FReLoN E2V camera. The sample holder is also compatible with a small rotation stage (SmarAct) that can be used to perform polarisation contrast imaging.

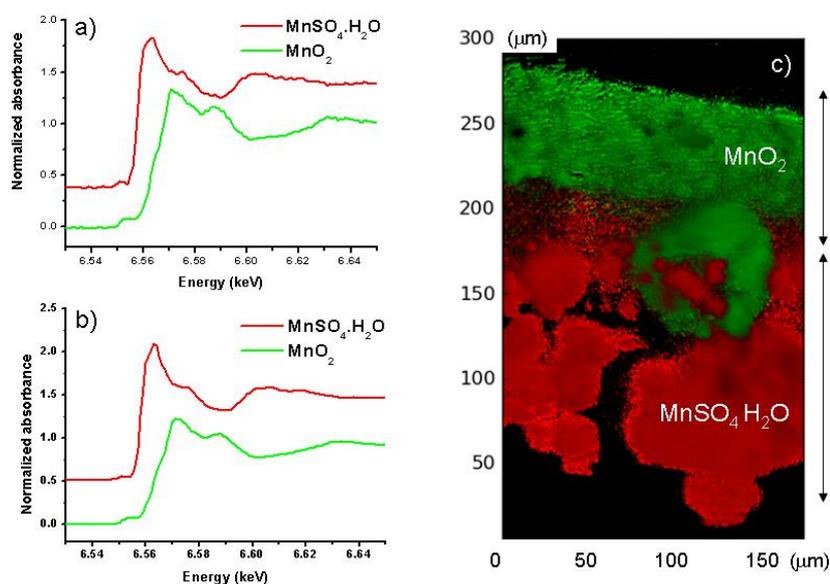


Figure 2. Results of a full-field acquisition showing the XANES spectra obtained from a) the arbitrary selection of a single pixel in each of the two manganese layers of the sample and b) the automatic selection of two wider regions (10^3 to 10^4 pixels) containing only pixels with similar absorption levels in a chosen spectral region of interest (ROI); c) 2D oxidation state mapping calculated after spectral ROI selection on the normalized spectrum of each pixel. This procedure allows for highly sensitive and quantitative 2D mapping of the two manganese species.

2.2. Data acquisition and pre-processing

Typically, series of about 300 radiographies are recorded over ~ 200 eV, with variable energy steps according to the XANES spectral regions and the monochromator spectral resolution. When compared to more classical x-ray transmission spectroscopy set-up, the final signal to noise ratio (SNR) of the spectra is mostly limited here by i) the movements of the sample due to repositioning after flat field acquisition and ii) the movements of the x-ray beam, since the flat field images are not acquired simultaneously with the sample transmission images.

To cope with the first limitation, an accurate procedure of realignment of the normalized images based on Fast Fourier Transform 2D image correlation has been implemented. The achieved precision for sample realignment is better than 0.1 pixel. To reduce the influence of beam motion or instabilities over the final XANES spectra SNR, several measures were taken: i) the use of a decoheror to smooth the beam spatial heterogeneities; ii) the use of a fast sample translation to reduce the time interval between flat-field acquisitions and sample image acquisitions; iii) the implementation of an “accumulation mode” for fast multi-frame 2D image acquisitions, *ie* the dynamic of the camera is artificially increased by acquiring several consecutive transmission images of the sample or several consecutive flat field images, each of them being only separated by the readout time of the camera; iv) the possibility of acquiring flat field images before and after sample images for a more accurate normalization.

The data pre-processing step that consists in image normalization and sample realignment is performed in the EDNA framework [6]; it allows for efficient online data pre-processing. The processing step that consists in XANES spectra normalization and statistical analysis (principal components analysis, positive matrix factorisation, etc) of the spectra over the 2D images is realized thanks to new development of the in-house PyMca software [7].

3. First experimental results

In the framework of a project aiming at studying the mechanisms of ageing of archaeological paintings, first tests have been carried out to evaluate the capabilities of the XANES full-field set-up to map the oxidation state of painting pigments as a function of their depth inside the painting layer. For the feasibility trial, biphasic painting samples were specially designed. They were composed of two layers, the first one based on manganese dioxide (MnO_2) pigment and the second one based on manganese sulphate (MnSO_4) pigment; the organic binder was linseed oil. The samples were then embedded in a resin (HistoResin, Leica) to be cut at a thickness of 30 μm . This preparation allows for studying the painting cross-section with a good preservation of the sample spatial structures.

The beam was collimated by a 1mm x 1mm aperture and the Si(111) monochromator was tuned around 6.55 keV. The PCO.edge camera coupled to a x10/N.A 0.42 objective gave a final pixel size of 0.65 μm . In these conditions, the saturation time for the camera was measured to be 50 ms. Several acquisition parameters were tested to optimize SNR vs total acquisition time. The result for a “fast acquisition mode” (dwell time set to 40 ms, three frames acquired at each energy point) is shown in figure 2. For clarity, only a selected region of the sample is shown, however the full-field acquisition was actually performed on a 0.8 x 0.5 mm^2 region and took 40 minutes. The edge jumps of the sample were measured between 0.1 and 0.7 and XANES spectra with good signal to noise ratio could be obtained with only one sample absorption image and one flat field image. Using up to 10 frames per energy point (400 ms of acquisition time per 2D image) was found to improve the SNR per pixel. Above this number of frames, the total experiment time would increase while the spectra SNR would not and could even decrease because of beam drifts not well corrected in the normalization procedure.

4. Conclusion

A fully vacuum compatible XANES full-field set-up in the 2-9 keV energy range was successfully developed and tested. Although it was not tested at low energy, the first results obtained at the manganese K-edge are very promising. Further development regarding improved data transfer efficiency and more sophisticated image normalization are under investigation. The set-up is well adapted to the study of heterogeneous systems, at various scale-levels and with chemical and structural heterogeneities. Many fields of applications could benefit from this new tool: medicine (e.g. bone remodelling studies, speciation of exogenous particles involved in tissue disorders), ancient materials (e.g. evolution of metals in pigments, glasses and metallic artefacts), environment (e.g. transport of metals in plants) and geological applications which were at the origin of the project.

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