



HAL
open science

The ID21 Scanning X-ray Microscope at ESRF

M. Salome, M. Cotte, R. Baker, R. Barrett, N. Benseny-Cases, G. Berruyer,
D. Bugnazet, H. Castillo-Michel, C. Cornu, B. Fayard, et al.

► **To cite this version:**

M. Salome, M. Cotte, R. Baker, R. Barrett, N. Benseny-Cases, et al.. The ID21 Scanning X-ray Microscope at ESRF. 11th International Conference on Synchrotron Radiation Instrumentation (SRI), Jul 2012, Lyon, France. 4 p., 10.1088/1742-6596/425/18/182004 . hal-01572759

HAL Id: hal-01572759

<https://hal.science/hal-01572759v1>

Submitted on 8 Aug 2017

HAL is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers.

L'archive ouverte pluridisciplinaire **HAL**, est destinée au dépôt et à la diffusion de documents scientifiques de niveau recherche, publiés ou non, émanant des établissements d'enseignement et de recherche français ou étrangers, des laboratoires publics ou privés.

The ID21 Scanning X-ray Microscope at ESRF

M Salomé¹, M Cotte^{1,2}, R Baker¹, R Barrett¹, N Benseny-Cases¹, G Berruyer¹, D Bugnazet¹, H Castillo-Michel¹, C Cornu¹, B Fayard^{1,3}, E Gagliardini¹, R Hino¹, J Morse¹, E Papillon¹, E Pouyet¹, C Rivard¹, V A Solé¹, J Susini¹ and G Veronesi¹

¹European Synchrotron Radiation Facility, 38043 Grenoble Cedex 9, France

²Centre de Recherche et de Restauration des Musées de France, UMR 171 CNRS, Palais du Louvre, 75001 Paris, France

³CNRS, Laboratoire de Physique des Solides, Université Paris Sud, 91405 Orsay cedex, France

salome@esrf.fr

Abstract. The ID21 Scanning X-ray Microscope (SXM) is optimized for micro-spectroscopy with submicron resolution in the 2 to 9.5 keV energy range. After a brief description of the microscope setup, we present here recent developments, in particular, the latest version of the compact Wavelength Dispersive Spectrometer and the refurbished cryo-stage.

1. Introduction and general beamline layout

The ID21 beamline at the European Synchrotron Radiation Facility (ESRF) is dedicated to Scanning X-ray Microscopy (SXM) in the 2 to 9.5 keV energy range and Fourier Transform Infrared (FTIR) micro-spectroscopy. Combination of these complementary micro-analytical techniques is of great interest for the correlative investigation of complex heterogeneous samples. Applications cover a broad variety of scientific fields, ranging from biology, to geochemistry, environmental sciences, materials sciences and archeometry.

ID21 is a 50 m long beamline, located on a 4.8 m long low-beta straight section of the ESRF ring. It is presently equipped with two undulators (U42 and U40) and a wiggler (W80). The first or the third harmonics of the undulators are used depending on the required energy range. Harmonic rejection (10^{-3}) is ensured by means of a fixed-exit double mirror system deflecting in the horizontal plane. The grazing angle of the mirrors can be tuned from 7 to 20 mrad and different reflective coatings (Si, Ni or Rh) allow the beam spectrum to be tailored to the needs of a given experiment. The branch hosting the Scanning X-ray Microscope endstation is equipped with a Kohzu fixed-exit double crystal monochromator. Sets of Si(111) crystals ($\Delta E/E=10^{-4}$) and Si(220) crystals ($\Delta E/E\sim 10^{-5}$) are routinely used for μ -XANES experiments. A set of two Ni/B4C multilayers ($\Delta E/E\sim 0.01$) is also available for experiments requiring higher flux but moderate energy resolution of the incident beam.

2. The Scanning X-ray Microscope setup

The ID21 Scanning X-ray Microscope (SXM), shown in Figure 1, allows micro-X-ray fluorescence (μ XRF) and X-ray Absorption Near Edge Structures spectroscopy (μ XANES) measurements to be performed with submicron resolution. The SXM underwent a major refurbishment in 2008, which was followed by continuous developments over the last few years to improve further its performance and capabilities. These efforts have resulted in a versatile and reliable instrument optimized for efficient

and user friendly operation. The microscope can host two different optical focusing configurations: either zone plates (ZP) or a Kirkpatrick-Baez mirror system (KB), which routinely achieve a typical spot size of $0.3 \times 0.7 \mu\text{m}^2$ (V x H) with a photon flux of 10^9 - 10^{10} photons/s in the 2-9 keV energy range. A modular optics stage including all the needed degrees of freedom and providing a kinematic mount allows a reproducible switching between the two optical modes. ZPs routinely in use at ID21 were purchased from Zone Plates Ltd (UK) and NTT (Japan). The KB mirror system, shown in Figure 1, was developed at the ESRF and is based on elliptically figured fixed-focus Ni coated mirrors (Zeiss, Germany). Its compact design ensures high mechanical and thermal stability and a short focal length for high source demagnification. The higher photon flux and the achromaticity of the KB configuration are particularly suitable for micro-spectroscopy.

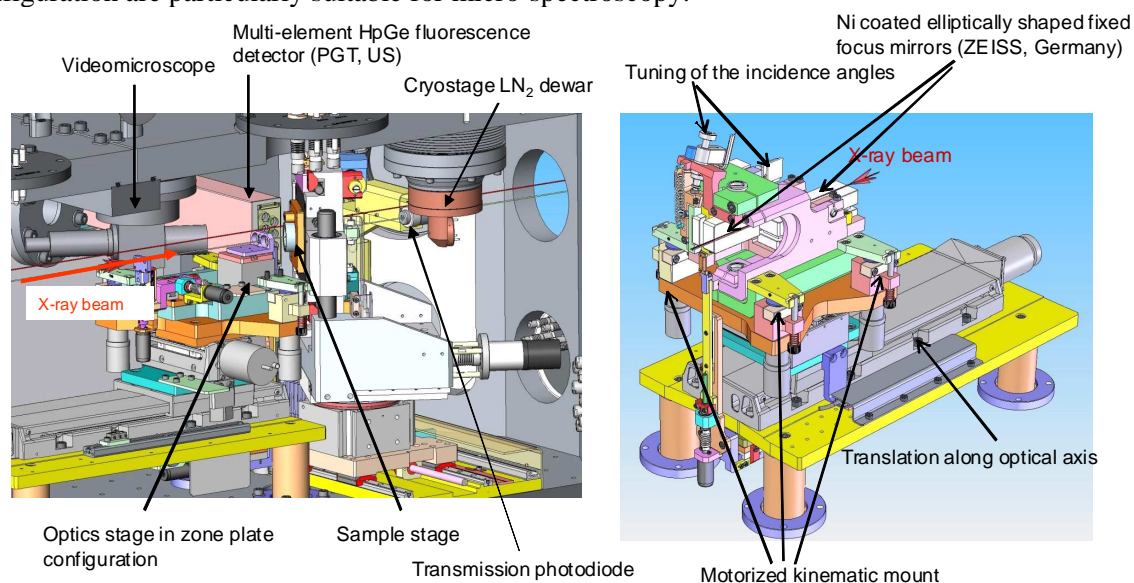


Figure 1. Inside view of the SXM (left). The KB mirrors mounted on the SXM optics stage (right).

The sample stage, which includes stepper motor actuated stages with micron-range resolution and a piezo-stage (PI, Germany) for finer resolution scans, has also been completely redesigned to accommodate various sample environments; In particular, the newly refurbished cryo-stage which is described in further detail in section 3. A load-lock now allows fast transfer of samples into the SXM vacuum chamber, and greatly facilitates operation in cryogenic conditions.

A major refurbishment of the fluorescence detection capabilities of ID21 has also been completed. Two complementary approaches were implemented: (i) improvement of the sensitivity of trace-element mapping by increasing the detection efficiency and collection solid angle of energy dispersive detectors (EDS). This motivated the purchase of a 7-element HpGe detector (Princeton Gamma-Tech, US) and a large area (80 mm^2 collimated active area) Silicon Drift Diode (Bruker, Germany). (ii) providing elemental detection with high specificity by improving the energy resolution. A compact Wavelength Dispersive Spectrometer (WDS) achieving a few tens eV energy resolution was developed for highly selective fluorescence detection [1][2]. Its latest version is described in section 4.

Control of the SXM is performed through a fully integrated graphical user interface which includes on-line visualization of the sample through a video-microscope and direct “grabbing” of regions to be scanned from a video image. Fast “on-the-fly” data acquisition during continuous scans has been developed and is used both for 2D mapping with collection of a full XRF spectrum for each pixel, and XANES energy scans with synchronization of the undulator gap, the monochromator angle and the ZP working distance. In terms of data processing, XRF data analysis is facilitated by the development at ESRF of the freely distributed PyMca package which can batch process large 2D data sets [3].

3. Cryo-microscopy

Cryo-microscopy is essential for the micro-analysis of radiation-sensitive samples, such as most biological samples. Freeze-drying sample preparation is an alternative, but may modify the ionic distribution and the chemical speciation of the element of interest in the sample. Hence, cryo preservation is preferred and allows the analysis of the sample in a stable hydrated near-native state, limits its oxidation by air during its manipulation (which is performed in a dry N₂ atmosphere) and preserves its morphological integrity under the beam.

A vibration-free cryo-stage, passively cooled by a liquid nitrogen (LN₂) dewar has been developed in that purpose. In cryo configuration, a copper cold finger is mounted on the sample stage and linked via copper braids to the cooled base plate of the LN₂ dewar (see Figure 2). This “floating” copper braid heat exchanger provides adequate thermal conductivity whilst maintaining sufficient mechanical freedom to ensure stress free sample scanning. The cold finger is thermally insulated from the sample stage mechanics by a Polyetheretherketone (PEEK) support. The sample holder is machined in a copper block providing sufficient thermal inertia during sample transfer. Samples are usually deposited on TEM grids or Si₃N₄ thin windows and kept in LN₂. Their mounting is therefore performed in cryo conditions, above a LN₂ bath in which the basis of the sample holder is plunged. The sample is deposited on the holder, maintained by a drilled copper plate and covered with a thin polymer film (Ultralene, Spex-Certiprep) which limits its sublimation. This stack is maintained by a “clip” cover plate which facilitates mounting. The sample holder geometry allows micro-spectroscopy measurements both in fluorescence and transmission modes. The sample holder is inserted in the load-lock chamber of the microscope and, after pumping, introduced inside the fixed cold finger. This transfer typically takes 2 min. The conical shapes of the cold finger and sample holder ensure a good thermal contact between them. Initial cooling of the cryo-stage requires 1 hour before settling in thermal equilibrium with a 100 K temperature on the cold finger. A 130 K temperature was obtained on the sample holder exclusively cooled by the cryo-stage and starting from room temperature. Lower temperatures (not measurable for technical reasons) are certainly achieved in normal operation, with the sample holder already LN₂ cooled. The LN₂ dewar offers an autonomy of 3.5 hours and is regularly refilled by means of a LN₂ tank connected via vacuum insulated tubes. A semi-automatic refilling procedure can be manually activated or integrated in data acquisition macros.

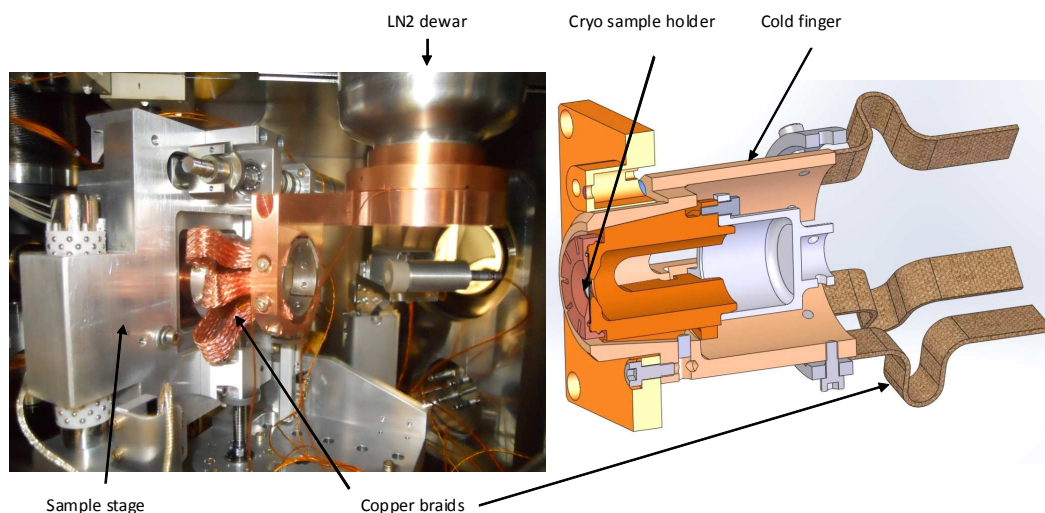


Figure 2. Picture of the cryo-stage mounted on the backside of the SXM sample stage (left) and design of the cold finger and cryo sample holder (right).

4. New compact Wavelength Dispersive Spectrometer

A new, even more compact version of the WDS was recently designed and commissioned. The device can now be permanently mounted in the SXM and facilitates the switching from multi-elemental

mapping with EDS detectors to elemental specific micro-spectroscopy. The ID21 compact WDS is based on a polycapillary collection optic (XOS, US) which is placed a few millimeters from the sample and collimates the divergent X-ray fluorescence emitted from the sample into a quasi-parallel beam, which is then directed onto a flat crystal at the required Bragg theta angle. The X-rays diffracted by the crystal are counted by a gas-flow detector (Parallax, US) placed at the 2-theta angle. Both crystal and detector are mounted on rotation stages, synchronized in theta/2-theta angular position, to adjust the incidence angle on the crystal to the required wavelength. In order to cover an x-ray energy range between 1.4 keV and 9 keV, the spectrometer is equipped with several different crystals mounted on a rotatable turret.

This device allows the selection of a given fluorescence emission line with high energy resolution and ensures an efficient rejection of unwanted scattering and fluorescence background; hence allowing the detection of trace elements even in a highly fluorescence emitting matrix. WDS is also a very powerful detection scheme to separate neighboring emission lines of different elements (typically weak L-lines overlapping with strong K-lines), which EDS detectors are unable to resolve. It is thus perfectly suited for micro-XANES measurement with high selectivity and low background [2].

A series of x-ray fluorescence spectra were measured from reference compounds. The obtained experimental resolution versus the measured photon energy is presented in Figure 3 as a function of the employed crystal. The energy resolution of the WDS is in the range between 4 eV to 40 eV.

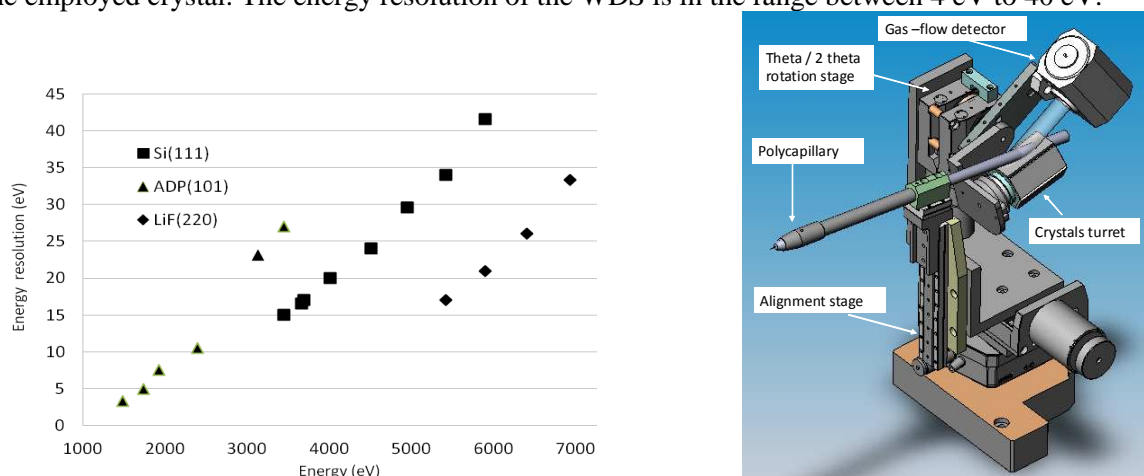


Figure 3. Measured energy resolution of the spectrometer equipped with ADP(101), Si(111) and LiF(220) crystals (left). Design of the WDS system (right).

5. Perspectives

To complete the panel of micro-analysis techniques available at ID21, two projects are in progress (i) a setup for parallel beam full-field XANES imaging has been installed in a dedicated chamber downstream of the SXM [4][5] (ii) a micro-diffraction endstation optimized for 8.5 keV is under development on the ID21 lateral branch.

References

- [1] Szlachetko J, Cotte M, Morse J, Salomé M, Jagodzinski P, Dousse JC, Hoszowska J, Kayser Y and Susini J 2010 *Journal of Synchrotron Radiation* **17** 400-408
- [2] Cotte M, Szlachetko J, Lahlil S, Salomé M, Solé VA, Biron I and Susini J 2011 *Journal of Analytical Atomic Spectrometry* **26** 1051-1059
- [3] Solé V A, Papillon E, Cotte M, Walter Ph and Susini J 2007 *Spectrochim. Acta B* **62(1)** 63-68
- [4] De Andrade V, Susini J, Salomé M, Bérardin O, Rigault C, Heymes T, Lewin E and Vidal O 2011 *Analytical Chemistry* **83** 4220-4227
- [5] Fayard B *et al.* this volume