

On SOLEIL beamlines and support laboratories, equipment keeps on evolving, according to the needs and expectations of the scientists. This new section aims at briefly introducing the latest setups, with a particular focus on two of them. For this first edition we look to DEIMOS and HERMES.

A new X-ray microscope at SOLEIL

Exploring the matter at a nanoscale

HERMES is a beamline dedicated to soft X-ray microscopy. The originality of HERMES is to combine two different approaches (STXM & XPEEM) on the same beamline with the goal to reach spatial resolution below 20nm.

While the XPEEM is already operational and open to the users since several years, the STXM microscope is a newly developed setup that has been recently delivered and installed at HERMES beamline.

The STXM microscope (Scanning Transmission X-ray Microscopy) takes advantage of the high coherence of the synchrotron beam to extremely focus the photon beam down to few tenths of nanometers using a dedicated diffractive optics. The beam is scanned across the sample and the transmitted photons are collected to form a magnified image of the scanned area.

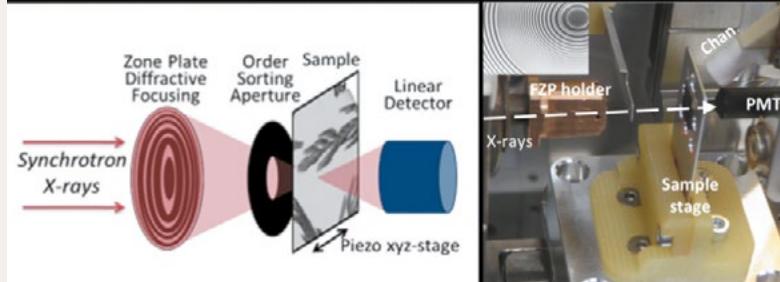
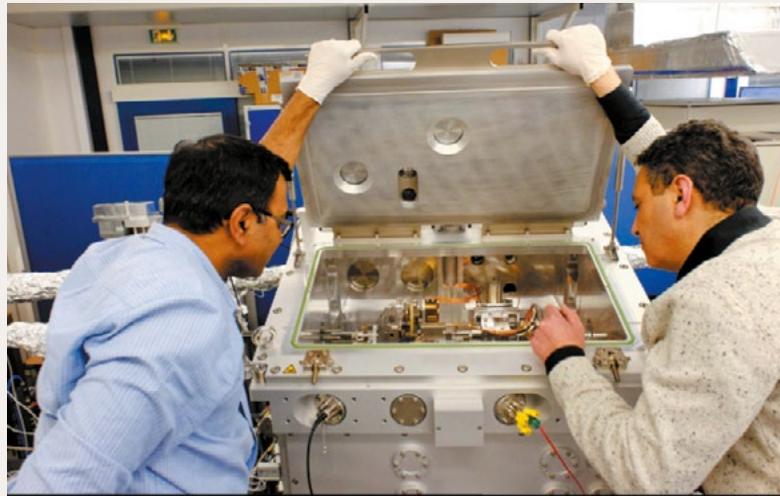
STXM is probably among the most suitable methods to respond to the growing demand to explore heterogeneous matter. Besides taking advantage of the unique properties of synchrotron X-ray, it allows to suitably combine spectroscopic and microscopic methods. Finally, the STXM allows to operate with a versatile sample environment: liquid samples, high magnetic and electrical field, cryo... which allow in turn to cover a very broad field of applications: Magnetism, soft matter,

environmental & biological science... The STXM microscope has been fully commissioned and all the targeted specifications have been demonstrated in terms of spatial and energy resolution, flux, stability...

The microscope is now fully operational and open to the users.

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Figure 1. Up: The STXM microscope installed at the HERMES beamline. **Down, left:** Schematic scanning transmission X-ray microscope (STXM) A Fresnel Zone Plate (FZP) is used to focus the x-rays in the sample plane. To generate an image, the sample is raster-scanned under interferometer and computer control. The transmitted X-rays are collected using a PMT placed behind the sample plane. **Down, right:** View of the internal scanning stages of the STXM microscope.



A new Ultra Low Temperature device on DEIMOS

*IPCMS: Institut de Physique et de Chimie des Matériaux de Strasbourg
*IMPIC: Institut de Minéralogie, de Physique des Matériaux et de Cosmologie (Paris).

References:

[1] https://en.wikipedia.org/wiki/Dilution_refrigerator; P. Sainctavit and JP Kappler, Magnetism and Synchrotron Radiation, Lecture Notes in Physics, Springer, p. 235 (2001)

[2] I. Létard, P. Sainctavit, Ch. Cartier, J.P. Kappler, P. Ghigna, B. Doddai, J. of Appl. Phys. 101, 113920 (2007)

The magnetic properties of matter depend essentially on two parameters, namely the temperature (T) and the magnetic field (H). The higher the H/T ratio, the more the ground state system will be accessible. From an experimental point of view, increase of this ratio by increasing H is quickly bounded within the limits of static fields (around 15 teslas), on the contrary decrease T by a factor of 20 is technically feasible. Industrial companies propose cryostats with few dozen of mK for set-up but without of course the constraints of Synchrotron Radiation (SR) environments. In the SR centers, on an X ray magnetic circular dichroism (XMCD) beamline, one can generally get a H/T ratio of about 1. The current DEIMOS CroMag is characterized by a ratio of 5 and the Dichro50 project aims a target ratio of about 100, by developing a cryogenic insert able to perform magnetic dichroic measurements at T=50 mK (- 273,05 °C).

The peculiarities of XMCD devices at Ultra Low Temperature (ULT)

The use of ^3He - ^4He dilution refrigerator [1] to produce ULT is quite widespread for standard measurements, but the XMCD set-up requires superconducting split coils (high magnetic fields), electrical insulation of the sample, optical access, adaptations to ultra-high vacuum and in situ sample transfers, making the XMCD device developments at ULT more difficult. History - Through associations initiated 20 years ago, at the time of LURE -before SOLEIL was built- with researchers and engineers of IPCMS* and IMPIC**, ULT devices dedicated to magnetic dichroism have been designed and implemented. They still hold the world record of low temperature (T limit=300mK) for XAS experiments, thanks to the SOLEIL-TBT device installed since 2006 on the SIM beamline at SLS (Switzerland), dedicated mainly to molecular magnetism studies.

The originality of the new DEIMOS device

For X-ray absorption experiments at low energy and ULT, the sample must be both electrically isolated and in contact with the cold source. The scientists have invented and validated the use of a sapphire ring which separates the mixing chamber in two parts; that allows filling the electrical insulation criterion without adding thermal impedance: the sample holder is in direct contact with the He bath (^3He - ^4He mixing). It was shown, through the observation of expected physical phenomena [2], that the temperature of the sample was indeed that of the helium bath, and then that measured by the thermometer. This is the key strength of the new DEIMOS device and a promise of success.

After a period of feasibility studies (2015), designing and building of the insert have begun in June 2016. The commissioning under X-ray beam is scheduled for early 2017.

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Dispersive CD on DISCO

The DISCO beamline consists of 3 experimental hutches, including one for synchrotron radiation circular dichroism (SRCD). Until now, the technique used was «classic» CD: absorption by the sample of circularly polarized light, first anti-clockwise, then clockwise, measured wavelength by wavelength. It takes about five minutes, using this method, to create a complete spectrum.

By using the naturally circularly polarized properties of synchrotron radiation, circulating clockwise above the orbit of the electrons and anti-clockwise below, it is possible to record in one go the clockwise and anti-clockwise components of the absorption by

spectrography. The only limit to the measurement speed relates then to the detection chain, i.e. of the order of one microsecond per complete spectrum. This technique, known as dispersive CD, is in the process of being installed: the intensifier and the camera are in place, the spectrograph is working and polarimetric measurements have been carried out. From the beginning of 2017, this device will make it possible, for example, to follow the «live» folding of an entire protein in solution.

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Dichro50 project

The project involves the construction of a device with an expected temperature limit of about 50 mK on the end station of DEIMOS, thanks to technology and know-how of Synchrotron SOLEIL transfer to the French CryoConcept company. In the framework of knowledge transfer, this project was selected for a SATT Paris-Saclay investment as well as others prestigious programs (LabEx, PALM, ASTRE). Moreover, the project got the supports from ten European laboratories inside the DILUX consortium.

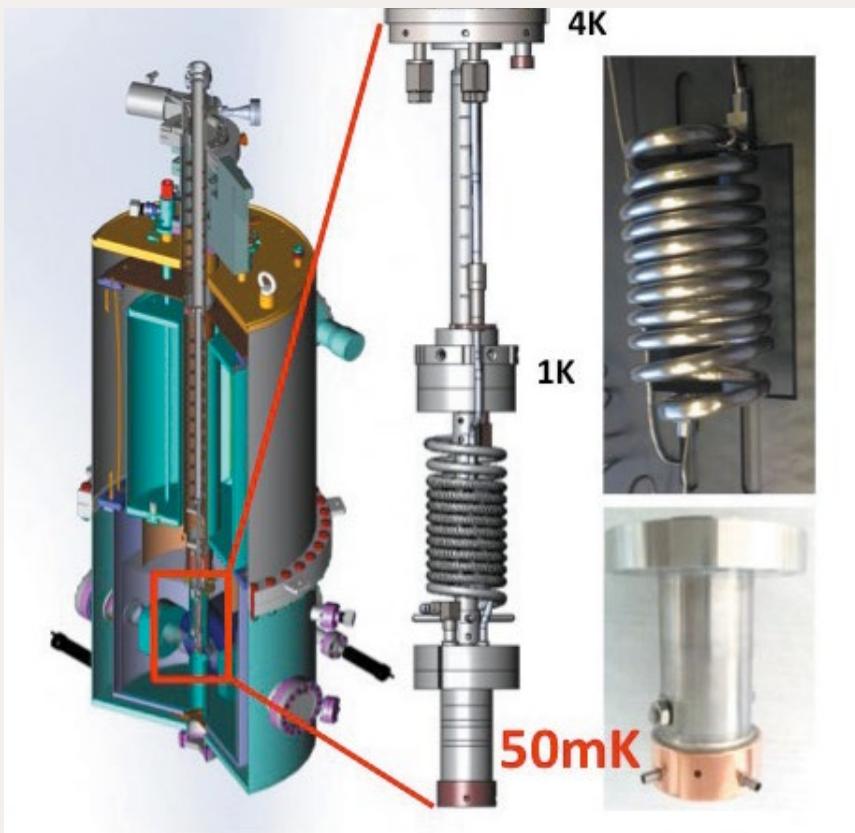


Figure 1 - 3D projection of DEIMOS cryomagnet, details of the low temperature stages of the device; right: pictures of the components before assembly (courtesy of Cryoconcept)

Scientific cases

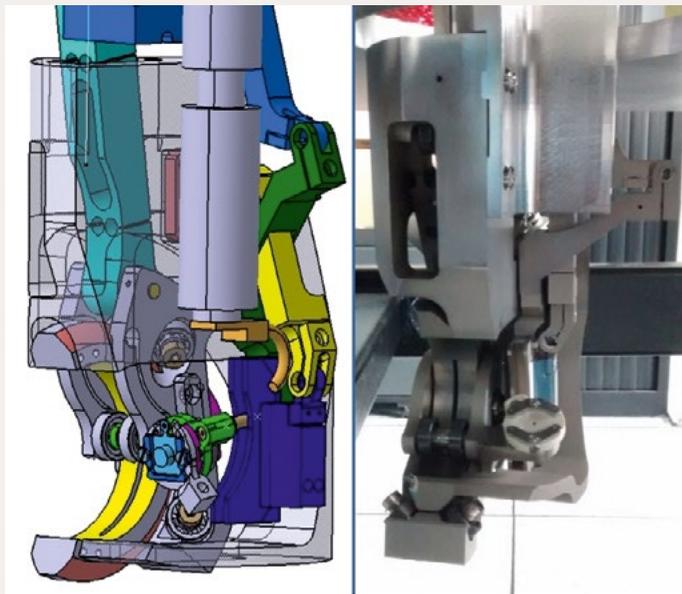
Magnetic studies of molecular magnets (Rayon de SOLEIL #25 (2015), page 4), strongly correlated systems, isolated paramagnetic ions, super-paramagnetism clusters, magnetic ordering in organic radicals or superconductor gaps are at the heart of the scientific community concerns which require Ultra Low Temperature (ULT) devices. These scientific topics take advantage of the specificities of the magnetic dichroism, the main tool of the DEIMOS beamline.

Continuous scans in reciprocal space on SIXS

SIXS is dedicated to studying the structure of surfaces, interfaces or nano-objects, using X-ray scattering / diffraction techniques. This beamline makes it possible to study samples prepared under vacuum or in different environments (reaction chamber, electro-chemical cells, Langmuir trough, etc.). Measurements can be taken in situ, in operando and in real time. SIXS has a two-dimensional hybrid detector coupled to a system of automatic attenuators, which allow data to be recorded over a very large range, with the detection response remaining linear throughout. Data acquisition is carried out using the «FLYSCAN» protocol (see Rayon de SOLEIL 22, page

9), which opens up the possibility of acquiring data in reciprocal space at unprecedented speeds. This type of scan allows you to exploit the accuracy of the diffractometer encoders by ensuring very high resolution measurements. In addition, these very fast scanning protocols can be used to measure evolving systems over very short time intervals. This new acquisition method will be available to users in 2017.

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Left: 3D view of the new CASSIOPEE manipulator.
Right: photo of the device taken in December 2016.

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Manipulator on CASSIOPEE

One of the two branches of the CASSIOPEE beamline, dedicated to photoelectron spectroscopy, is equipped with a high-resolution angle-resolved photoemission setup. The measurements consist then in collecting electrons as a function of their emission angle. To do this, the sample is attached to a manipulator which allows to orient it with respect to the (fixed) electron analyzer as well as to control the temperature of the sample. The present manipulator rotates the sample only about a vertical axis, and can cool it down to about 5 K. To increase performance and to make measurements easier, a new manipulator is being installed, developed in the framework of the collaboration between SOLEIL and MAX IV. It will provide three rotations: about the vertical axis, about a horizontal axis contained in the surface of the sample, and about the normal to the sample surface. The minimum temperature will be 9-10K. First tests are planned for January 2017.

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NanoIR on SMIS

The nanoIR2s microscope technology is based on the coupling of an atomic force microscope (AFM) with an infrared (IR) source, with the aim of carrying out nanoscale spectroscopy and imaging. The main advantage of this approach is to push the limits of resolution which, for conventional IR microscopes, means a few microns. The nanoIR2s microscope includes two different physical approaches to obtain absorption measurements, one photothermal (developed by the

A. Dazzi group, Paris-Sud) and the other using optics. In both cases, the idea is to probe the surface of a sample with an AFM tip (with a 10 nm radius of curvature) and to measure its local absorption when irradiated with IR radiation. In the first approach, known as AFM-IR, the AFM tip records the dilation of the sample when the sample heats up by absorbing the incident wave. The repetition frequency of the IR source is tuned to the resonant frequency of the AFM tip, enhancing the sensitivity by several orders of magnitude and allowing the measurement of objects only a few nm thick. In the second approach, scanning near-field optical microscopy, or SNOM, uses the tip as an antenna to concentrate the incident electromagnetic field and backscatter a carrier wave of chemical information. This technology has already proved its worth and it is now possible to analyze objects as small as protein assemblies (amyloid fibrils) or even to detect nanoparticles inside macrophages.

The possibility of obtaining a vibrational (IR) spectrum on a nanometric scale opens the door to an incredible number of applications, and in all fields of science (biology, materials science, agronomy, chemistry, photonics, etc.). The commercial system generally works with a tunable laser that does not cover the full IR range and is often very unstable energy-wise. The SMIS project is to couple the nanoIR2s microscope to the synchrotron radiation source, in order to measure IR spectra over a range that no laser based source can provide and thus gain access to more detailed analysis of matter at the nanometric scale.

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Protein purification in the biology laboratory

To purify macromolecules in solution prior to their analysis on beamlines, the Biology Laboratory has an HPLC system from Agilent Technologies with a size exclusion column (size-separation): HPLC-SEC. This technique is calibrated with globular proteins. For non-globular, rod-like or, on the contrary, more compact molecules, the retention time cannot be straightly used to evaluate their molecular weight. Recently, a multi-angle laser light scattering detector (MALLS) and an index refractometer (RI), were coupled to the HPLC-SEC. These make it possible to evaluate the molar mass of each eluted molecule, independently of the retention time on the column and of the «theoretical» molar extinction coefficient (calculated for a protein from its amino acid sequence). This combined instrument-environment has already shown its effectiveness, notably on a protein for which the elution profile more corresponded to a dimer, but was actually found to be a monomer. This can therefore provide essential structural information, complementary to that given by SAXS or SRCD. For molecules that are modified (glycosylation), ramified proteins (with aliphatic chain) or membrane protein, their interpretation may be more complex; this expertise is being acquired.

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