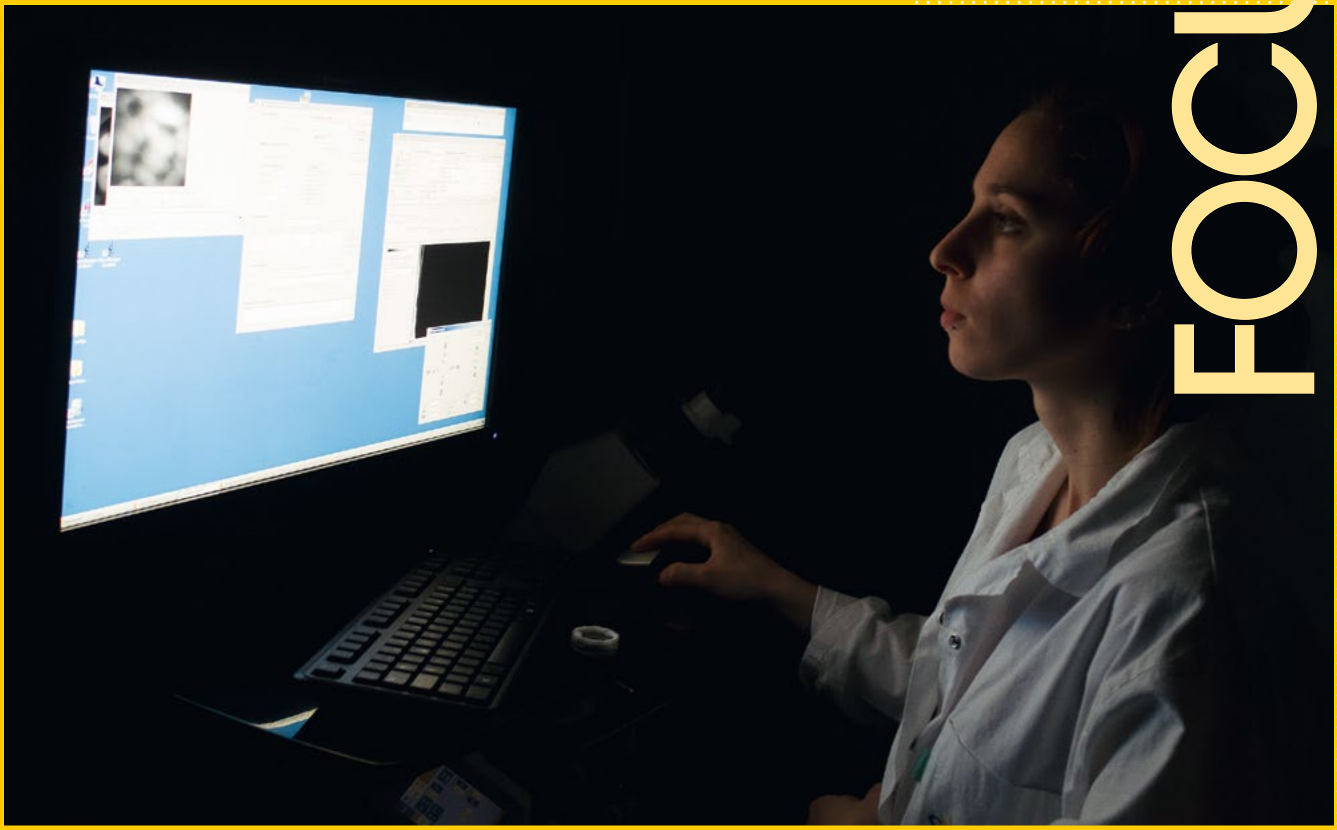
2- and 3-dimensional imaging



Very early on, Man felt the need to access what his eyes could not see. Diving into the infinitely small—from the first lenses and magnifying glasses, followed by the very first microscopes by the end of XVIth century and finally today's transmission electron microscopes—over time, the resolution of the images obtained has gained many orders of magnitude. With resolutions now reaching a few hundred picometers (10^{-12} m), scientists are able to detect details as small as two separate carbon atoms in a diamond.

At SOLEIL, imaging techniques are available on about half of the 29 beamlines, and this proportion is set to increase in the years to come.

HERMES

Weaving 3D magnetic memories using nanowires

There is a tremendous need for high data storage capabilities, which continues to increase exponentially these last years. Requirements other than the amount of storage also include, depending on the application: access speed, endurance, volatility, energy for writing / reading a bit and the cost per bit. Within the framework of an EU project (M3D), several European laboratories are involved to address materials for non-volatile mass storage, where the density and cost per bit are the key issues. The original approach of the project is to envision 3D magnetic memories using magnetic nanowires: series of bits would be shifted along vertical magnetic wires densely packed in arrays, requiring only one single read/write element per wire.

Understanding the magnetic properties in a single magnetic nanowire and nanotube is a first and mandatory step toward the full design of such devices. We have recently carried out XMCD-PEEM observation of such nanotubes at HERMES beamline. To the best of our knowledge this is the first direct observation of a clear domain pattern in magnetic nanotubes. However, contrary to the predictions, we evidence in these tubes orthoradial magnetization with multiple well-defined domains. Such tubes with orthoradial magnetization are appealing for a 3D race-track memory as their domain walls give rise to a very small dipolar field, unlike the case of nanowires, which warrants absence of cross-talk (magnetic interaction between the nanotubes) despite the dense matrix.

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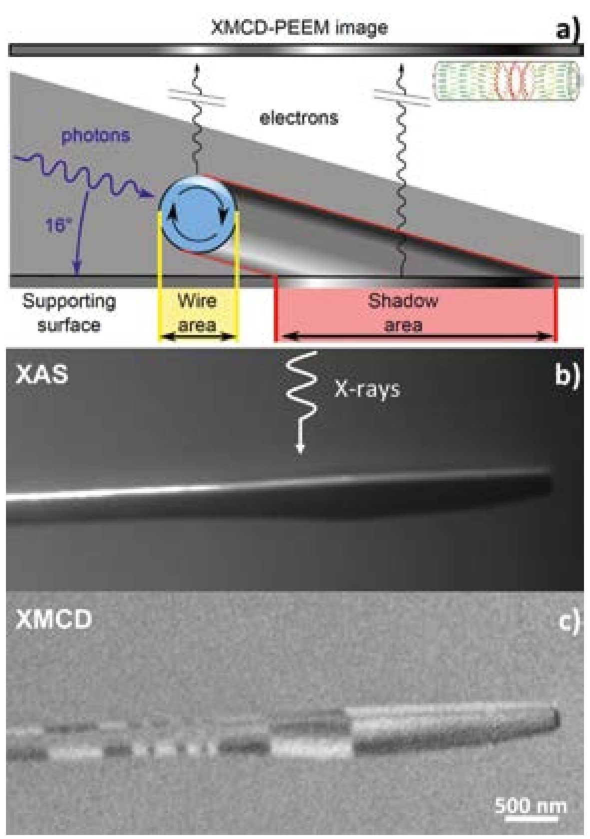

Figure 1 : High resolution XPEEM image of a CoNi tube. a) Schematic of the geometry of XMCD-PEEM used in a combined surface/transmission mode. The nanotube is lying on a supporting surface, which gives rise to a shadow of the tube from the 16° grazing-incidence beam. The analysis of the helicity-dependent intensity in the shadow provides information on the magnetization distribution in the bulk of the object. b) XAS-XPEEM image recorded at the Ni edge. The bright region corresponds to the tube while the dark one is the shadow of the tube. c) XMCD-XPEEM image at the Ni edge evidencing the surface (top) and the bulk (bottom) magnetic domain structure.

The microscopy techniques that the scientists use to achieve the highest resolutions[1] do not rely on visible light, as is the case with optical microscopes. Instead, they use other ranges of electromagnetic radiation, or even electrons. In addition, the images no longer form directly in the eye of the experimenter: they must be «reconstructed» in order to be interpreted. The reconstruction methods include one or more stages, from the signals recorded by detectors that are no longer our eyes. Several imaging techniques exist depending on the type of radiation employed and the interaction between the radiation and the investigated object: absorption, emission, diffraction, to name a few.
Although for the time being, electron microscopy remains unbeatable in terms of image resolution, the synchrotron techniques are making great progress by achieving resolutions of a few tens of nanometers, while providing additional data revealed by the interaction of radiation with matter. Moreover, in the case of imaging techniques based on electromagnetic radiation, a synchrotron naturally offers the largest variety of techniques as a result of the wide spectrum of photons and diversity of analytical devices currently available.
SOLEIL makes it possible to explore the various scales of matter, from the micron to the nanometer, and to collect information that come as a complement to the "topographic" data recorded by a microscope, such as the magnetic or electronic properties of a material, or its chemical composition.

[1]Resolution: The minimum distance between two contiguous points so that they are correctly distinguished by an observation or measurement system..

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NANOSCOPIUM

MMX-I: a software for multi-modal X-ray imaging

Scanning hard X-ray imaging allows simultaneous acquisition of multimodal information, i.e. of images in which each pixel contains several types of data. The output is, for instance, a map of the sample in absorption, phase and dark-field contrasts and X-ray fluorescence, providing with a single scan both structural and chemical details.

Combining this scanning technique with the FLYSCAN infrastructure developed for fast data acquisition at Synchrotron SOLEIL [1] permits to perform multimodal tomographic imaging and tomographic reconstruction during routine user experiments at the NANOSCOPIUM 155m long beamline [2]. A computerized analysis reconstructs the 3D inner structure of the observed object from these tomographic data, which are a large set of images of the sample in different orientations.

A main challenge of such imaging techniques is the online processing and analysis of the important amount of generated multimodal data. This is particularly important for the wide user community working at the user oriented NANOSCOPIUM beamline (e.g. biology, life sciences, geology, geo-biology), having sometimes no experience in such data-handling.

MMX-I is a new and unique multi-platform open-source freeware for the processing and reconstruction of scanning multi-technique X-ray imaging and tomography datasets [3]. The MMX-I project aims to offer both expert users and beginners the possibility of processing and analyzing raw data, either on-site or off-site. Therefore we have developed a multi-platform (Mac, Windows and Linux 64 bit) data processing tool, which is easy to install, comprehensive, intuitive, extendable and user-friendly. A dedicated data-input stream copes with the input and management of large datasets (several hundred of Giga Bytes) collected during a typical multi-technique fast scanning and this even on a standard PC. The data of each scanning imaging technique (included into the software) or any of their combinations can be treated by MMX-I. Experienced users can use this program as a library for their existing software or develop new functions, which are then released for other users. MMX-I is now routinely used by the NANOSCOPIUM users community either on-site or off-site and has demonstrated its effectiveness in dealing with big data.

MMX-I is available online at the following address: https://bitbucket.org/antoinebergamaschi/mmx-i

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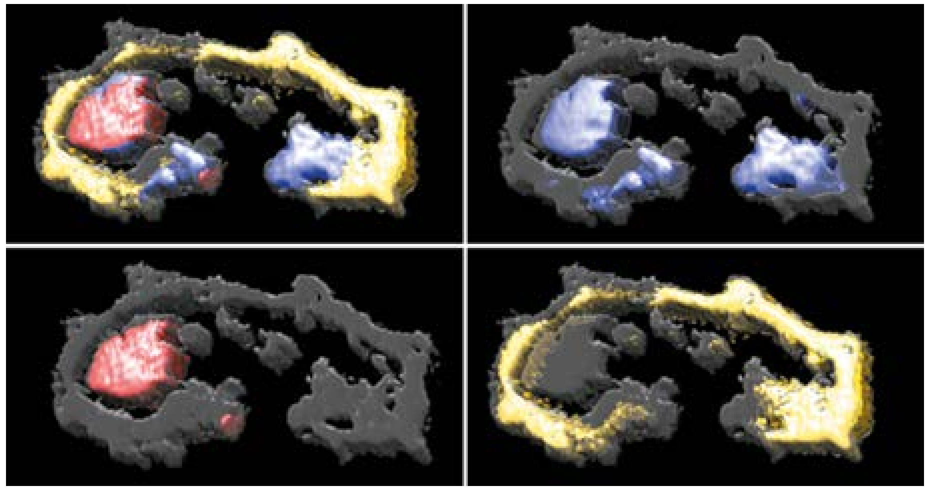
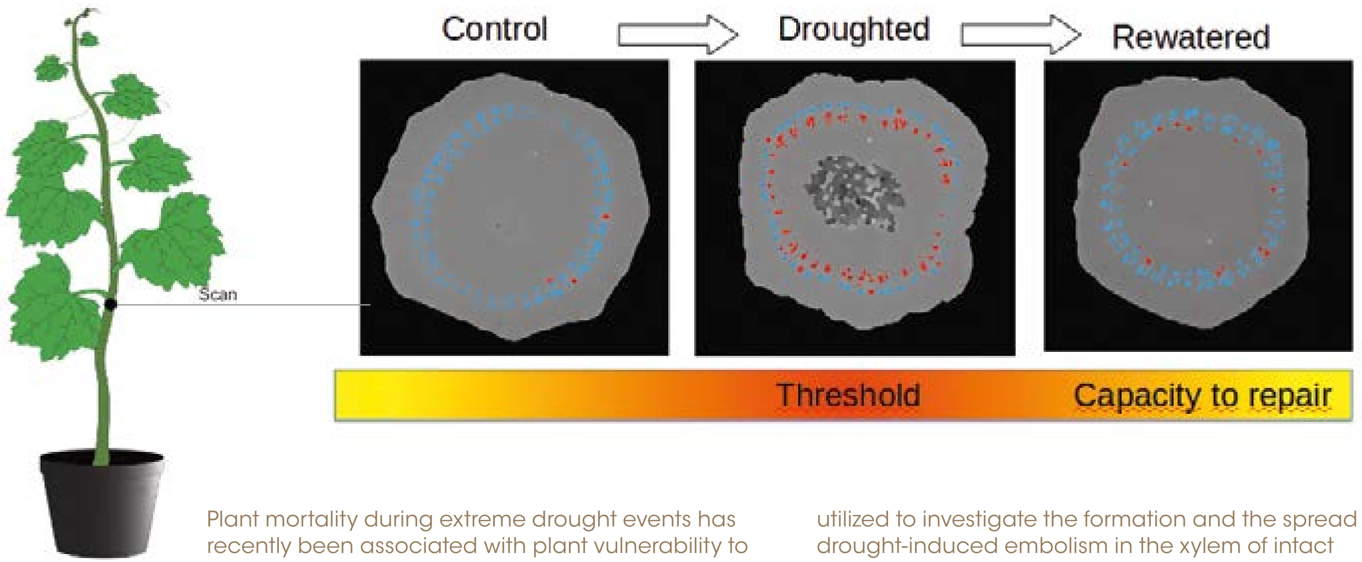


Figure 1: 3D volume rendering of the multimodal tomogram of a fossil single-cell foraminifera. Scanning multi-technique tomography provides simultaneous structural and elemental information for the study of heavy metal incorporation mechanisms during bio-mineralization processes. The different contrast modalities were reconstructed by the MMX-I data-processing software developed at the NANOSCOPIUM beamline. The reconstructed phase is shown in grey, the dark field is in blue, the iron distribution is in red and the calcium distribution is marked in yellow in the figures.

References:
[1] K. Medjoubi *et al.* In X-Ray Nanoimaging: Instruments and Methods, (2013/09/26), Proc. SPIE 8851:88510P. San Diego, California, United States. doi:10.1117/12.2026680.
[2] A. Somogyi *et al.*, Journal of Synchrotron Radiation (2015) 22, 1118.
[3] A. Bergamaschi *et al.*, Journal of Synchrotron Radiation (2016) 23, 783.

PSICHE

Plant embolism and drought resistance



Plant mortality during extreme drought events has recently been associated with plant vulnerability to xylem cavitation, a phenomenon corresponding to the disruption of water transport in embolized vessels. Despite the recent advances in the field of plant hydraulics, there is still debate as to whether plants routinely face embolism and recover easily from it or are highly resistant to embolism. Some studies have suggested that plants are highly vulnerable to embolism but recover from it on a daily basis. In both branches and leaves, this recovery would involve the refilling of vessels, via transpiration during the daytime or through root pressure. However, this process has been observed only in species with long vessels, such as laurel, poplar and grapevine, possibly reflecting methodological issues that bias conventional measurements in species with long xylem pipes. Synchrotron based micro tomography has been utilized to investigate the formation and the spread of drought-induced embolism in the xylem of intact plants and test whether plants are thus routinely exposed to high levels of embolism even when well-watered.

During their first SOLEIL campaign in 2015 [1], scientists from BIOGECO (INRA – Bordeaux University) first demonstrated how high resolution computed tomography (HRCT) - a non-invasive method, which allows the direct observation of air and sap-filled xylem conducting elements in the wood of intact plants - can provide non biased assessment of plants adaptation to drought. Their direct observations of vessel have evidenced the remarkable ability of long vessels such as oak to resist embolism. Second, in 2016, they evidenced xylem refilling and an increase in the number of functional vessels after the plant was re-watered (Figure 1). Refilling of xylem vessels was not observed as long as the pressure remained negative. However, as soon as positive pressure was measured, xylem refilling could be seen. They also demonstrated that the most living cells remained alive (Figure 2) 10 days after scanning.
Part of the activity of the PSICHE beamline is tomography and imaging for a wide range of applications, including the example shown. The synchrotron source provides very high x-ray flux, much brighter than a laboratory or medical tomograph. This can be used to make very fast measurements to study time resolved phenomena such as cracking or deformation in engineering materials. Alternatively, a monochromator can be used to select just one wavelength from the beam in order to study delicate living samples, such as these living plants, which would be damaged by the higher flux. Measurements are nonetheless much faster than in a laboratory, taking just 90 seconds for a complete 3D measurement. This allows the researchers to study large numbers of samples in the course of a week of beamtime.

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References:
1-B. Choat et al. Plant Physiology (2016) 170, 273.
2- G. Charrier et al. Plant Physiology (2016) 172, 1657.

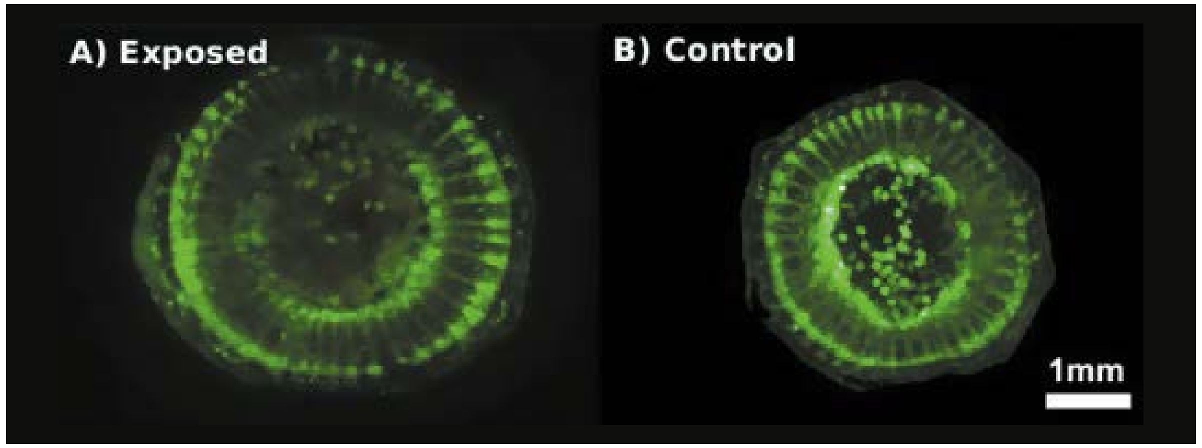

Figure 2. Cross section of the upper stem part of a grapevine plant that was scanned with HRCT 10 days before treatment with fluorescein-diacetate. The fluorescent, green colored cells represent living cells, which were slightly more pronounced in the control image (B) compared to the area that was exposed to x-ray beam (A). White bar = 1mm scale [2].

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As early as SOLEIL's first years of operation

Since 2007, DIFFABS has made it possible to combine analyses in high-energy diffraction and absorption on the same area of the sample and under identical physicochemical conditions, with the possibility of using an X-ray microbeam (10 x 10 µm²). In the case of complex materials, the obtained mappings with moderated resolution reveal the inhomogeneity of the sample—for ancient materials, for example, but also in the medical field. The measurements can also be carried out under stress to study the mechanical behavior of the sample under extreme conditions of temperature—the field of excellence of this beamline—and pressure.

The beamline LUCIA, on the other hand, offers a more focused microbeam (2.5 x 2.5 µm²) for absorption and X-ray fluorescence mappings at energies lower than DIFFABS, hence the complementarity between these two beamlines for the chemical mapping of heterogeneous objects.
The first time-resolved magnetic imaging results at SOLEIL were obtained by electron photoemission spectroscopy and soft X-ray absorption spectroscopy on TEMPO, another beamline that was commissioned as early as 2007. This work was carried out in collaboration with Institut Néel in Grenoble.
With the installation of new microscopes, the beamline TEMPO has shifted its focus towards a better consideration of the time variable of the measurements. Making it possible to determine the reaction kinetics along a surface or at interfaces, and to measure the magnetization dynamics in magnetic materials, for example. Photoemission imaging results were also obtained on the second branch of TEMPO, using the microscope of CEA laboratories IRAMIS and LETI.

[2] Reflets de la Physique (2013), 34-35, p38-42.

In the low-energy range

At SOLEIL, every possible wavelength range is covered by at least one beamline for imaging studies. In the infrared (IR), the beamline SMIS combines absorption spectroscopy and Fourier Transform Infrared spectroscopy (FTIR). For nearly 10 years, this beamline has made it possible to create chemical mappings of various compounds with a few micrometer resolution: polymer films, mineral inclusions, materials of biological, biomedical, or archaeological interest, and more. SMIS was recently equipped with a station called «nanoIR» for the chemical analysis of the samples with spatial resolution up to a few tens of nanometers—a leap of three orders of magnitude compared to traditional IR microspectroscopy. The IR beam illuminates the sample from the top. Two analysis techniques called AFMIR and SNOM can then be provided. See page 12 for more information.
Although NanoIR currently operates with a pulsed IR laser source, coupling with a wide-band laser source (access to the range of C-H, O-H and N-H stretching vibrations) and with the synchrotron source (access to the entire mid-infrared region) will be available shortly.

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SMIS

Infra-red imaging and spectro-tomography

For a number of years the team of the SMIS beamline has developed a worthwhile collaboration with researchers of the IAS (Institut d'Astrophysique Spatiale d'Orsay) in respect of the analysis of small extra-terrestrial dust systems originating from primitive objects (asteroids, comets, see Rayon de SOLEIL, no. 18 p. 4) using micro-IR and micro-Raman spectroscopy. This helps to improve our understanding of the start of the Solar System and the evolution of these materials in different astrophysical environments ranging from primitive solar nebula, through to the accretion process and incorporation in planetesimals up to changes in the surface of asteroids and comets and the addition of volatile materials to the terrestrial planets.

A new FTIR microscope-imager equipped with a matrix detector of focal plane array type of 128x128 pixels has recently been installed on SMIS, which makes it possible to simultaneously obtain a very large number of spectra over surfaces extending from several centimetres down to a few microns. The speed of this type of detector also allows development of a new analysis method: three-dimensional microtomography. This technique was developed in 2013 at the Synchrotron Radiation Center at Madison (USA) by M. C. Martin and his team and is now in course of implementation on the SMIS beamline. The first analyses were performed on grains of the "Paris" carbonaceous meteorite, mounted at FIB points.

The results show the 3D distribution of different phases of these grains, which have a very heterogeneous composition. Complementary analyses have been performed using X-ray tomography on the PSICHE beamline. The coupling of these two techniques makes it possible to determine at a small scale, the relationships between organic materials and minerals that is fundamental in understanding the origin and evolution of organic material in the different development phases of our planetary system. These analyses constitute the initial phase of a measurement protocol, extending from the least destructive to the most destructive analytical techniques, aimed at characterising all of the properties of samples, with an eye to the next space missions that will bring back samples (Hayabusa 2, JAXA, and OSIRIS-REx NASA).

The objective is to develop and exploit a wide range of analyses using FTIR three-dimensional microtomography for use on various materials of extra-terrestrial origin by the researchers of the IAS and for other samples (biological, medical, heritage, physics of materials, etc.) for scientists and other users of the SMIS beamline. This analytical method represents a major technological breakthrough that will permit non-destructive characterisation of the composition of these materials at a scale that has never yet been achieved.

The instrument will likewise be available for large surface area 2D IR spectroscopy. Coupling with the synchrotron source is in progress and it is expected that the instrument will be available for calls for projects in the second half of 2017.

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As for the range of energy of the ultraviolet: one of the three branches of beamline DISCO is a UV fluorescence imaging station for studies in biology (living cells) and materials science. New possibilities of sample excitation and detection (autofluorescence, used to avoid probe-based labeling) have proved particularly promising for applications in biomedical diagnosis [3] or treatment: for example therapies targetting tumors that usually cannot be reached by photochemotherapy [4], or the improvement of radiotherapies using nanoparticles [5].

Electronic and magnetic properties of advanced materials at the nanometer scale

To make the best use of the remarkable properties of nano-objects, scientists must carry out an exhaustive study of their electronic structure through the detection of core levels, and the analysis of the structure of the delocalized valence bands, which are directly responsible for chemical bonds, electrical transport and thermal and mechanical properties. Angle-resolved photoelectron spectroscopy (ARPES) is the appropriate technique in this case, allowing for precise measurements of the dispersion of the strip structure of materials in the reciprocal space. Since 2011, the beamline ANTARES has offered a Nano-ARPES microscope capable of performing, with a spatial resolution of a few tens of nanometers, the imaging of the electronic properties of systems close to the devices used for applications in tomorrow's electronics as well as quantum engineering.

[3] - D. Bazin *et al.*, Comptes Rendus Chimie (2016) 19, 1439.
[4] - S. Kaščáková *et al.*, Nano Research (2015) 8, 2373.
[5] - L. Štefančíková *et al.*, Cancer Nanotechnology (2014), 5, art.n° 1.

A little higher up in the energy range is HERMES, another spectro-microscopy beamline that is the combination of two types of equipment: one for photoemitted electron microscopy (PEEM), a technique which, unlike ARPES, preserves the spatial provenance of the photoelectrons, and one for scanning transmission X-ray microscopy (STXM). One of the many applications made possible by spatial resolutions of a few tens of nanometers consists in physically imaging the magnetic domains. It then becomes possible to manipulate magnetic bits within a magnetic structure.

SOLEIL also offers another technique of magnetic imaging available on beamline SEXTANTS: Fourier transform holography, with a spatial resolution of a few nanometers—limited by the wavelength of the X-rays. The team of the beamline showed that linearly polarized X-rays can be used to image the magnetic domains in thin layers, usually studied using circular polarization. This technique makes full use of the characteristics of X synchrotron radiation, i.e. high intensity and short pulses (for time resolution), energy and polarization tunability (for chemical selectivity and magnetic sensitivity) and high degree of coherence (for a large field of view).

The CRISTAL beamline is dedicated to the X-ray diffraction study of powders and non-biological (single)crystals. The beamline gives access to the atomic structure of crystals by measuring the intensity of the diffraction peaks (Bragg reflections). Users can also take advantage of the coherence of the X-ray beam: the modification of the shape of the Bragg peaks is related to the shape of the diffracting crystal and to its internal deformations due to the constraints imposed by its environment (coherent diffraction, Bragg ptychography). And just like TEMPO, CRISTAL provides users with the additional asset of the time dimension thanks to time-resolved diffraction, as shown by the first pump (IR)-probe (RX) experiments that started in September 2016 using the slicing mode for a temporal resolution of the order of 200 fs (10^{-15} s).

And the volume, too

Four beamlines provide (or will soon provide) tomography and/or imaging systems with coherent diffraction scanning (ptychography), in addition to two-dimensional analyzes.

In tomography, similar to a medical scanner except with much better space resolution, the sample, placed on a rotation stage, is illuminated by X-rays and a projected image (X-ray) is formed on a detector positioned downstream. During the rotation of the object across 180°, it is radiographed from all angles. The 3D structure of the object is reconstructed from all the projected images through calculations. This non-destructive technique allows for visualization of the inside of the sample thanks to X-rays and without having to cut through, while making it possible to study sample evolution over time, for example under stress.

The high brilliance and partial spatial coherence of synchrotron radiation make it possible to use the technique known as phase contrast imaging, which is more sensitive than the absorption technique traditionally used in radiography. After traveling through the sample, the electromagnetic waves dephase, thus creating an interference phenomenon observable by the detector when placed at a distance optimizing this contrast. Small variations in the density of the sample can thus be revealed, and this makes it possible to thoroughly study micro or even nanometric objects, depending on the beamline.

Finally, in ptychography, 3D reconstruction is the result of the «compilation» of hundreds of thousands of diffraction images of the sample: for each orientation of the sample, several hundred diagrams are recorded, allowing to reconstruct the 2D image of each projection (orientations of the sample). All the 2D images collected are then used in the final 3D reconstruction—a procedure that requires the implementation of especially powerful algorithms.

Beamline PSICHE offers these 3 techniques when it operates in non-focused, monochromatic X-ray mode. This beamline is optimized for experiments in materials science: composite or structured materials, batteries, studies on corrosion, fatigue or deformation, and more.

In 2017, the beamline PUMA, for studies of ancient materials, will include a hard X-ray spectromicroscopy station (2D imaging and point microanalyses) and an X-ray hard microtomography station—both aimed at resolutions of the order of one micrometer. PUMA is one of the three long beamlines available at SOLEIL. Its length was defined to ensure sufficient X-ray beam coherence (long distance between the X-ray source point and X-ray/sample interaction point), as well as to create a large field of view for full-field measurements on large samples.

This distance is even more important for beamlines ANATOMIX and NANOSCOPIUM (160 m) to achieve appropriate coherence and size (30-100 nm) of the X-ray beam expected at the sample. Dedicated to full-field radiography and tomography in absorption and phase contrast, beamline ANATOMIX will cover a large range of space resolutions: between 30 nm and 10 µm. NANOSCOPIUM allows to perform 2D/3D qualitative, chemical and structural analyses (ptychography) in various fields such as microelectronics and materials, biomedical, geo-biological and environmental sciences, with the aim of achieving the highest resolution: 30 nm.

Processing data and constructing images

The different techniques listed, and especially those allowing for the 3D reconstruction of the samples, require the processing and analysis of enormous amounts of data. On beamline NANOSCOPIUM, for example, scanning hard X-ray microscopy allows for the acquisition of multimodal data, meaning that it acquires images in which each pixel contains several types of data. For example, the same sample can be mapped using absorption, phase, dark field and X-ray fluorescence contrasts through a single scan, as made possible by the FLYSCAN infrastructure for rapid acquisition.

A free and open-source software for the processing and reconstruction of multimodal X-ray imaging and tomography data was developed at SOLEIL to support scientists in their work. Known as «Multi-Modal X-ray Imaging» or MMX-I, this software allows users to process their raw data either directly on beamline NANOSCOPIUM, or on their personal computer. The most experienced programmers can also integrate MMX-I as a computing library into an existing software, or develop new functionalities that will benefit the rest of the community.

Another project called «Défi imag'in» is underway, involving scientists from DESY, KIT and SOLEIL. With support from the CNRS mission for interdisciplinarity (MI), its objective is to develop the tools (computing platform, graphical interface) necessary for the processing of rapid imaging measurements by X-ray diffraction over the course of the experiment, or even in real time. This is quite the challenge since the increase in brilliance of the X-ray sources and the detectors' improved efficiency and dynamics have led to an increase in collection speeds by several orders of magnitude over the last two decades.

So users can rest assured: there is no doubt that the terabytes of data collected over shorter and shorter times across SOLEIL's different beamlines will be processed and converted into superb 2 or 3D high-resolution images.