N°21

LE RAYON DE SOLEIL

THE SYNCHROTRON MAGAZINE

Chemistry in the light

SOLEIL SYNCHROTRON

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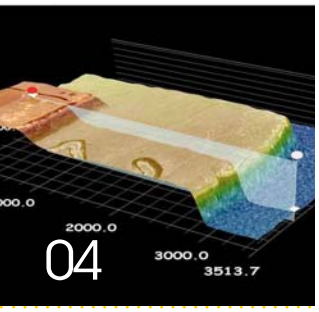

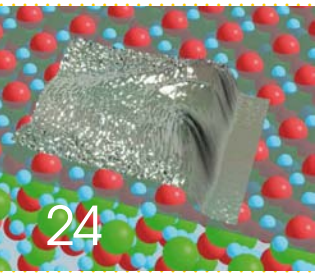

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Editorial

Jean Daillant
Director General

I am delighted to be signing my first editorial at a time when the scientific activity at SOLEIL is bustling and several major projects are underway.

The construction of the NANOSCOPIUM beamline, which began in mid-October, will considerably increase our experimental capabilities in terms of x-ray imaging, reaching a resolution of 30 nm. The time has also come for the IPANEMA building, dedicated to ancient materials, to be built. Even more than the originality of this cross-disciplinary platform based on the synchrotron, the very close collaboration of its partners will ensure its success. This constant concern at SOLEIL to conduct research in open partnerships has also led to the development of new agreements, with the future Swedish synchrotron MAX4, and the tightening of some connections that were already very close, for example with our German colleagues at DESY and our Swiss colleagues at SLS.

We have also submitted EQUIPEX project proposals: the ROCK project, accepted in 2010, will allow us to respond to the increasing demands of communities in the fields of energy and chemistry. A proposal for an x-ray nanotomography beamline project, which is also very open to fundamental and applied research in materials science and biology, was submitted in 2011.

Our commitment to user support never wanes, and nor do our rigour or our efforts in these difficult budgetary times that have affected everyone. We too are trying to overcome these difficulties, to continue to carry out the best possible research.

NEW TECHNOLOGIES

ROCK for energy

REDUCE GREENHOUSE GASES, PRODUCE ENERGY FROM RENEWABLE RESOURCES by seeking to optimize battery performance and biofuel production processes; these are some of the targets of ROCK, a future SOLEIL beamline. The first European beamline dedicated to research on batteries and catalysts, ROCK is one of 52 Equipex projects announced by the French Prime Minister, François Fillon. This project, funded to the extent of 3.3 million euros, brings together the skills of SOLEIL, the French electrochemical energy storage network and the UCSS (Lille 1, CNRS) and LRS (CNRS, UPMC) laboratories.

CELEBRATION

Foundation stone of IPANEMA

ON SEPTEMBER 16TH AT SOLEIL WAS LAID THE FOUNDATION STONE OF IPANEMA, the European platform of research dedicated to the study of ancient materials, in the presence of several politicians and scientists (see photo opposite). A hundred or so people gathered for the ceremony.
IPANEMA supports all synchrotron projects submitted to SOLEIL beamlines concerned with research on ancient materials.
In addition, a beamline dedicated to this subject, called PUMA, will be added to the 26 SOLEIL beamlines already in operation.
A preparation room for ancient materials, a laboratory dedicated to archeology, paleontology, ancient environments and conservation science, allows users to prepare their samples on site. Eventually, its equipment will be available as part of the IPANEMA platform.
The construction of these buildings will soon begin.

From left to right: Bertrand Lavedrine (French Natural History Museum), Louis B.J. Vertegaal (NWO, Netherlands Organisation for Scientific Research), Jacques Stern (French Minister for Higher Education and Research), Jean Daillant (Director General ,SOLEIL), Isabelle This-Saint-Jean (Île de France Region), Bertrand-Pierre Galey (French Minister of Culture and Communication) and Bertrand Girard (CNRS).

5

5 years ago, INRA and SOLEIL started a fruitful collaboration, based on making three INRA engineers on secondement at SOLEIL. It already gave almost 50 results.

449

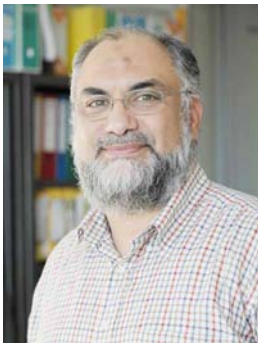
Number of projects submitted during the 9th SOLEIL call for projects, with a closing date of 15th September, 2011. This figure, up from 382 in the previous call, has increased with each new call for projects.

17,000

Total number of people (users, visitors, suppliers, etc) visiting the SOLEIL site each year.

IN BRIEF

APPOINTMENT OF AMOR NADJI



After a PhD in nuclear physics, Amor Nadji turned to accelerator physics in 1990, joining the groups at LURE in Orsay. Involved with the "SOLEIL adventure" from the pilot study stage, he served as Head of the Physics and Accelerators Group from 2002.
In July, Amor Nadji succeeded Jean-Marc Filhol who was appointed to other duties, as Director of the Sources and Accelerators Division.

NANOSCOPIUM

Construction of the long NANOSCOPIUM beamline began on 19th October.
The work is scheduled to last 12 months and follows very specific preparation of the foundations to meet the stability requirements of this unique X-ray imaging beamline (see page 9).

IMAGING AND NANO-ARPES

An innovative and powerful tool for the nanosciences

In recent decades we have witnessed exponential advances in the different areas of the new nanotechnologies. These advances, seen particularly in nanoelectronics, nanomagnetism and nanochemistry, among others, affect almost every aspect of our lives and are major players in our evolution towards the "information and automation age".

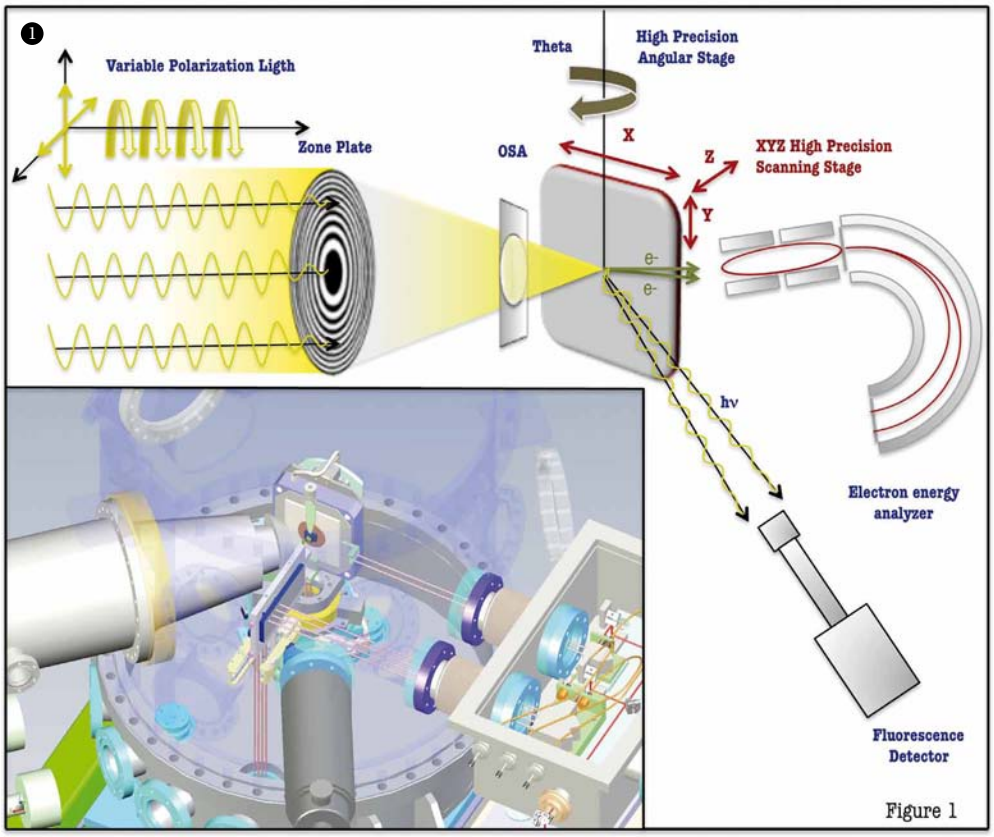


Figure 1

Following the fundamental step in the creation of nano-objects and even if these "building blocks" have shown remarkable properties, they would have remained unexploited if, at the same time, we had not developed new tools capable of viewing and scrutinizing objects on a wide range of scales, from a few microns to a few tens of nanometers.

Recently, great progress has been made as a result of the rapid expansion of modern microscopies. However, even if they have achieved nanometer spatial resolution, the challenge still remains to provide powerful high-energy-resolution spectroscopic tools for probing nano and micro-areas.

Electronic properties at nanometer resolution

The challenge is to quantify and analyze the electronic properties of advanced materials on a nano and micrometer scale. For such a result, analysis of the electronic structure must be comprehensive, not only with regard to detection of core levels, but also to study the structure of delocalized valence bands, directly responsible for chemical bonds, electrical transport and the thermal and mechanical properties.

Traditionally, angle-resolved photoemission spectroscopy (ARPES) is the only technique capable of making sufficiently precise measurements of the dispersion of the band structure of materials in reciprocal space. The state of the art ARPES equipments installed at synchrotron radiation sources is such that it can offer energy and angular resolution of better than 5 meV and 0.1°, respectively. Yet, until now, no instrument has been capable of performing spatially resolved ARPES experiments on the nanometer scale.

This paper presents the first results of the Nano-ARPES microscope recently installed on the ANTARES beamline at SOLEIL. This sophisticated instrument is able, with a spatial resolution of several tens of nanometers, of carrying out the direct imaging of core levels and their chemical shifts, band electronic structures in reciprocal space and constant

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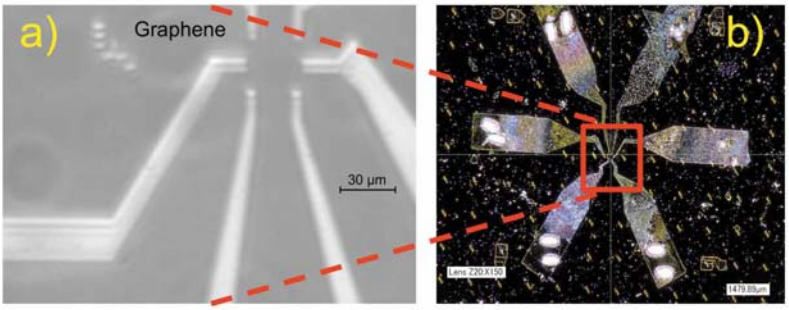

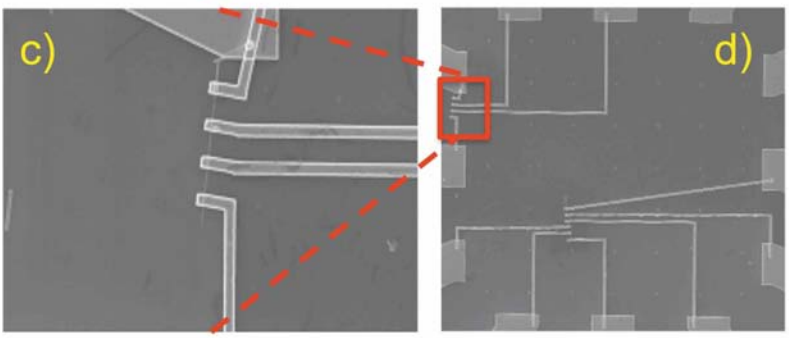

Figure 1: Diagram of the Nano-ARPES instrument. The ZP's central stop and the order sorting aperture (OSA) intersect the zero and higher diffraction orders and lets through only the first order. The microscope is equipped with a fluorescence detector to carry out in situ X-rays adsorption experiments.

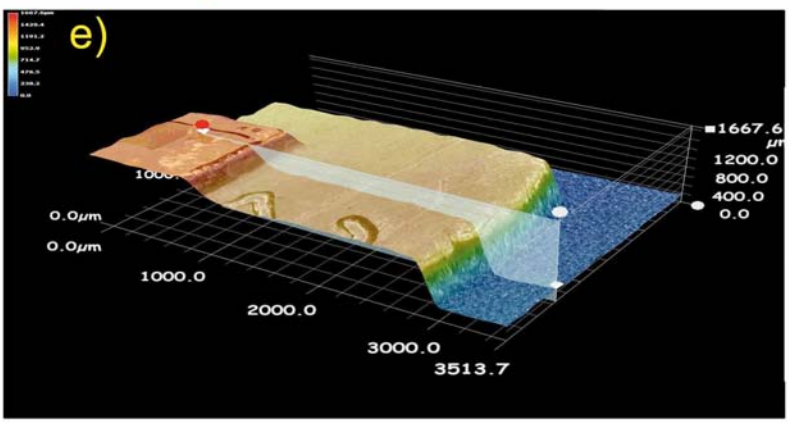

Figure 2: Exfoliated graphene film visualized by optical microscopy (a) and SEM (b). Parts (c) and (d) show SEM images of isolated silicon nanowires. Finally, visualization of a HOPG crystal by optical microscopy is shown in (e).

energy surfaces in reciprocal space, especially the Fermi surfaces.

First results on ANTARES

Figure 1 shows a diagram of the ANTARES microscope. It differs from conventional ARPES instruments mainly in that the sample can be mounted on a high-precision plate that ensures nanoscale scanning of samples in the x, y and z directions. The polar angle (θ) and the azimuth angle (φ) can also be automatically scanned over a 90° range. Finally, the soft X-ray beam (from 20 to 900 eV) with a controlled linear or circular polarization can be focused to about 80 nm (or better), using Fresnel Zone Plate lenses ("flat area", ZP). The ANTARES microscope has two operating modes, spectroscopy with nano-spot and spectroscopic imaging.

Figure 2 shows some examples that, far from being an exhaustive list, were selected to illustrate the types of the most representative specimens currently studied by the ANTARES Nano-ARPES microscope. Several samples of exfoliated graphene less than 30 µ m wide and one atom thick (the thinnest material ever isolated) were oriented and largely characterized using this Nano-ARPES microscope with excellent reproducibility of results (Ref. 1). The ANTARES microscope has also measured the valence states and core levels of isolated nano-objects such as boron-doped silicon nanowires (Figure 2, ref 2). Finally, Figure 2 shows the visualization of a crystal of highly oriented pyrolytic graphite (HOPG), which, despite its apparent homogeneity, is composed of micrometric grains.

To demonstrate the capabilities of the new microscope, we present the HOPG imaging and spectroscopy study. The sample is a polycrystal composed of grains of micrometer-sized single-crystals of graphite randomly oriented in the basal plane of the crystal. The basic unit of the HOPG is graphite, which has a planar structure, where in each layer, carbon atoms are arranged in a hexagonal lattice. The sp^2 electrons in the carbon atoms in each plane are bonded by strong covalent σ bonds and covalent π bonds for their other p electrons. These π links are delocalized conjugated bonds which are perpendicular to the atomic planes, and are responsible of the high electrical and thermal conductivity of graphite.

Figure 3 shows a 7 µm x 7 µm map of the photoemission intensity of a reduced energy window around the Fermi level, which is the energy that separates the occupied from the unoccupied bands. The microscope's detection geometry was fixed so that it can only detect the intensity from the grains oriented in the ΓK direction, where only the π bands approach the Fermi level. The thermal and mechanical stability of the microscope makes it possible to obtain high contrast and highly reproducible images. The visualization of each grain, each measuring 1-2 microns, is direct and fast. The Nano-spot mode of the microscope allows performing a full spectroscopic and electronic band

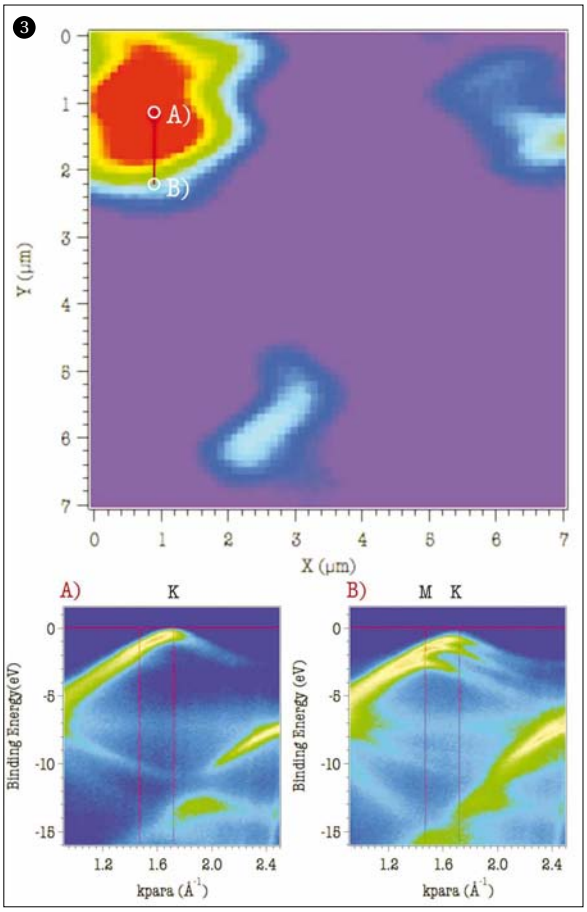

Figure 3: Spatially and angularly resolved photoelectron intensity maps of an HOPG crystal.

dispersion determination not only inside the grain but also in its surroundings (Figs. 3A and 3B).

Another way of studying the electronic band dispersion is to analyze photoelectron intensity maps at constant energy. However, for this, very precise angular scanning is necessary that requires almost perfect coincidence of the light source (or ZP), the Scienta analyzer focus and the mechanical axis of rotation of the microscope in the region of the sample to be measured, which in this case is the surface area of a grain only 2 µm wide.

Figure 4a shows the dominant features of the Fermi surface mapping of a single grain of the HOPG crystal. It reveals small pockets of electronic states concentrated in the six corners of the Brillouin zone of graphite. Figure 4b shows the same Fermi surface map detected without ZP, in traditional ARPES mode. The Fermi surface is now characterized by a ring with a ΓK radius, which includes the six points of all individual grains randomly oriented in the HOPG crystal.

Thus, the new Nano-ARPES microscope recently installed on ANTARES, is already capable of providing spectroscopic images with a spatial resolution of several tens of nanometers, while preserving angular and energy resolutions comparable to the best performing ARPES instruments installed on synchrotron radiation sources.

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1. Contact the ANTARES group for more information.
2. Contact Bruno Grandidier at IEMN, Lille for more information.

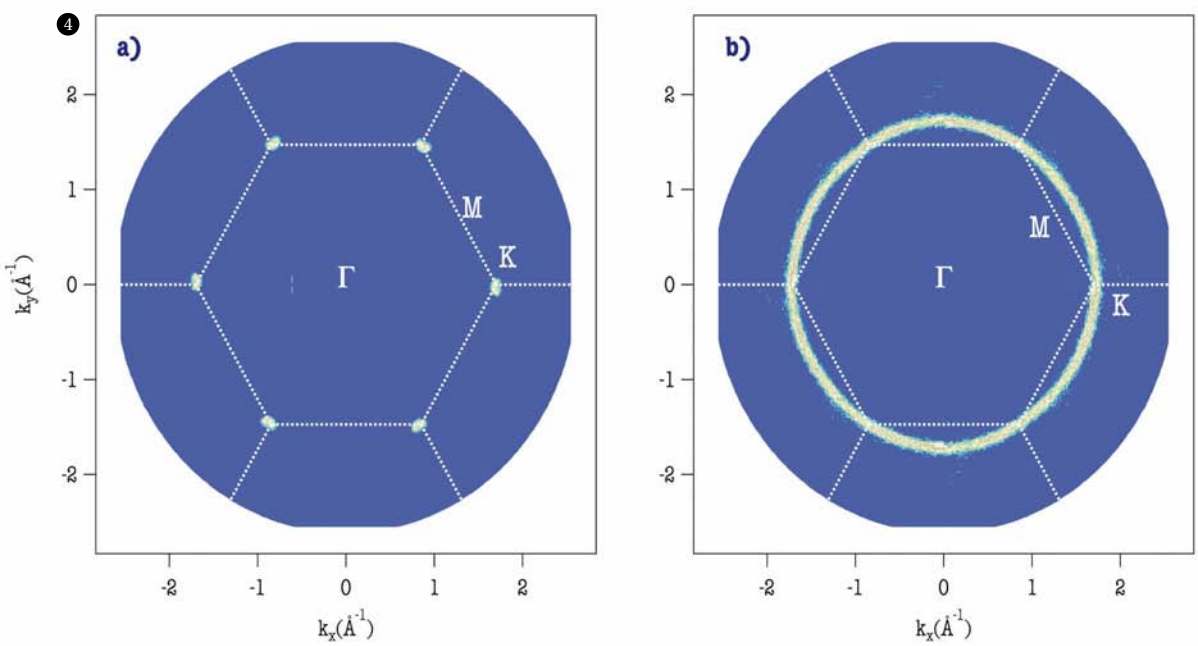
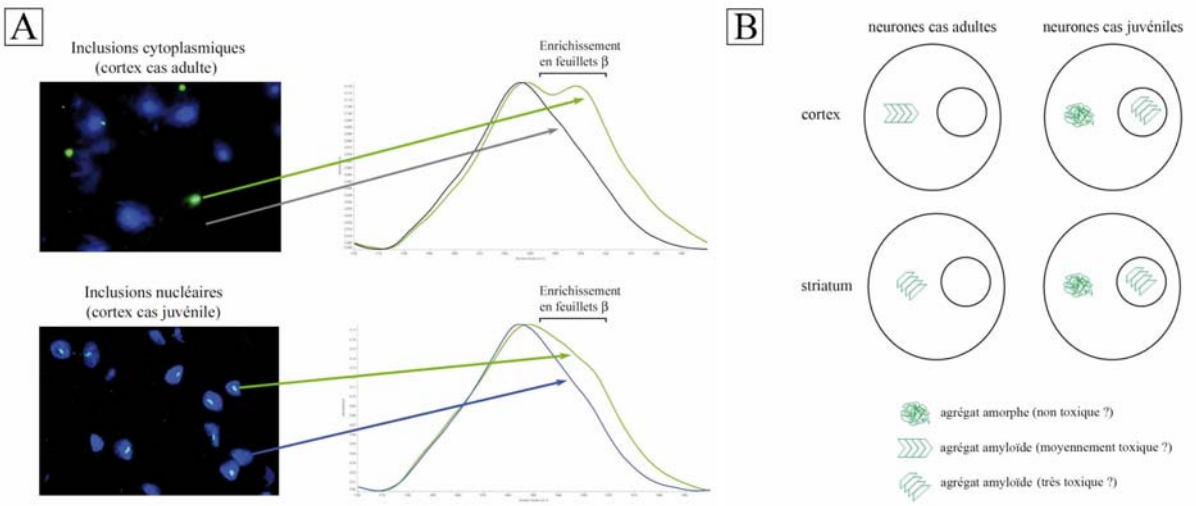


Figure 4: Fermi surface of a single grain of graphite in a HOPG crystal by Nano-ARPES (a) and Fermi surface of a HOPG crystal by conventional ARPES (b).

NEURONAL INCLUSIONS IN HUNTINGTON'S DISEASE

Study on SMIS beamline by infrared micro-spectroscopy

Huntington's disease (HD) is characterized by the formation of protein aggregates (inclusions) in certain regions of the brain. Recent data suggest that the secondary structure of aggregated proteins could play a major role in neuronal degeneration. Infrared micro-spectroscopy using synchrotron radiation is an ideal tool to study the secondary structure of aggregated proteins and to determine whether this structure varies according to their subcellular or tissue localization, or whether the patient has the adult or juvenile form of the disease.



Analysis of the secondary structure of proteins in the inclusions. (A) Obtained by fluorescence microscopy, these images reveal the presence of inclusions in the cytoplasm or the nuclei of cells on sections of the cortex of patients suffering from adult or juvenile HD. Nuclei are stained blue and inclusions in green. The infrared spectra of the inclusions (green) and control compartments (cytoplasm and nucleus, respectively, in black and blue) are distinguished by the presence of a shoulder corresponding to an enrichment in β sheets. B) A model of protein aggregation in HD suggested from the results obtained on the SMIS beamline.

HD is a genetic neurodegenerative disorder that affects about 6,000 people in France. The symptoms include movement disorders, behavioral disorders and dementia. The mutation responsible involves the expansion of a repeated sequence of the CAG codon in the huntingtin gene. The translation of this sequence leads to the synthesis of an excessively long polyglutamine chain (polyQ) in the protein. In healthy individuals, this sequence contains from 20 to 35 repeats. A long sequence of 36 to 60 repeats leads to the adult form of the disease and a sequence of over 60 repeats gives the rare juvenile form. The disease is mainly characterized by progressive very pronounced degeneration of the striatum, but also the cortex. The disease is more severe and progresses more rapidly in juvenile cases. Inclusions are formed in neurons of the striatum and cortex and are predominantly cytoplasmic in adult cases and nuclear in juvenile cases.

Importance of the secondary structure of protein aggregates

The aggregation mechanisms involved in HD have been studied since the early 90s and the ability of synthetic polyQ peptides to form aggregates rich in β sheets (called amyloid) was quickly demonstrated. Huntingtin can form a variety of aggregates in vitro: oligomeric (small soluble aggregates), annular, amorphous (without apparent structural organization) and fibrillar. Recent studies have shown that the experimental conditions influence the secondary structure adopted by proteins in polyQ aggregates, and that this structure could play a role in cell toxicity. For example, a simple change in temperature can change both the structure of a polyQ peptide and its cytotoxicity.

The secondary structure of protein aggregates in the brain of patients with HD is unknown and there is no evidence that the aggregates formed in vitro mimic the aggregate structure in patients. If the environment plays an important role in the conformation adopted by the aggregates, the biochemical complexity of the human brain could modulate the aggregation differently from that reported in vitro. The examination of brain samples from patients provides an opportunity to determine the structure of inclusions in situ. For this type of study, analysis by infrared (IR) microspectroscopy using synchrotron radiation is particularly suitable.

Guylaine Hoffner, William André and Philippe Djian, CNRS group "Régulation de la Transcription et Maladies Génétiques", Université Paris Descartes.

Analysis of the secondary structure of protein inclusions by synchrotron infrared micro-spectroscopy

Because of the small size of the inclusions (a few microns), analysis of the inclusion structure requires a very sensitive technique, such as a synchrotron IR source, since the narrowness and intensity of the beam make analysis at the cellular level possible. The IR spectrum of a sample is used to define the chemical composition of the sample and the conformation of its diatomic bonds, thus providing access to information on the secondary structure of proteins. This technique is not destructive and it is possible to couple the IR analysis with fluorescence microscopy to identify the inclusions labeled with antibodies coupled to a fluorescent marker.

Guylaine Hoffner and William André, who work in Philippe Djian's group, have studied the brains of patients with HD in collaboration with the SMIS beamline. Brain sections were placed on slides and inclusions localized by fluorescent labeling of huntingtin with a specific antibody. The sections were then analyzed with a ThermoNicolet Continuμm XL IR microscope in transmission mode. For analysis in the mid-IR (4000-800 cm^{-1}), the synchrotron beam was focused onto the sample with a spatial resolution of 6 microns. Under these conditions, synchrotron IR radiation is 100 times brighter than that emitted by a conventional source.

The researchers acquired IR spectra of different types of inclusion and their control compartments (cytoplasm or nucleus). They focused on the amide I absorption band (1600-1720 cm^{-1}), a spectral region very sensitive to the secondary structure of proteins. The study of differences between the spectra of inclusions and controls revealed the structural characteristics of the inclusions. Amyloid structures have a particular signature in the IR (Fig. 1A).

Demonstration of the structural polymorphism of the inclusions and its links to neuronal degeneration

Cytoplasmic inclusions in the cortex and striatum of adult patients were both highly enriched in β sheets (Fig. 1A), but their IR spectra and therefore their amyloid conformations differed. The fact that the degeneration was more pronounced in the striatum than in the cortex of adult cases suggested that the amyloid structure of inclusions in the striatum was more toxic to neurons than that of the cortex. As for the juvenile cases, their nuclear inclusions had an amyloid conformation similar to that of cytoplasmic inclusions in the striatum of adult cases whereas their cytoplasmic inclusions were amorphous aggregates lacking an amyloid structure. Nuclear inclusions would seem to be toxic to juvenile patients, while cytoplasmic inclusions appear harmless.

This study confirms the existence of amyloid aggregates and describes a complex picture of aggregation at its final stage in HD (Fig. 1B). It also suggests a link between the nature of aggregate amyloid structures and their neuronal toxicity. It remains to verify experimentally the toxicity of amyloid conformations described in this study and try to understand the mechanisms underlying this toxicity. The flexibility of protein aggregates may, for example, depend on their amyloid conformation, allowing the exposure or not of the polyQ on their surfaces. The latter could then interact with cell components and lead to cell death.

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FOCUS ON

The future long beamlines of SOLEIL

The NANOSCOPIUM and NANOTOMOGRAPHY "long" beamlines will be dedicated to state of the art hard X-ray nano-imaging techniques. A 2,200 m² extension to the SOLEIL experiment hall will be built during 2012, to accommodate these beamlines.

Modern synchrotron-based hard X-ray imaging opens fundamentally new ways of spatially resolved sample characterization by fully exploiting the coherence of the X-ray beam. It can reach resolution down to 10-50 nm, filling the resolution gap between optical and electron microscopy, and provides quantitative information with high sensitivity on density variation, elemental distribution, and/or chemical speciation. Moreover, the high penetration power of hard X rays enables non-destructive studies of buried structures on intact specimens with large thicknesses. While NANOSCOPIUM will yield scanning images, NANOTOMOGRAPHY is dedicated to full-field techniques.

Why use coherent X-rays?

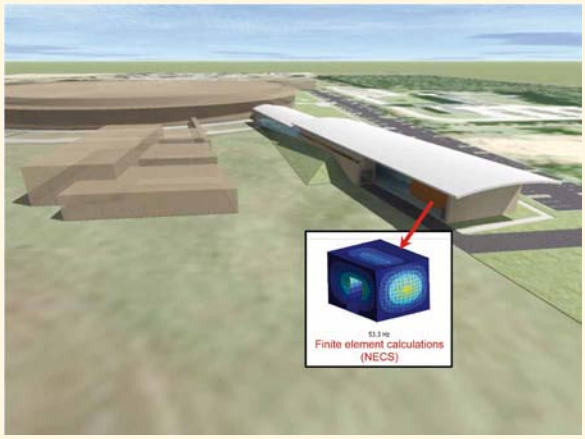
Coherent X-rays not only can be focused into a beam only tens of nm wide; they can also be used to simultaneously generate multiple, complementary images of a given object, in which contrast is generated by different mechanisms. In these "multimodal" imaging techniques, one of the images represents absorption in the sample, analogous to a conventional radiograph. Another maps the change in phase of the radiation, like perturbations in a wavefront. Many interesting samples like biological cells and soft tissue are transparent to X rays, yet interact strongly with the phase, giving very high contrast projection images even in the hydrated state. A third contrast mode, sensitive to scattering, reveals the presence of sub-resolution-size nanostructures and their ordering. In scanning mode, these principles even allow us to simultaneously produce structural images of a sample at "super-resolution" significantly finer than the beam size, using a technique known as "ptychography".

Why so long?

Over 160 meters, NANOSCOPIUM will produce a more coherent and narrow beam in the end-stations than is possible for a short beamline. Focusing optics designed to capitalize upon this long distance will create intense and stable nano-beams. A "secondary source" aperture halfway along the beamline protects against vibrations and drifts, and provides control over the coherence and flux. On NANOTOMOGRAPHY, the beamline length of 200 m not only gives a beam of several cm width for the study of large samples, but also ensures very high transverse coherence of the X rays and, thus, sensitivity to extremely subtle density differences in the objects studied.

Experimental techniques

NANOSCOPIUM, dedicated to scanning techniques in the 5 – 20 keV range, will provide unique research opportunities by combining the analysis of sample chemistry, via X-ray fluorescence and absorption spectroscopy, with structural analysis from coherent imaging at high spatial resolution (= 30 nm) in 2 and 3 dimensions. Elemental distributions and oxidation states can be quantified at the trace level in geological and biological samples for most of the elements starting from Titanium. Fluorescence spectroscopy will target elements as light as phosphorous. Beamline NANOTOMOGRAPHY will operate between 5 and 25 keV and provide 3D volume data over a wide range of length scales, from a resolution of 30 nm (pixel size) to an object width of 40 mm, with a detection limit for density variations down to 0.5 mg/cm³ (0.2 electrons per nm³).



The building extension for the NANOSCOPIUM and NANOTOMOGRAPHY beamlines. The radioprotection hutches must ensure high thermal and vibration stability. Responsible for building construction: P. Eymard.

What is the outlook?

The features, specifications, and new techniques for NANOSCOPIUM have motivated all groups involved to work to make the line available to users by late 2013. For NANOTOMOGRAPHY, a funding application through the EQUIPEX program has been submitted.

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EXPERT PORTRAIT

ALAIN LESTRADE, HEAD OF THE ALIGNMENT/ METROLOGY GROUP

Alain Lestrade doing offset measurements of the DISCO beamline cold finger fiducials

When did you join the world of synchrotrons?

In the early 90s, a maintenance contract with the Grenoble-based company in which I was then a surveyor, led me to work at the ESRF. I finally stayed 12 years, specifically in charge of alignment of the storage ring and basic calibration. When the SOLEIL project restarted, I joined the team working from the detailed pre-project (APD); this was in January 2002.
I had meanwhile acquired knowledge, away from the synchrotron environment, in optics and electronics through resuming my studies in the field of microwave & optics.

What does your work consist of?

The Alignment/Metrology group became involved very early on in the project on the "instrument" side and it is always beneficial to be present upstream of a project. This allowed us to agree with the Sources Division on the design, construction and installation of beams supporting the various magnets that guide the electron beam. It is easy to understand that, to guarantee precision and reliability in the beam path, the positioning of each element of the accelerators and storage ring is critical. This necessary accuracy was pushed to the extreme in the case of SOLEIL:
The objective was to achieve 20 microns between magnets on the girders, and 50 microns between adjacent girders.
The specifications followed and methods used at SOLEIL were those chosen during the detailed pre-project stage.
But we are also present alongside those responsible for beamlines, starting with their design, in parallel with our work with Sources. I remember, for example, meetings with the CASSIOPEE or DESIRS groups while we were in ALGECO as part of the d'Orsay Faculty: even though the first stone had not even been laid at SOLEIL!
This double interaction with Sources and Experiments already existed at ESRF. But at SOLEIL this has been reinforced by the beamlines. In addition, we are in permanent contact with the Optics group, headed by François Polack (see Rayon de SOLEIL n°19, p10): these exchanges occur on a daily basis. I wanted the alignment work at SOLEIL to extend beyond the traditional framework covering the very principles of purely geometric techniques, usually adopted in synchrotrons. It was not possible to stay within the usual definition, i.e. "the measurement of large dimensions." Geometry remains an essential tool for us, but only one tool among many. This is, I think, the originality of the approach chosen by our group.

How do you define this process?

Always to analyze as much as possible the components on which we work, by including the range of measurement methods available and using procedures from mechanics and optics, which are two fundamental aspects of a synchrotron.
My engineering background has undoubtedly influenced this approach.
Specifically, we have redesigned and developed several instruments, including ways to improve reliability and accuracy. This is for example the case of the wire ecartometer and HLS (Hydrostatic Leveling System), a kind of electronic "water level", which will be installed along the entire length of NANOSCOPIUM. This is the beamline which, at present, has needed the most R&D, because of the conditions of extreme stability that it requires over a long-distance: precision of up to a few hundredths of a micron (vertical movement) for the last 70 meters before reaching the sample.
We started with a device already on the market, which we are now optimizing.
Other challenges ahead: the accuracy of sample positioning under the photon beam, with for example, goniometers or, now, a translation stage required to operate the monochromator on the GALAXIES beamline. It should detect movement of about 5 nanometers! We need to rethink the entire assembly and measurement methods of our interferometer, in order to increase its resolution.
Our work with the Experiments Division is far from over, as all the beamlines have not yet been built.

No time to get bored, it seems?

Indeed, no! Especially as I am preparing lectures, which I have been asked to give for years in-house as part of vocational training. Over the last few months I have delved into all the theoretical aspects of alignment and metrology to develop them further and to be up to answering my future "students". This is another facet of my work that I have not really experienced before but which also motivates me a lot. Another new challenge!

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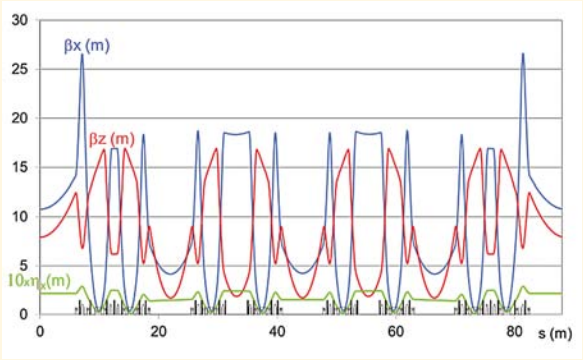
SPOTLIGHT ON

New optics for the SOLEIL Storage Ring



Optical functions of the nominal optics for a quarter of the ring from the middle of a long straight section to the middle of the next long straight section.
The magnetic structure is shown in black.
The β_z-function is minimized in the middle of the three medium and the two short straight sections.
The horizontal emittance ε_x is 3.7 nm.rad.

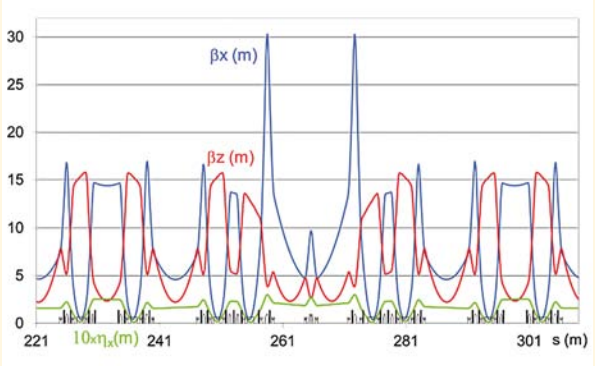
The nominal optics of the SOLEIL storage ring has been optimized to satisfy users in terms of electron beam size at «source» points, while guaranteeing an excellent performance with regard to the operation of the machine (lifetime and injection efficiency). There are several reasons that might require changing the optics: overcome performance limitations imposed by the undulators and wigglers, meet user needs in terms of beam characteristics, or take into account modifications of the machine for the construction of new beamlines.

What is an optics?

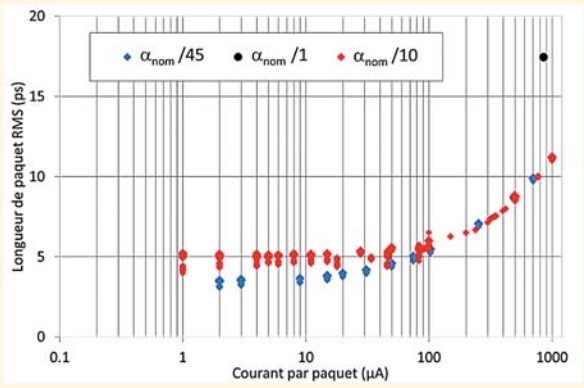
In the storage ring, the focusing system of the electron beam is provided by 160 quadrupoles. Each quadrupole focuses in the horizontal plane and defocuses in the vertical plane, or vice versa. Assembled by two or three, the quadrupoles consist of a focusing system in both planes and the electrons undergo continuous refocusing which gives them an oscillating movement. Verification of the optics is done by measuring the number of oscillations per turn in the horizontal and vertical planes, i.e. the horizontal (ν_x) and vertical (ν_z) betatron tunes. Setting the gradient of the 160 quadrupoles makes it possible to focus the beam transversely at strategic locations such as the middle of straight sections (undulators[1]) and dipoles. Three parameters are commonly used in accelerator physics to represent this focusing effect, which are the optical functions: β_x for horizontal focusing, β_z for vertical focusing and η_x for the horizontal dispersion function (path of electrons with energy deviation). The smaller these are, the stronger the electron beam is focused. The magnetic structure of the ring (arrangement of dipoles, quadrupoles and sextupoles) has a 4-fold symmetry that is repeated on the optical functions. Figure 1 shows the optical functions on a quarter of the machine for the nominal optics.
The optical functions associated with horizontal emittance (ε_x), coupling and energy spread, make it possible to calculate the value of the transverse size and divergence of the electron beam at any location in the ring. Using these optical functions one can also calculate the effect of any magnetic field inserted at a given location in the machine. The linear and nonlinear effects of an undulator, for example, are even more important when the optical functions at the undulator location are large.

Reduction of the effect of the HU640 undulator on beam lifetime

The HU640 undulator is installed in the long straight section SDL05 and serves the DESIRS beamline. When used with vertical linear polarization (horizontal magnetic field), its nonlinear effects increase with the magnetic field and cause a significant reduction of beam lifetime and injection efficiency. The origin of these effects has been identified from beam based measurements that revealed that the field integrals of the undulator, as seen by the electrons, are abnormally large. A magnetic correction system is currently under study and in parallel, optical functions have been modified in the SDL05 straight section and in the other three long straight sections to maintain the 4-fold symmetry of the optics. The β_x-function, amplification factor of the nonlinear effects at large horizontal amplitudes, has been reduced by a factor of 2, the optical functions remaining unchanged elsewhere in the ring. This optics, which has the same horizontal emittance and the same betatron tunes as the nominal optics, significantly reduces the effect of the HU640 undulator, and maintains a lifetime larger



Optical functions of the "NANOSCOPIUM" optics in the long straight section SDL13 and in the adjacent medium and short straight sections. The horizontal emittance ε_x is 3.9 nm.rad.



Variation of the RMS bunch length versus current per bunch for α_{nom} /10 and α_{nom} /45. The RF voltage is 4MV. Bunch length measurements have been performed using a streak camera.

than 10 h and an injection efficiency above 60 % even in the presence of other undulators. In addition, the new optical function values in the HU640 undulator improve the quality of the photon beam. ***This new optics has been in operation on the machine from November 2010 to July 2011.***

Installation of two canted[2] in-vacuum undulators in the long straight section SDL13

The long straight section SDL13 will host two in-vacuum undulators that will serve the two long tilted beamlines, NANOSCOPIUM[3] and NANOTOMOGRAPHY, leading to a significant change in the machine with the installation of new equipment: four dipole magnets to deflect the trajectory of the electron beam only into the two undulators to separate the two photon beams of the two beamlines, two new position monitors, three quadrupoles and two sextupoles in the middle of the straight section. Indeed, as for all the in-vacuum undulators, the electron beam must be focused vertically, which is not the case in the long straight sections with the nominal optics. The three additional quadrupoles ensure the vertical focusing in both undulators and a new optics has been optimized: the so-called "NANOSCOPIUM" optics. The optical functions in the other straight sections are identical to the nominal ones except in the short straight sections where the β_x-function has been reduced to minimize the nonlinear effects of the in-vacuum undulators and wiggler (Figure 2).

The extra quadrupole triplet breaks the present four-fold symmetry of the magnetic structure of the machine and has a very significant impact on the transverse nonlinear dynamics of the electron beam. The experimental optimization of this optics, in terms of lifetime and injection efficiency, has led to choose different betatron tunes. The lifetime measured in the presence of the in-vacuum undulators is larger than the nominal one, demonstrating the beneficial effect of reducing the β_x-function in the short straight sections, which has a slight impact on the electron beam size. The quality of the photon beam will not be changed by using this new optics. ***The validation of this optics, in the presence of the two in-vacuum undulators, is scheduled for fall 2011.***

Short bunch production

A request of the users to work with shorter electron bunch lengths, for the time resolved experiments but also for Coherent Synchrotron Radiation (CSR) in the THz range, led to optimize a "low-alpha" optics. In a storage ring, the electron bunch length depends mainly on the electron energy, on the applied voltage in the radiofrequency (RF) cavities and on the α parameter, the momentum compaction factor. This α parameter characterizes the relative variation in trajectory length, depending on the relative variation in electron energy. For the nominal optics (α_{nom} = 4.4 10^{-4}), the bunch length at zero current is 15 ps RMS (4.5 mm) for a RF cavity voltage of 3 MV and when the current per bunch increases, the interaction of the electron beam with its environment leads to an increase in bunch length.

One way to reduce the bunch length is to reduce the parameter α by a significant factor. For SOLEIL, an optics has been optimized by Maher Attal (SESAME, Jordan) to reduce α_{nom} by a factor of 10, while maintaining a small horizontal emittance and large longitudinal energy acceptance. The optical functions are very different from the nominal ones, and the implementation of this optics on the machine requires the power supply reversal of one quadrupole and one sextupole families. Two critical points were studied. The first one was the stability of the transverse position (the smaller α becomes, the larger the transfer of energy oscillations in the horizontal plane), obtained by means of the two (slow and fast) position feedbacks. The second one was the injection efficiency. Its low value (10 %) is imposed by the specific nonlinear dynamics of this optics. To avoid frequent injections with poor efficiency, the lifetime was increased to 20 h by enlarging the vertical electron beam size.

Using this optics, the value of α was reduced by a factor of 100 for a current of 2 µA per bunch and bunch lengths of 3.2 ps RMS have been measured at α_{nom} /45. The natural bunch lengthening occurs from 50 µA per bunch and the benefit of the reduction of α, below α_{nom}/10 is maintained until 100 µA per bunch (Figure 3). The production of Coherent Synchrotron Radiation in the THz region has been successfully tested on the AILES[4] Infrared beamline and the optimal current per bunch has been optimized in order to increase by 3 to 4 orders of magnitude the emitted power[5]. Finally, a hybrid filling pattern, consisting of 20 mA in ¾ of the ring and an isolated 70 µA bunch, should satisfy both types of experiment. ***This optics is planned to be used in operation on the 10th and 11th December 2011.***

The group "Accelerator Physics", from left to right, standing: Marie-Agnès Tordeux, Amor Nadji, Director the Sources and Accelerators Division, Laurent Nadolski, Ryutaro Nagaoka. Sitting: Jianfeng Zhang, Pascale Brunelle and Alexandre Loulergue.

1. http://www.synchrotron-soleil.fr/SourceAccelerateur#Insertions
2. To install two beamlines from the same straight section, the axes of the two undulators have to be shifted at an angle.
3. http://www.synchrotron-soleil.fr/Recherche/LignesLumiere/NANOSCOPIUM
4. http://www.synchrotron-soleil.fr/Recherche/LignesLumiere/AILES
5. C. Evain et al., «Terahertz coherent synchrotron radiation at the synchrotron SOLEIL», IRMMW-THz 2010, Sept. 2010.

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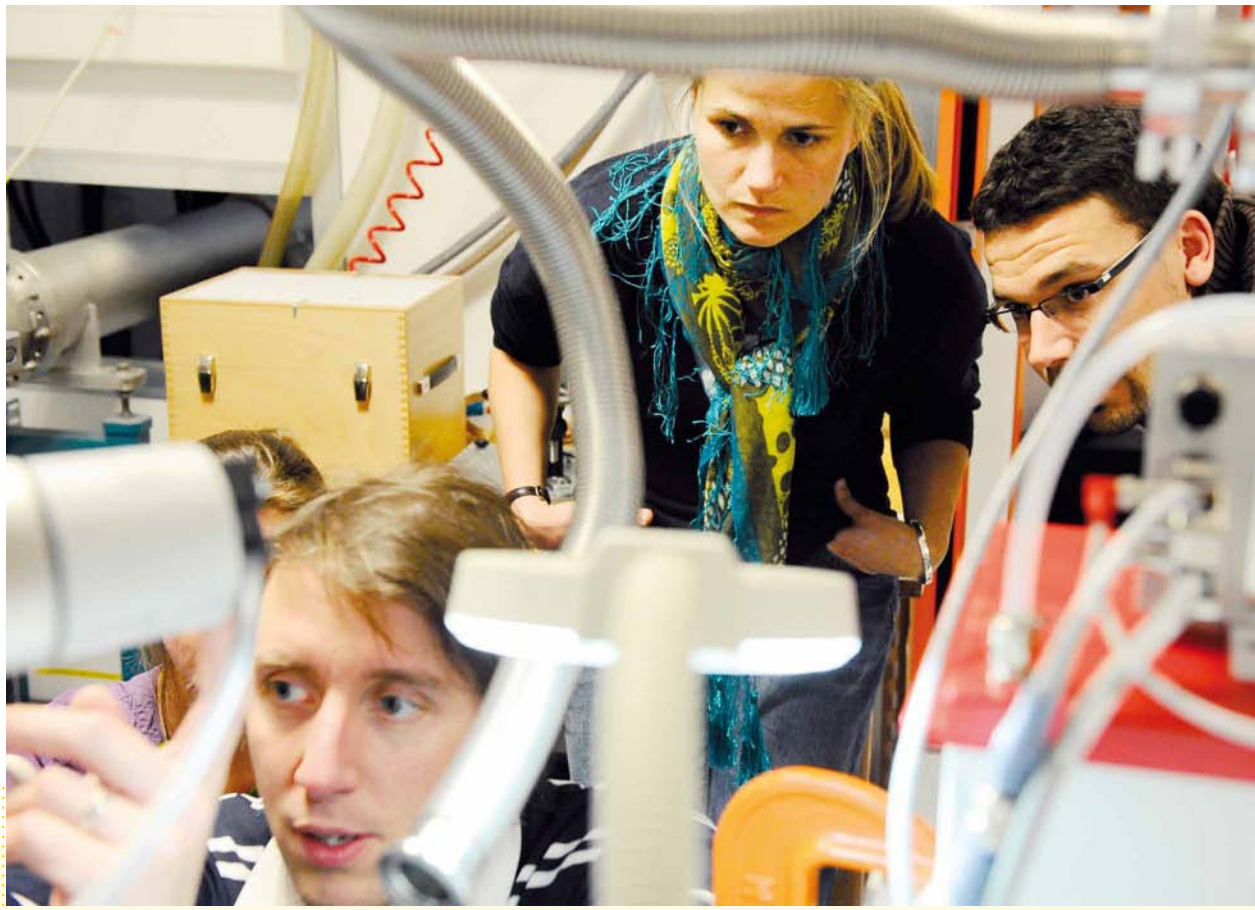
Chemistry in the light

Synchrotron radiation offers unique possibilities for analysis and understanding of basic mechanisms, of reactivity and the properties of assemblies and interfaces. Based on the electronic properties of the elements, these are the basis of chemistry, a fundamental discipline in the natural sciences. Because of its omnipresence in our lives, the importance of chemistry is sometimes forgotten.

Chemistry in the light

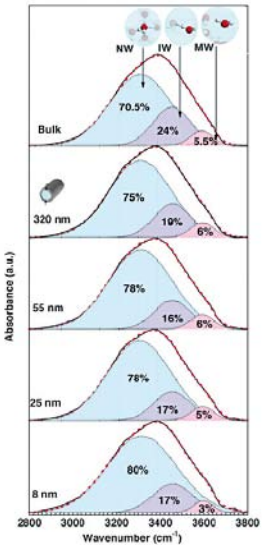


A part of AILES beamline's team

Synchrotron radiation offers unique possibilities for analysis and understanding of basic mechanisms, of reactivity and the properties of assemblies and interfaces. Based on the electronic properties of the elements, these are the basis of chemistry, a fundamental discipline in the natural sciences. Because of its omnipresence in our lives, the importance of chemistry is sometimes forgotten.

Current developments, considered revolutionary in terms of scientific and technological innovations, are accompanied daily by new applications that affect us all, whether involving energy, materials, health, transport or communications. These developments create new requirements in terms of fundamental knowledge, and quality control, with major economic and environmental impacts.

The interfaces of chemistry with other disciplines are growing and tools for characterization and analysis should make significant contributions. Third generation synchrotrons play a major role. Other sources of very intense and pulsed light will also soon contribute. Third generation synchrotrons, due to the energy range and the properties of the photon beams they deliver, will make it possible to implement all the methods for the study of light-matter interactions with ever growing accuracy, sensitivity and resolution, revealing the properties of matter so far unknown but essential to the requirements mentioned above. To compile this section, we relied on results from a selection of the SOLEIL beamlines whose research topics are most strongly related to chemistry. The articles brought together illustrate applications in diffraction, X-ray or IR absorption by elements, X-ray absorption with polarized beams, and a few applications that exploit the coherence of the X-ray beam. The variety of areas that these methodologies have a strong impact on is remarkable, ranging from biology to molecular self-assembly, CO_2 sequestration batteries, new heritage materials, molecular asymmetry to planetary atmospheres, or even the chemistry of nanoparticles and interfaces to that of complex, heterogeneous



Infrared spectra in the 2800-4000 cm^{-1} spectral range (OH stretching mode) of bulk water and in the case of water confined in 8 nm, 55 nm et 320 nm. The curves have been normalized. The infrared spectra have been measured in the ATR mode and then corrected from the variation in effective path length. The experimental data are the dotted points and the line corresponds to the global fit. The decomposition into the three Gaussian components is presented together with the percentage of their respective contribution.

AILES

Water network in nanoporous material: the role of interfaces

When water is confined in a nanoscale cavity, its structural and dynamical properties are modified with respect to those of bulk water. The properties of water confined in oxides present fundamental interest as well as practical relevance but very little is known about the state of water network in these rigid cavities. Vibrational spectroscopy is a powerful tool to study water as it can provide information about: (i) intramolecular vibrations above 1000 cm^{-1} ; (ii) librational band spanning from 300 to 1000 cm^{-1} and (iii) hindered translational bands in the 30–300 cm^{-1} energy range. Up to now, most results have been obtained on water confined in soft matter. Using the AILES beam, we have measured the infrared spectra of water confined in porous silica for various pore sizes using the ATR (attenuated total reflection) technique. We chose silica glasses containing a well-defined network of pores with size ranging from 8 to 320 nm. The infrared spectra of water confined in glasses of various pore sizes are reported for the O-H region together with equivalent spectrum for bulk water (see figure). The three Gaussians correspond respectively to water with fully established H bonds (NW), Intermediate (IW) and no or few established H bond (MW). The distinct differences compared to the bulk spectrum suggest that even in the large pores, the water network is significantly perturbed. The establishment of network is found to increase when the pore size decreases, indicating that confinement increases the H-bonding between neighboring water molecules. This can be related to some ordering induced by the rigid walls of the pores.
Furthermore, the partial filling of pores causes a significant modification to the water network, resembling heating of the trapped liquid thus suggesting a role played by the water/air interface. Indeed, these molecules are expected to present pendant bonds as can be found in water at higher temperature. It would be worth verifying the effect of these poorly connected water molecules on the reactivity.

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For more details: S. Le Caer et al, PCCP, DOI: 10.1039/c1cp21980d

« SOLEIL REVEALS THE INNER STRUCTURE OF MATERIALS, WHETHER ANCIENT OR RECENTLY DESIGNED BY CHEMISTS. IT IS AN UNPARALLELED TOOL TO PROBE THE ARRANGEMENT OF STRUCTURES ON AN ATOMIC SCALE, OR INVESTIGATE THEIR REACTIVITY OVER VERY SHORT TIMES. »

GILBERT CHAMBAUD, UNIVERSITY PROFESSOR, MARNE-LA-VALLÉE

and hierarchical organizations. In terms of methodology, temporal aspects also become important, both to study reaction kinetics (µs ... ms) or to elucidate ultrafast dynamics (ps... fs) that govern the evolution of a particular system over a longer scale.

Three additional beamlines

In addition to those beamlines for which the results are presented in this section, three other beamlines will also contribute to the "chemistry" area by 2013. These beamlines will benefit from major technological developments in X-ray optics and in their components, sensors and energy sources themselves. These developments are themselves the result of multidisciplinary research, and their implementation has been possible only by the joint effort of groups with complementary skills. It is now possible to go further and design and build beamlines over 150 meters long, to exploit the coherence of the beam and focus it down to the size of a few nanometers on the same sample, for multimodal studies (see article on page 9).

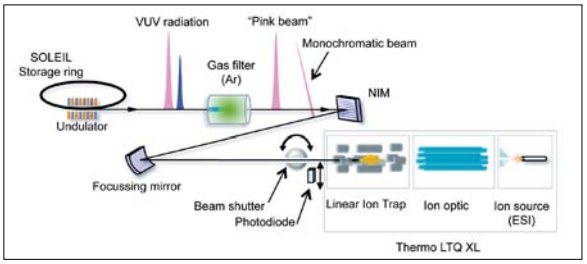
With such instruments available to scientific communities, SOLEIL synchrotron will provide methods and analytical tools to understand and explore matter on this scale with performances of an order of magnitude higher than the current possibilities.
Although spatial and three-dimensional imaging information on a nanoscale is obviously important in all areas of chemistry, temporal information is no less so.

To be continued on page 16...

DESIRS

Ion trap - Synchrotron coupling in the VUV: mass spectrometry and photoionization of biopolymers in the gas phase

An original device has just been developed, coupling a commercial ion trap mass spectrometer to the DESIRS beamline at SOLEIL, where synchrotron radiation in the VUV offers a wide tunability of the photon energy (5-40 eV), and a high photon flux (10^{12} - 10^{13} photons / s / 0.1% BP). Such an important quantity of photons allows obtaining a significant signal despite the low concentration of the species analyzed, such as positive or negative ions of biopolymers (a few tens of kDa) in the gas phase for which we want to understand the structure and photodynamics. The interaction between the photon beam and the stored ions takes place in a trapping volume of 1x1x 10 mm^3, requiring a very accurate alignment, beter than 100 µm, with respect to the beamline.
Initial results on cytochrome c, a heme protein of 12 kDa, show that the device can ionize the protonated molecule in numerous charged states (4+, 5+ ... 15+), previously produced by electro–spray and selected in the trap. Measurement of the ions yields for the different species present inside the trap after interaction with synchrotron radiation resulting from photoionization or photodissociation as a function of the photon energy, one obtain action spectra. From those spectra, spectroscopic information, such as ionization energy, may be derived. This energy increases with the charge state: the higher the protein is protonated, the higher the energy required to remove an electron (photoionization process). However, in the case of cytochrome c, this increase is quite low. This could be related to the relatively flexible secondary structure of the molecule, free of disulfide bonds, readily adopting different conformations in the gas phase, leading to a decrease in the ionization potential of the protein. Thus, these results already provide indirect information on the tertiary structure of the molecule under study, even without a fragmentation step - which could be implemented later.
Analytical and structural chemistry appears to be one of the areas of research for which this device is a promising tool. Being able to analyze large molecules such as proteins in the gas phase can also provide access to intramolecular relaxation mechanisms such as fragmentation, charge transfer or ionization processes. Finally, we can foresee the study of the effects of UV radiation on biological molecules, this time from a medical perspective (VUV phototherapy).



Schematic of the coupling of the linear ion trap with the DESIRS beamline. The VUV radiation produced by the OPHELIE2 (HU640 type) electromagnetic undulator of the beamline is filtered by a gas filter, removing the harmonic content of the source spectrum. The 7% bandwidth so-called "pink beam" may either be used directly for flux-hungry experiments (zeroth order of the monochromator) or be monochromatized by the Normal Incidence Monochromator (NIM), typical 10 meV bandwidths. A fast beam shutter is used to allow the incoming VUV light inside the trap for controlled duration.

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... Continued from page 15

Such information is essential with regard to reactivity, especially in the catalysis and energy fields. SOLEIL will provide these communities with the ROCK beamline dedicated to these studies, which will link the monitoring of catalytic or electrochemical phenomena on the tens of millisecond scale through the almost simultaneous analysis of the state and speciation of several elements, on the formation or transformation of components, reactants and products. Prospects in the field of time-resolved studies at SOLEIL are increasing in the short-term with the development of "slicing" techniques to transfer some energy to the electron bunches with an intense femtosecond laser and thus generate pulses of 100-150 fs X (see "Rayon de SOLEIL" n° 20, p 11). Its major advantage will be to aim for even shorter temporal resolutions.

Tools suited to a complex science

Chemistry is creative, situated at the interface with all other disciplines and as the basis of a science that is growing in complexity. Its connections with biology have always been strong and are now developing further through "biomimetic" and "bioinspired" innovations. There are major new directions in materials science, while supramolecular chemistry, nano-objects and their applications, organo-mineral hybrids, soft matter and soft chemistry are encouraging new and emerging fields.
This complexity in modern science, a potential for innovation and improvements, requires leading-edge platforms for analysis and characterization, of which the French Synchrotron, SOLEIL, will contribute fully with its developments and performance records.

SIRIUS

Semi-fluorinated alkane monolayers

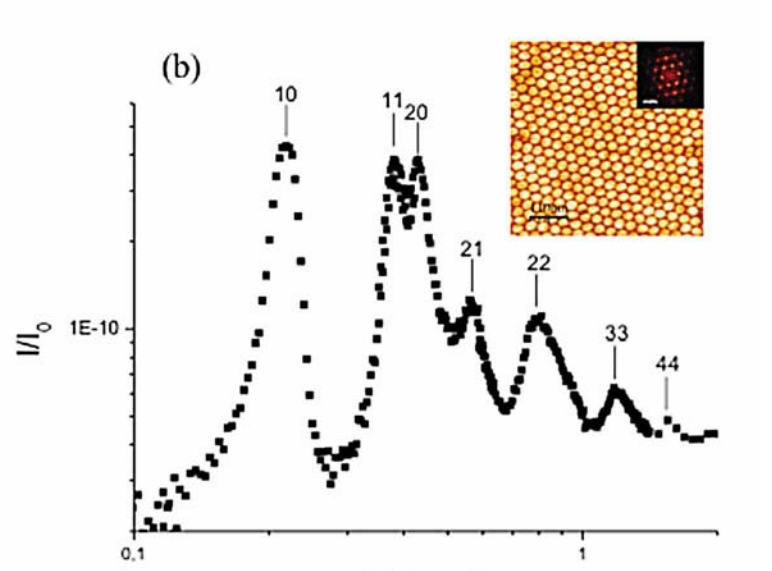
Semi-fluorinated alkanes ($C_nF_{2n+1}C_mH_{2m+1}$) are diblocks made of a hydrogenated, hydrophobic chain and of a fluorinated, hydrophobic and lipophobic* chain. These molecules have numerous potential applications ranging from pharmacology to emulsions due to these antagonist properties and to the ability of fluorinated chains to act as gas carrier. Their application often depends on their self-assembling properties, which remains poorly understood. In the case of monolayer at the air/water interface (Langmuir monolayer) or transferred on solid substrates, different techniques (Optical microscopies, AFM, grazing incidence x-ray diffraction or x-ray reflectivity) led to contradictory results. Structures have been proposed which range from homogenous monolayer organized where molecules are organized on a hexagonal network to domains formation of a few ten of nanometres size and of various shape (circular, elongated, spirals …) (ref. 1, 2). We studied this system by grazing incidence x-ray scattering at small and wide angles (GISAXS et GIXD). These techniques enable to determine the organisation of molecules (GIXD) and the supramolecular organisation (domains formation) or density fluctuations (GISAXS). These experiments evidence the formation of two lattices at two different length scales. Molecules are organized on a hexagonal lattice which parameter corresponds to the fluorinated chain cross section (2.7 nm^2). However the extent of the order is rather small limited to a few molecules. A second network is observed with a rather larger lattice parameter varying between 30 to 50 nm depending on the molecular architecture (ref. 3,4,5). This second lattice is compatible with a crystalline organisation of circular domains. The weak correlation length of the molecular network is explained considering that molecules are organized within a domain without any correlation between molecules of neighbouring domains.

The simultaneous observation of these two networks closes the controversy in the literature concerning the structure of these monolayers not only on the surface of water, but also on solid substrate.

Such simultaneous measurements at small and wide angle will be possible very soon on the same sample on liquid or solid substrate on the SIRIUS beamline at SOLEIL using the diffractometer. It may hosts dedicated sample environment and is equipped with two detector arms.

This will enable to use two different detection setups.

Langmuir trough installed at the ESRF for GISAXS experiment. The same setup will be available on SIRIUS beamline.



GISAXS spectrum of the F8H18 Langmuir monolayer at the bidimensional pressure of 5 mNm^{-1}. Diffraction peaks are indexed on an hexagonal lattice of 40.3 nm parameter. Inset: AFM Image of a F8H18 monolayer deposited by spin coating on a solid substrate showing the domains organisation evidenced by GISAXS on water substrate.

* Molecules were provided by M.-P. Krafft (ICS, Strasbourg) and E. Filipe (IST, Lisbonne).

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SAMBA

Quick-EXAFS and sulfur tracking

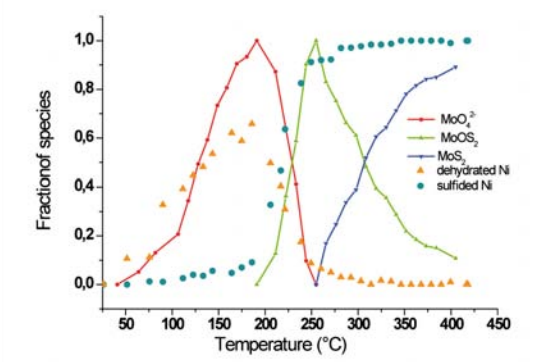

Figure 1 : Proportions of the different phases of molybdenum and nickel present during the sulfidation of the dried NiMo catalyst.

Figure 2 : Experimental setup.

To meet environmental regulations that have become ever more stringent and obtain "clean" petroleum products, the oil industry needs to improve hydrotreating processes and especially the hydrodesulfurization process (HDS) used to remove most of the compounds producing polluting sulfur molecules after combustion in an engine (SOx). The HDS reaction requires the use of a heterogeneous bimetallic catalyst generally consisting of a lamellar phase of molybdenum sulfide, MoS_2, and a promoter (Ni or Co) "decorating" the periphery of the MoS_2 slabs[1].
To form the active species, the catalyst, initially prepared in oxide form, is sulfided under H_2S gas flow. The activity and selectivity of the catalyst depend on both the good dispersion of the active phase on the support and the good decoration of the slabs by the promoter.
The improved performance of the catalyst thus requires control over the preparation of these active species and the detailed understanding of the reaction mechanisms involved.
With the new equipment on the SAMBA beamline[2] (Quick-EXAFS double monochromator, analysis cell[3], gas distribution system, etc…) we performed for the first time the simultaneous *in situ* analysis of the K edges of nickel and molybdenum on the same sample under realistic sulfidation conditions.
We were able to determine the catalysts sulfidation processes whose preparation parameters varied. Thus, during sulfidation of the dried catalyst, two intermediate phases were identified by XAS: an isolated molybdate MoO_4^{2-} that turns into an oxysulfide $\{MoOS_2\}$ before being converted into MoS_2. Furthermore, simultaneous characterization of the two elements Ni and Mo showed that sulfidation of the promoter begins before that of molybdenum.
The sulfidation of nickel starts in competition with the dehydration of the oxide species. Then the kinetics of molybdenum and nickel sulfidation have the same conversion rate within the same temperature range. These results led us to the conclusion that only in the second stage of sulfidation, does the active MoS_2 phase decorated by the promoter takes place and that about 10% of the promoter forms a non-active NiS_x sulfide phase.
Thus, from its genesis to its activation and the setting up its reaction conditions, each key stage in the life of the catalyst can now be characterized in situ under actual working conditions on the SAMBA beamline.

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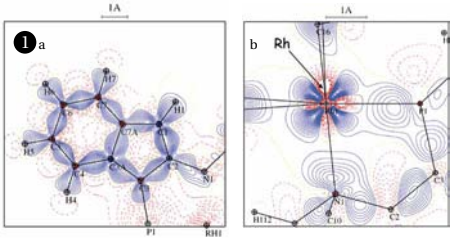
SWING

Nanocomposite materials with controlled anisotropic reinforcement triggered by magnetic self-assembly

Out of the whole range of polymeric materials, nanocomposites filled with hard colloids are the prolific in industrial use due to their strong reinforcement properties. However, the understanding and description of the local contributions of both fillers and chains on the macroscopic response of the material under deformation remain to be documented It is well-known that if the size of the reinforcing nanoparticles is decreased down to the nanometer range, the resulting increase of contact surface between fillers and polymer chains strongly enhances the mechanical properties of the nanocomposites[1]. The originality of our work is to apply a moderate external magnetic field during the processing of the nanocomposites resulting in a throng of anisotropic structures. The spherical magnetic nanoparticles, or fillers, self-organise giving rise to structures ranging from quasi-isotropic to homogenously dispersed aligned chains of nanoparticles. This was evidenced by a structural study combining Small Angle X-ray Scattering (SAXS) experiments performed on the SWING beamline, and Transmission Electron Microscopy (TEM) experiments (Figure 1).
The resulting anisotropy of the mechanical properties is spectacular. The Young modulus can reach values three times higher when the bulk material is stretched parallel counter to perpendicular to the chains.
This correlates quantitatively in a linear way with the anisotropy of the microstructure. Such a quantitative correlation is a new insight into both the processing of nanocomposites and the understanding of the relationship between the local filler nanostructure and the macroscopic properties of

CRISTAL

Chemical bonds revealed by high resolution X-ray diffraction



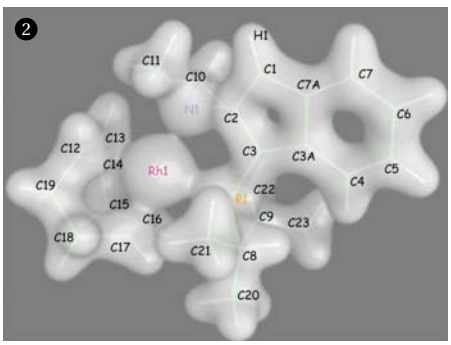


Figure 1a: Static deformation density map (electron density remaining after subtracting the contribution of core electrons) in the aromatic rings of a rhodium complex, clearly showing the C-C bonds (contour = 0.05 $e^-/Å^3$).
Figure 1b: Static deformation density map around the Rh atom (contour = 0.05 $e^-/Å^3$).
Figure 2: Three-dimensional representation of the total (static) electron density in a rhodium complex in its zwitterionic form.

X-ray diffraction is an indispensable technique for determining the atomic structures of materials in the crystalline state. It applies equally to the study of materials containing only a few atoms per unit cell as to compounds containing several thousands (eg, macromolecules such as proteins). The single crystal diffraction technique remains the most effective for the most detailed analysis. In particular, when the crystalline quality of the sample permits, the intensities diffracted by the crystal planes up to the larger angles of incidence (i.e. large scattering vectors) can be measured, making it possible not only to go down to atomic positions and displacements but also to electron distribution on the atomic and molecular scale.

Such experiments require very precise measurement of a large number of structure factors and are usually carried out using laboratory diffractometers and X-ray sources. However, in difficult cases where the sample is very absorbant and / or perfect (extinction phenomena), synchrotron radiation (e.g. the CRISTAL beamline at SOLEIL) remains the last resort (brilliance, wave length tunability and diffraction at short wavelengths) to minimize those effects that interfere with the accuracy of the measurements.

The contributions of core electrons and valence electrons can then be distinguished (multipolar model of the electron density), allowing modeling of the deformation of electron clouds under the effect of interatomic interactions and the formation of chemical bonds. Thus, these experiments not only give access to the arrangement of atoms in the motif or in the asymmetric unit but especially to interatomic interactions / chemical bonds and thus, more generally, the physicochemical properties of the materials under study.

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the material. From an applicative point of view, this opens the way for the design of new types of materials with the choice and optimization of the parameters governing the final material's properties (size of nanoparticles, filler content, applied field intensity...). From a fundamental point of view, such a quantitative correlation will allow to improve the understanding of current macroscopic mechanical models, by providing the relevant spatial length scale of such nanostructures.

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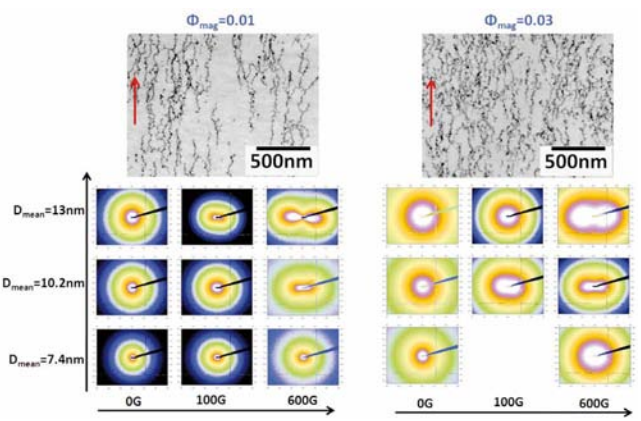

Figure 1 : Influence of the magnetic field on the nanoparticle's dispersion in two concentrated regimes as a function of the nanoparticle size. 2-D SAXS patterns, in reciprocal space, of the nanostructure of the films for Φ_{mag}=0.01 (left) and Φ_{mag}=0.03 (right) and for 3 nanoparticle mean diameters (from bottom to top: nanoparticles S (D_{mean}=7.4 nm), nanoparticles M (D_{mean}=10.2 nm) and nanoparticles L (D_{mean}=13 nm) for 3 magnetic fields (from left to right: B=0 Gauss, B=100 Gauss and B= 600 Gauss). Corresponding TEM pictures of films for Φ_{mag}=0.01 and Φ_{mag}=0.03 for nanoparticles L (D_{mean}=13nm) synthesised under a 600 Gauss magnetic field. The red arrow indicates the direction of the applied magnetic field.

XPAD detectors:
from the laboratory to industrialization

Hybrid pixel detectors will mark the end of the CCD camera era in some experiments using synchrotron radiation. The XPAD technology, born of a collaboration which involved SOLEIL, is one example. Its development led in 2010 to the birth of ImXPAD, a Marseille startup dedicated to its commercialization.

“Our contribution was to optimize all the XPAD performances and parameters”

Stéphanie Hustache, Head of the Detector Group – Experimental division

How did the XPAD project start?
To start with, hybrid pixel detectors were designed for particle physics. Then, groups who worked with these detectors saw that there was a real interest in using them around synchrotrons. Three competing detectors have thus emerged, which have been marketed. The Swiss synchrotron, Swiss light source (SLS) backed by the Paul Scherrer Institute, has developed a version called Pilatus. On the French side, scientists at the Centre de Physique des Particules de Marseille (CPPM), in collaboration with a beamline based at the Grenoble synchrotron (CRG-D2AM), have also developed this type of detector. At SOLEIL, we become involved at the beginning of the collaboration between Marseille and Grenoble to develop the next generation: the XPAD3.

What advantages does the XPAD have over earlier detectors?
XPADs are hybrid pixel detectors. They measure the number of incident photons and their positions with specific new characteristics compared to detectors that were previously used in synchrotrons.
Often, detectors based on CCD cameras are used. The accumulation of photons over a given period is measured, but they are not counted individually, whereas they are in these new detectors. They therefore have the advantage of reduced noise. In addition, it is possible to accumulate more photons without any notion of saturation, therefore gaining “dynamic range”. Finally, the reading speed is much greater than in CCD cameras: a few frames per second at best for a CCD compared to hundreds or up to one thousand of frames per second for XPADs.

What did SOLEIL contribute specifically to the design of these detectors?
The CPPM had the necessary expertise for the overall design of the microelectronics and sensor. At SOLEIL, our contribution was to optimize all the performances and all the parameters to create a detector useful for beamlines. Given their performance, we designed, with SOLEIL scientists, potential new experiments that would have been impossible with conventional detectors. Currently, we are working on improving still further the integration to the software framework of SOLEIL.

The development is nearing completion. Will the collaboration continue?
Nothing has been formalized yet but we plan to continue. XPAD detectors presently use silicon sensors as the technology has been well tested, but detection efficiency is quickly lost from 15-20 keV onwards. In parallel to the conventional silicon XPAD detector, we have also worked on the use of more efficient sensors and during the XPAD collaboration. We obtained very good results with CdTe-type sensors (cadmium telluride). The prototypes are still only available in limited size, so it is hoped to renew our collaboration with Marseille to create CdTe detectors for larger surfaces.

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IN BRIEF

SOLEIL AT THE CENTRE OF FRENCH NANOMETROLOGY

Created at the initiative of the “Laboratoire National de métrologie et d’Essais” (LNE) and the “Centres de Compétences en Nanosciences” (C’Nano) network and launched on the 6th October 2011 during the MesurExpoVision trade show, the Nanometrology Club now has over 110 French members, of whom 1/3 are industrialists and 2/3 research scientists. SOLEIL is a member of the limited first circle of club partners, which also includes the industrial group ARKEMA and the Essonne company 3S PHOTONICS.

SOLEIL PRESENT FOR THE SECOND CONSECUTIVE YEAR AT THE “RENDEZ-VOUS CARNOT” IN LYON

In the context of the 4th edition of this R&D rendez-vous for companies, aimed specifically at industrialists looking for scientific and technological know-how adapted to their innovation projects, SOLEIL was present on the 12th and 13th October 2011 among the 800 professional exhibitors of research partnerships and will participate in business meetings.

LIGHT, MATTER, ETC.

SOLEIL was a partner in the 2011 "Science of Art" festival devoted to light. Stefan Kubsky supported Flavien Théry, Patrick Le Fèvre and Matthieu Réfrégiers took a scientific approach to the work of Michel Semeniako, while Jean-Louis Marlats and his group contributed to the creation of "light does not stop there", an installation on display at SOLEIL from 14th November to 30th November.

→ http://doud.objets.lumiere.free.fr/
→ www.michel-semeniako.com

VISITS

Embark on SOLEIL Pursuit

Nearly 15,000 students and teachers have already visited SOLEIL. You too, perhaps. But you have probably never seen SOLEIL as Celine Lory, science communicator, has imagined it with Valérie Péduzy, teacher on secondment from "Rectorat de Versailles". Puzzles, treasure hunts, challenges, experiments, interviews and classes that began to discover this new approach in November have all been put to the test ... bursting with laughter! Another way to look at this great instrument, open to all.

→ **For more information** www.facebook.com/profile.php?id=100002948707819&ref=ts
www.synchrotron-soleil.fr/RessourcesPedagogiques/SOLEILpursuit

Stay connected

The second issue of SOLEIL's email newsletter has been published. To subscribe to it log on to: **www.synchrotron-soleil.fr/Soleil/ToutesActualites/newsletter**

Staff management, fascinating in itself, is particularly so at SOLEIL: as well as the close proximity of all personnel, there is an excellent scientific and technical environment.

Jean-Michel Cassagne,
Head of Human Resources and Social Relations at SOLEIL

SOLEMIO

THE FIRST EDITION OF THE SYNCHROTRON RADIATION SCHOOL "SOLEMIO" was held, from 2nd to 6th of May 2011, at the synchrotron SOLEIL. The purpose of school is to train European students and junior researchers to the newly developed X-ray synchrotron microscopy methods. In addition, the training also aims to highlight the complementarity between synchrotron microscopy techniques with other laboratory-based microscopies such as in nearby field or electronic microscopy. The school is organized in the form of lectures, tutorials and practical hand-on with the synchrotron as well as in laboratories. SOLEMIO is organized by SOLEIL and benefits from the strong participation of the laboratories of the Saclay plateau within the triangle of physics framework (RTRA). The school is free and open to French as well as to European public. The lectures were delivered by teachers and researchers from SOLEIL and by European experts. Practical hand-on is organized at synchrotron SOLEIL beamlines and in collaboration with laboratories from Saclay plateau and the University Paris-Sud. Given the success of the first edition (120 applications for 45 participants admitted), the school will be extended and will run in two parts: a common and recurring general core of lectures: synchrotron radiation, optics, techniques... and a more specific set of lectures focused on microscopy and its application to given scientific topics. This part should be renewed each year, emphasizing the contribution of microscopy and synchrotron radiation. Therefore, each year the school will have a different topic: Life sciences, chemistry, soft matter, materials, magnetism... The first edition held in May 2011 focused on "Magnetism and magnetic imaging." For the sake of community renewal, the next edition will be dedicated to the "Soft matter & Life Sciences" communities.

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See also:
→ **www.synchrotron-soleil.fr/Workshops/2011/SOLEMIO_School_2011**

28-29 June - Workshop "Magnetization dynamic in the light of pulsed X-ray sources: from storage ring to X-FELS"

THE AIM OF THIS WORKSHOP WAS TO PROVIDE AN OVERVIEW OF RECENT ACCOMPLISHMENTS in the field of magnetization dynamics and near future developments using pulsed X-ray sources. Ultra-fast magnetization dynamics has evolved to one of the most fascinating subjects in condensed matter physics and it is today at the focus of world-wide research activities. In these, X-ray based techniques play a key role due to their ability of combining time resolution with sub 50 nm spatial resolution, using the powerful element specific X-ray magnetic dichroism effect as contrast mechanism. A time resolution of the order of 100 ps is readily available through the inherent time structure of 3rd generation storage rings.
Their femto-slicing installations provide sub picosecond X-ray pulses. The limited intensity of these is overcome by X-ray Free Electron Lasers that provide X-ray flashes with unprecedented peak brightness and pulse shortness of down to a few fs.

Recent results obtained at these sources were presented during the workshop and an outlook was given on what will become possible with future pulsed X-ray sources. This discussion has been complemented by a series of talks presenting experimental highlights obtained with alternative approaches like all-optical laser techniques. The workshop, that took place in the Auditorium of SOLEIL, attracted an audience of about 70 registered participants from 6 different countries. The sessions of the meeting were dedicated to the following topics:
• the field of ps magnetization dynamic studied by X-ray PEEM, STXM and also IR laser sources. The talks were complemented by a discussion on the capacities of today's micro-magnetic calculations for the prediction of ps magnetization dynamics in nanostructured materials.
• New results from the BESSY slicing sources: study of magnetization dynamics occurring in rare-earth compounds; separation by fs time resolved XMCD of dynamical properties of spin and orbital component of the magnetization; past theoretical predictions in the field of ultra-fast magnetization dynamics, followed by a discussion on spin dynamics in multi-centered molecules.
• ultrafast magnetization dynamics probed using X-ray Free Electron Laser (X-FEL) sources.
• possibilities offered in the field of ultrafast magnetization dynamics by the Laser based X-ray sources
• new "pulsed X-ray source projects" in development in France.

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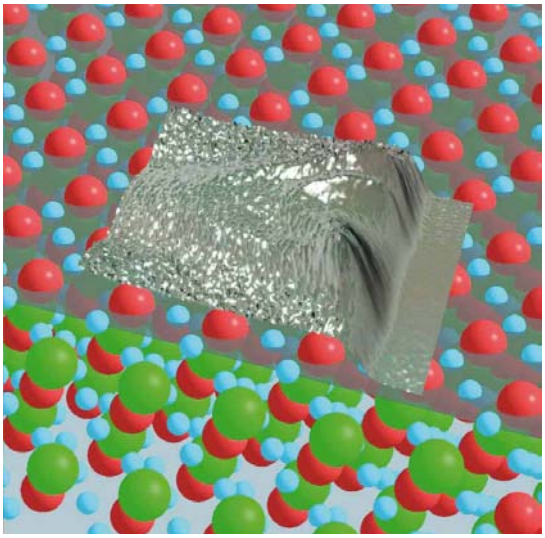
See also:
→ **www.synchrotron-soleil.fr/Soleil/ToutesActualites/Workshops/2011/MAGDYN2011/Program**

MICROELECTRONICS

New generation of transistors

Chips everywhere! In computers, mobile phones, bank cards, game consoles... integrated circuits or "chips" are in everything, and their ever-increasing performance in just a few decades has made it possible to reduce the size while increasing the power of everyday objects. In this race for the "always smaller, always more versatile," there is a great deal of active research. On the CASSIOPEE beamline a material is being studied whose properties suggest its use in a wide range of applications far beyond those of conventional transistors.



G. Sordi (ILL) et A.F. Santander-Syro (CSNSM)

3D view of dispersion E = f (k) (binding energy vs. wave vector) of electronic states of two-dimensional electron gas, as measured by angle-resolved photoemission spectroscopy, and the crystal structure of $SrTiO_3$.

Set on a square of silicon the size of a fingernail, a "classic" chip consists of millions of interconnected transistors, like so many electronic switches, using the binary language, 0 / 1. In the past ten years a new approach is being explored by scientists in order to diversify the functions performed by the components. In this context, transition metal oxides are candidates with interesting physical properties. These include magnetoresistance (related to electrical resistance, it is used in the read heads of hard disks) and thermoelectric properties (to convert heat into electricity). Strontium titanate ($SrTiO_3$), a transparent and insulating material, is one of these oxides. Experiments carried out, notably by the A.F. Santander-Syro group (CNRS – Univ. Paris-Sud 11), on the CASSIOPEE beamline have just shown that by breaking a piece of $SrTiO_3$ under vacuum, a two-dimensional electron gas is formed on its surface. The presence of this gas with metallic characteristics, which is simple and inexpensive to obtain, allows us to envisage the development of new devices combining the intrinsic properties of this material and the electrical conduction properties of electron gas. When can we expect non-volatile memory sticks in $SrTiO_3$, or transparent chips?.

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Reference : Santander-Syro, A. F. et al. "Two-dimensional electron gas with universal subbands at the surface of $SrTiO_3$". Nature, 2011, 469(7329): 189–193.

Alessandro Nicolaou, on the CASSIOPEE beamline.

Three important dates in the history of microelectronics

1 **1904 :** J.A. Fleming invented the first diode (or vacuum tube), considered as the starting point of electronics.

2 **1948 :** J. Bardeen, W. Brattain et W. Shockley, scientists at the Bell Telephone Company, invented the transistor. They received the Nobel Prize for Physics in 1956.

3 **2011 :** a "standard" transistor now measures 32 nanometers. At the beginning of the 1950s, they measured about 2 centimeters.

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