Crystallography at SOLEIL

On their beamline SIXS, Alina Vlad and Alessandro Coati are setting up the diffractometer coupled to a ultra-high vacuum chamber.

The International Year of Crystallography in 2014 will celebrate the centenary of Max von Laue being awarded the Nobel Prize in Physics,"for his discovery of the diffraction of X-rays by crystals". This is a small review of the history of crystallography, from ancient times to the latest synchrotrons.

Crystallography at SOLEIL

Erik Elkaïm, scientist at CRISTAL beamline, and one of the three diffractometers of the beamline, dedicated to powder diffraction.

Although crystallography or the "study of crystalline materials at the atomic level" has experienced unprecedented growth due to X-ray diffraction techniques, Man's interest in crystals did not, of course, start just a century ago.

Crystallography before X-rays

Since ancient times, crystals and gemstones have fascinated and made people reflect. The word "krystallos" or ice in Greek was used for the first time in the first century BC to denote quartz. Their geometric shapes, their different faces and translucent aspect evoking purity intrigued many, and these minerals quickly became subjects for study. And Plato gave his name to the definition of the five types of "Platonic solids", classified according to the shape of the regular polygons that constitute their convex faces.

Moving on to the end of the 18th century, Jean-Baptiste Romé de l'Isle and his assistant Arnoult Carangeot noticed that, the angles between the crystal faces of a given species are constant, whatever the lateral extension of these faces and the origin of the crystal. He thus established the law of the constancy of interfacial angles, paving the way for the scientific study of crystals.

At the same period, Abbé Haüy first had the idea (legend has it that he let a calcite crystal drop and continued to break the already broken pieces) that a crystal is a periodic stacking of small entities, creating facets and then faces on a large-scale. From this fundamental discovery was born the "integrant molecules" concept, later called the unit cell, corresponding to the basic unit or building block from which crystals are periodically assembled.

In about 1840, Auguste Bravais, going back to the integrant molecule, translated into mathematical functions the notion of periodicity. Using the concept of

DIFFABS

Ultra-fast acquisition of pole figures: a remarkable change in texture during silicidation

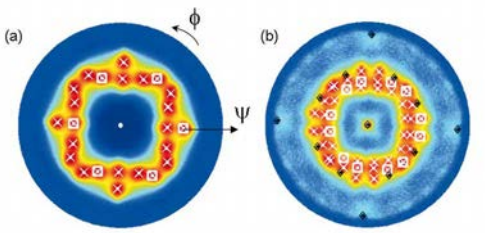

Figure 1 : (a)-(b) Pole figures measured for a Pd_2Si film on Si(001) around $111Pd_2Si$ and $212Pd_2Si$ reflections. $0° \leq \phi \leq 360°$ and $2° \leq \psi \leq 88°$. The white crosses (black diamonds) show the simulated position of the Pd_2Si film peaks (substrate).

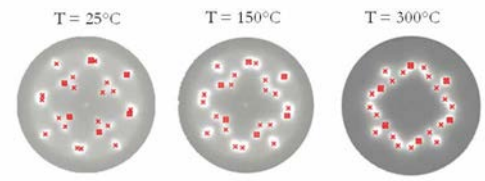

Figure 2 : Pole figures measured *in situ* for a Pd_2Si film on Si(001) around the $111Pd_2Si$ reflection at different temperatures.

Many physical properties of materials are closely related to their microstructure. The crystal structure of a material and its degree of crystalline order are significant parameters. Its texture can be described as the preferred orientation of crystallites in the material. In thin films, the texture influences, in particular, their mechanical, thermo-electric and magnetic properties.
The use of X-ray diffraction to obtain pole figures is a well-established method of characterizing texture. Recently, the use of a two-dimensional detector (XPAD) combined with continuous scanning of the motors on the DiffAbs beamline made it possible to collect complete pole figures in less than a minute.
To validate this original new experimental approach, the texture of the Pd_2Si phase was characterized *in situ* during its formation during the annealing of a thin film (<100 nm) of palladium (Pd) deposited on a silicon substrate (Si). Figure 1 shows two pole figures around the $111Pd_2Si$ and $212Pd_2Si$ peaks obtained after annealing at 200°C. They were used to determine the orientation of the (111) and (212) planes and the texture of Pd_2Si. The figures show the remarkable texture of the Pd_2Si phase characterized by four crystallographic orientation variants. Figure 2 shows changes to the pole figure around the $111Pd_2Si$ peak, as a function of temperature. The figures reveal a continuous rotation of Pd2Si grains around a crystallographic axis during annealing. This remarkable change in the texture can be explained by a diffusion mechanism during the formation of the Pd_2Si phase.
The ultra-fast acquisition of pole figures opens the way to *in situ* monitoring and analysis of the crystallography and texture formation in (ultra) thin films.

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"ALTHOUGH THE SEDUCTIVE POWER OF SOLID MATTER HAS LONG DEPENDED ON ITS PERFECT ATOMIC ORDERING, NOWADAYS ONE IS INCLINED TO VALUE THE LACK OF SUCH PERFECT PERIODICITY..."
HUBERT CURIEN, BORDEAUX IUCR CONFERENCE, 1990.

symmetry, he concluded that there are only 14 ways to periodically assemble sets of virtual points, the so-called lattices, where these virtual points are then not identified specifically. But it already appeared that 5-fold symmetry posed a problem.
This first "mathematization" of crystals was completed at the end of the 19th century by several scientists. They showed that, if each point in such lattices is identified by a motif, the constraints dictated by symmetry limited the number of different assemblies, and therefore the possible types of crystals to 230. This gave birth to the theory of space groups, still in use today.
It was not until the 20th century that this pattern was defined as a set of atoms, since the existence of atoms was not yet fully accepted at the time by all physicists.

From theory to practice

The discovery of X-ray diffraction by crystals in the early 20th century was made possible by the convergence of several streams of scientific research and a supportive environment at Munich University. On the one hand, researchers were working on X-rays, electromagnetic waves with such a short wavelength that no slit was thin enough to diffract them. On the other hand, scientists were trying to show that a crystal was indeed an assembly of atoms. This time, the notion of atoms was

To be continued on page 18...

PSICHE

Studying materials in extreme conditions

Figure 1 (up): Paris-Edinburgh cell
Figure 2: multi-anvil cell.

In the universe, materials are found much more rarely at "ambient" pressure and temperature than under extreme thermodynamic conditions; these include very high temperatures in stars and planets (including the depths of the Earth) and very low or zero temperatures in space. Being able to reproduce such conditions is therefore of major importance to geophysicists and geochemists, in order to understand these extreme environments better.
It is also now clear that there is a link between the form and function of materials, and changes to one will also lead to changes to the other. This is true in biology; a protein acquires its function through folding, but also in physics, since a change in the inner structure of the material affects its properties (mechanical, electrical, electronic and magnetic, etc.). In the first case, applying moderate pressure can force the molecule to take on this or that form, which it naturally adopts under given biological conditions, and studying the stability of these forms is often associated with different functions. In physics, due to much higher pressures, the interatomic distances of the sample are modified. Numerous possibilities to "play" with material properties present themselves to the scientists. Finally, in chemistry, in addition to the standard "initial composition" and "temperature", the 3rd parameter "pressure" also opens up numerous possibilities to synthesize new materials. Exploring all the conditions of this three-dimensional matrix is no longer possible by trial and error. The answer lies in in situ analysis: for a given composition, the pressure and temperature can be made to vary, and diffraction can track the creation of new phases until the material of interest is obtained. The aim is then to optimize the thermodynamic path that was followed.
Several facilities are available on the PSICHE beamline to tackle these issues:
• Diamond anvil cells, to achieve the highest pressures (300 GPa), at very low (10 K, cryostat) or very high temperatures (3000 K, laser heating). The volumes are, however, limited to a few µl, thus preventing re-use of the sample.
• Paris-Edinburgh cells, for larger volumes (a few ml) and high temperatures (2000 K) ; the pressure is limited to 15 GPa (IMPMC, P. and M. Curie University collaboration).
• Multi-anvil cells, which, thanks to a press weighing more than 2 tons, can be used to increase samples of a few ml to 40 GPa (in collaboration with the "Laboratoire des Magmas et Volcans", Clermont-Ferrand and the Néel Institute, Grenoble).
Finally, to cite some results already obtained on this beamline, opened to users in 2013: synthesis of Mg carbide (Paris-Edinburgh cell, an IMPMC and P. and M. Curie University collaboration) and using diffraction to determine its crystallographic form; study under pressure of the charge transfer in Prussian blue analogues in order to optimize their response to a light stimulus (ICMO, Paris-South University collaboration); monitoring the compressibility of nanoparticles, of Prussian Blue analogues, depending on their size (Coordination Chemistry Laboratory, Toulouse).

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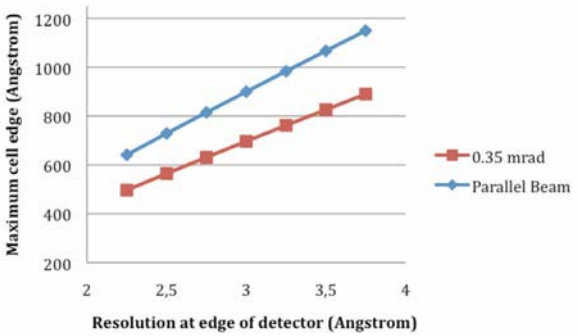
acquired, fractions of nanometers apart. And Max von Laue, a specialist on interference phenomena, urged that this form of radiation must diffract on such objects. In view of the results obtained in 1912 on a crystal of copper sulfate, it is clear that these scientists needed all their motivation to persevere and ultimately record the spots showing that diffraction had occurred. Von Laue received the Nobel Prize in Physics for this discovery two years later.
Then the Bragg, father and son, took up the mantle and improved the experimental setups, developed the first X-ray spectrometers and determined the first structures (KCl, NaCl, then ZnS, FeS_2, etc.). They received the 1915 Nobel Prize in Physics for "services in the analysis of crystal structure by means of X-rays" and founded a school that would train for decades prestigious crystallographers, such as Watson and Crick, co-discoverers of the double helical structure of DNA (Nobel Prize in Medicine in 1962), with Maurice Wilkins and Rosalind Franklin, unjustly forgotten at the time.

X-Rays, electrons and neutrons; crystals and quasicrystals...

Crystallography will then benefit from the development of knowledge and theories in quantum mechanics, which were able to show in 1930 that diffraction was not limited to X-rays, other particles than x-rays could be used as complementary probes: electrons and neutrons.
More recently, in 1982, the discovery of quasicrystals represented the last major paradigm shift in crystallog-

PROXIMA1

Bio-crystallography of macromolecules



Resolution of the unit cell on PROXIMA1, for parallel and divergent beams.

The PROXIMA 1 beamline is used to collect X-ray diffraction data from crystals of biological macromolecules (proteins, nucleic acids and their complexes). In particular the advanced properties of the SOLEIL source allow the beamline to provide a small, intense X-ray beam whilst preserving the beam parallelism (beam divergence around 0.3 milliradian at the sample position). Thus the beam produced by PROXIMA 1 is very well matched to the size, unit cell dimensions, resolution limit and sample mosaicity of large macromolecular complexes such as the ribosome. This point is illustrated in Fig 1 below, which illustrates the maximum unit cell dimension resolvable on PROXIMA 1 as a function of the resolution of the diffraction pattern, and for two different beam divergences (firstly with the beam divergence at the sample position of 0.3 mrad, which is the normal focused condition, and secondly with the beam rendered close to parallel after the mirrors, with a net divergence of 0.05 mrad). PROXIMA 1 uses a variable focus "bimorph" mirror system, coupled with spatial filtering, to adapt the size and divergence of the X-ray beam to the size and divergence acceptance of the crystal samples under study. In addition the possibility, given by the 3 circle "kappa" geometry goniostat, allows the crystal to be orientated in order to position a specific crystal axis in the X-ray beam (for example, to orient a long axis in a direction in which it is easy to control the beam divergence). Using this technology has enabled a number of large macromolecular complexes to be studied on the beamline. An example, where data collection needed careful optimization of the X-ray beam, is given below.

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Diffraction of X-rays leads to a diffraction image in "reciprocal space", where large distances in the real world lead to short distances in the diffraction pattern. Hence diffraction spots from large molecules tend to be much more closely spaced in the diffraction pattern than those from small molecules. In order to solve a structure with X-rays, the intensity of a maximum of Bragg reflections must be accurately measured. When the diffraction spots are too close together, this measurement is subject to error (or even impossible). Hence the X-ray beam size, crystal to detector distance and X-ray beam divergence have to be matched to the diffraction properties of the crystal.

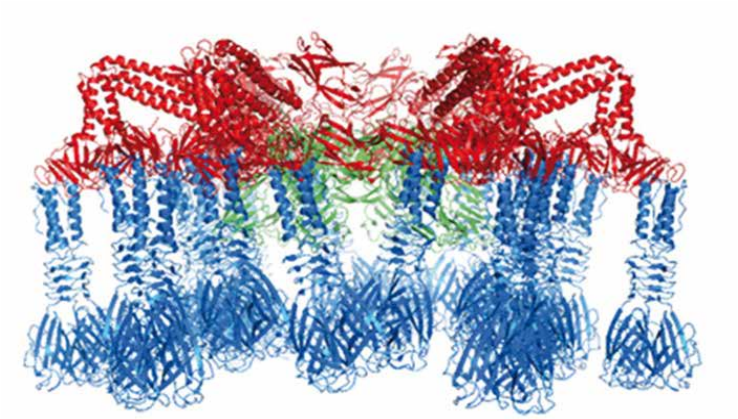
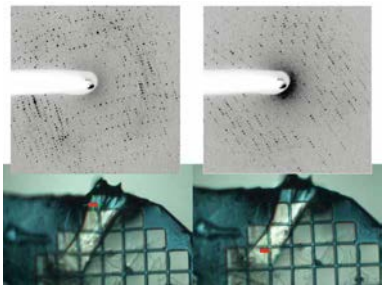


Figure 2 : The structure of the 1.8 MegaDalton baseplate of the phage TP901-1, composed of 78 proteins that self-assemble spontaneously, revealed an unexpected result: an alternative host adhesion mechanism. Unlike the infection strategy known for other phages of lactic bacteria, the Calcium ions, very present in the milk, do not play any role in the case of TP901-1. Furthermore, the way the 78 proteins forming this enormous complex articulate with each other once self-assembled is also original. These results are published in PNAS. Ref: Veesler, D. et al. PNAS, 2012, 109(23): 8954.

PROXIMA 2

Micro-focused X-rays for the Good, the Bad and the Ugly in Bio-crystallography



Two X-ray diffraction images taken from an "ugly" crystal on PROXIMA 2: On the left, a zone at the top of the crystal was exposed, and the diffraction pattern is messy with spots at irregular spacings. On the right, a zone at the bottom of the crystal was exposed, and the diffraction pattern is clean with spots at regular spacings. The red rectangle marks the position and size of the X-ray beam. The sample is mounted on a plastic support with a 25 micron mesh.

The 3-dimensional structure of a biological macromolecule (proteins, DNA, RNA, complex assemblies, viruses, etc) can often explain its function and even help the pharmaceutical industry to make new, more potent and tailor-made drugs.
X-ray crystallography is the most common method used to determine the structure of a biological molecule, but one of the biggest challenges faced by the scientist is to grow single crystals of sufficient size and quality. Typically, hundreds of crystallization conditions are tested before finding one that will produce crystals, which at first are often "ugly" – in other words, clumped, cracked and/or very small – just a few microns in size. Very rarely are these first crystals "good" – nicely shaped, big, single crystals which diffract X-rays well, and sometimes they are "bad" - nicely shaped, big crystals which DO NOT diffract X-rays at all. The bio-crystallographer needs to know whether his crystals are "good, bad or ugly", so that he can optimize the crystallization conditions to produce "good" crystals, which will lead to an atomic 3-dimensional model of the biological macromolecule under study.
On PROXIMA 2, which opened in March 2013, the powerful flux of X-rays are focused down to less than 10 x 5 microns (HxV, FWHM*) to allow users to collect from either very small crystals, or to single out the best zone of an "ugly" crystal. This fine focus improves the signal to noise ratio of the X-ray diffraction pattern from small crystals, but more importantly it can also avoid spurious X-ray diffraction patterns from unwanted material (precipitates, cracks or neighboring crystals). In larger X-ray beams, this extra unwanted material adds extra noise and/or complicates the diffraction pattern, which becomes impossible to process. The micro-focusing of X-rays is crucial for success in the most important and difficult structural projects. For the scientist wishing to obtain good diffraction, it can save months of work preparing better crystals. The screening for the best zone of a crystal is being automated on PROXIMA 2 with 2-dimensional "grid" scans. The diffraction images from these scans will be processed on-the-fly to score the zones and permit the scientist to choose the optimal part of the crystal to collect from. As such, PROXIMA 2 is complementary to PROXIMA 1, where the former permits scientists to screen "ugly" crystals, while the latter employs more specialized structure determination methods.

*FWHM: Full width at half maximum

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raphy, with the questioning of one of the fundamentals of this science: the possibility of 5-fold symmetry in materials with long-range ordered atomic structure (proved by X-ray diffraction). Quasicrystals still remain a relevant research topic.
In synchrotrons, crystallography naturally occupies a prominent place. But structure determination is not limited only to studies using X-ray diffraction, absorption spectroscopies (XANES and EXAFS) can also bring essential structural information...

Thirteen SOLEIL beamlines

Indeed, although crystallography and X-ray diffraction have been historically linked for over a century, the International Union of Crystallography has enlarged the circle by adding X-ray absorption in the study techniques. At SOLEIL, thirteen beamlines are diffraction or scattering beamlines, and two of them allow X-ray diffraction and absorption to be combined. Five research areas emerge from studies conducted on these beamlines.

■ Structural biology

PROXIMA1, the first biocrystallography beamline at SOLEIL, has published more than 300 scientific articles since its opening to users in 2008, reflecting the quality and efficiency of the beamline and its running group, notably in solving such structures as macromolecular complexes, as explained in more detail on page 19. Since March 2013, PROXIMA2 complements PROXIMA1, opening up prospects in terms of crystal screening (see above) and in situ data

CRISTAL

Distribution of cations in materials for photovoltaic cells - resonant X-ray scattering on a Cu_2ZnSnS_4 single crystal

The development of thin film photovoltaic cells without Si, but with high conversion efficiency, is a very active area of research in the current economic climate. Although thin film solar cells based on Cu(In,Ga)Se_2 (CIGS) have already proven themselves, finding new non-toxic materials containing elements abundant on Earth is crucial for the large-scale production of photovoltaic cells. Compounds derived from Cu_2ZnSnS_4 (CZTS) appear to be very good candidates as absorbents in such solar cells [1]. A detailed knowledge of the crystal structure is crucial for improving performance. As Cu and Zn can occupy the same crystallographic site [2,3], the nature of the distribution of these atoms (ordered or disordered, in this case leading to a more symmetrical structure - see figure 2) is crucial in order to link it with their electronic properties. However, as the atomic scattering factors of these atoms are too similar, conventional X-ray diffraction experiments are not suitable in this case. The technique called "resonant diffraction" (or anomalous diffraction) is then essential to differentiate these atoms of adjacent atomic numbers that can occupy the same crystallographic site.

The energy of the X-ray beam has to be adjusted very precisely, so that it is near the absorption edge of one of the elements. As a result, for this element, the number of electrons contributing to the X-ray diffraction is artificially reduced (figure 1), thus increasing the contrast with the adjacent element. The use of synchrotron radiation is therefore essential since only this X-ray source allows for a precise adjustment of the beam energy. The precision required for the beam energy is very high ($\Delta E/E \sim 0.01\%$) and must be maintained over the entire duration of the experiment (from a few hours to several days). A resonant x-ray diffraction experiment at the K-edge of Cu on a CZTS single crystal was performed for the first time, on the CRISTAL beamline. The results obtained made it possible to clearly distinguish the Cu and Zn atoms and thus specify the nature of the distribution of this disordered kesterite-type structure within the cationic planes (figure 2b). The study will now continue to try to understand why a deviation from a 2: 1: 1: 4 stoichiometric ratio seems to improve the conversion efficiency.

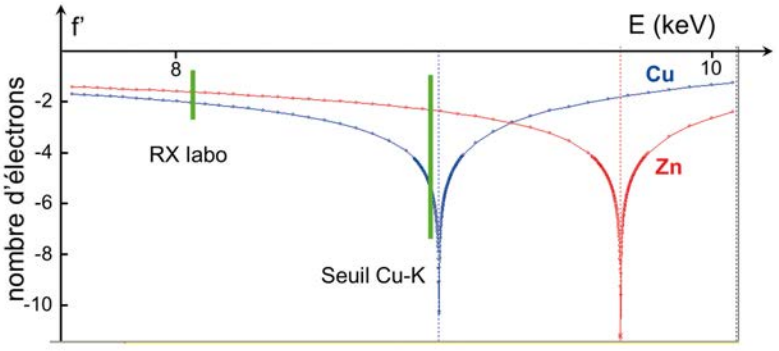

Figure 1 : Evolution of the f' component of the X-ray diffusion factor as a function of energy. The Cu/Zn contrast increases markedly close to the absorption edge of copper.

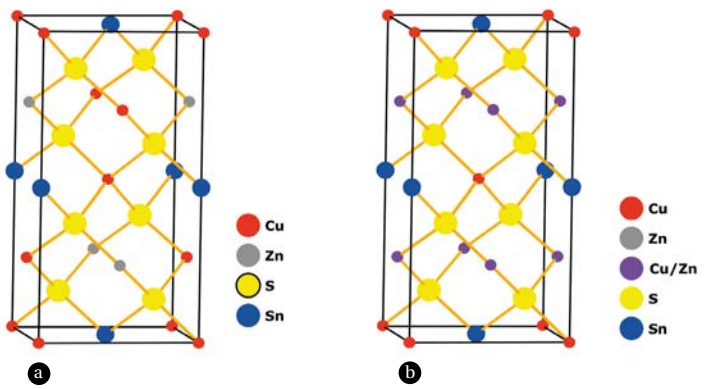

Figure 2 : Kesterite-type structure of Cu_2ZnSnS_4 a) ordered (space group $I\bar{4}$) or b) disordered (space group $I\bar{4}2m$)

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References:

[1] Todorov, T. K. et al. Adv. Energy Mater. **3,** 34 (2013)
[2] Fontané, X. et al. Alloys Compd. **539,** 190 (2012)
[3] Choubrac, L. et al. Phys. Chem. Chem. Phys., **15.** 10722 (2013)

SIXS

Nanoalloys: crystallographic structure and stability

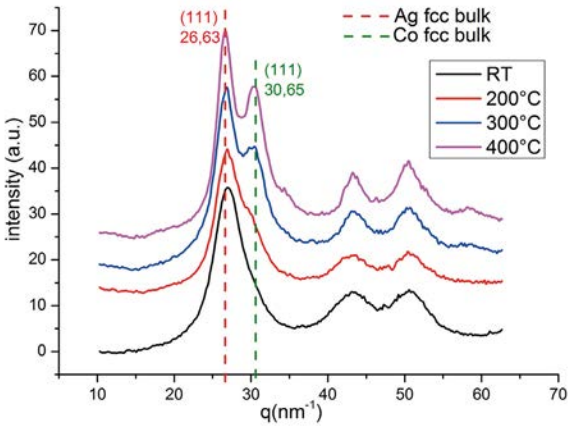

Figure 1 : X-ray diffraction spectra of a Ag@Co nanoalloy during annealing.
Figure 2 : schematic representations of a nanoparticle, at room temperature (a) and after annealing (b).

The physicochemical properties of materials change deeply depending on the amount of material involved. Thus, by adjusting the size of objects, in particular on the nanometric range, it is possible to highlight or reveal new properties.
A very significant example is that of metal nanoparticles, potentially very interesting for use in the catalysis or magnetic memories fields. Their properties can be tailored by the size reduction and also by alloying with other metals on the nanoscale. These «nanoalloys», the composition of which can be controlled, can be obtained by various methods, either from chemical solutions, or physically via deposits on substrates, etc.
The SixS beamline can be used to study nanoalloys prepared either by the simultaneous deposition (co-deposition) or by sequential deposition of different elements. The properties of nanoalloys depend on their size, their crystallographic structure and the organization of the chemical species. The beamline's experimental setup allows to prepare the nanoalloys in situ in order to study the evolution of their structure, organization and morphology during growth and/or heat treatment, or even a chemical reaction. In addition, using anomalous X-ray scattering (energy measurement close to the absorption edge of elements), the measurements can be selective to one element of the alloy.
The example shown in Figure 1 is that of an Ag@Co nanoalloy obtained by co-deposition under UHV. This is wide-angle x-ray diffraction spectrum, which permits to follow the evolution of the alloy atomic structure during thermal annealing. At room temperature, the Co signal is not present, so it can be inferred that the nanoparticle is composed of a core of Ag atoms surrounded by an ultra-thin Co shell stabilized on the surface by partial oxidation, a metastable configuration, as sketched in Figure 2a. During annealing under UHV of the sample, the emergence of a contribution corresponding to metallic Co is increasingly observed, suggesting

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collection, for example in microfluidic chips.
Small-angle X-ray scattering (SAXS) is also one of the techniques used in structural biology. The SWING beamline has provided users, since 2009, with a versatile sample environment (see Rayon de SOLEIL 18, p.9) for automatically injecting and/or purifying biological samples before SAXS analysis. The device has been optimized since February 2013 to gain even more time in sample studies.

■ *In situ* and *operando* experiments

The experiments carried out at SOLEIL are tending to become more often a study of a process over time, not only to characterize a "frozen" sample. Fine examples have been obtained on CRISTAL, when monitoring the composition of lithium batteries in operation (see Rayon de SOLEIL 20, p.4) or the growth of carbon nanotubes. The box on p. 17 describes how DIFFABS provided in situ analysis of crystallography and texture formation in thin films. For its part, the SIXS beamline allows the *in situ* study of thin layer formation or the evolution of a surface, interface or nano-objects during deposition, thermal treatment or reaction (see example above). In this case, the coupling of diffraction and scattering (GISAXS) is used to monitor changes in samples on different scales, from atomic structure to the morphology of nano - objects. The SIRIUS beamline, complementary to SIXS with lower energy X-rays, will soon extend these possibilities.

a segregation of the Co and Ag; in this case, the nanoparticles can present a configuration so-called Janus nanoparticles, as sketched in Figure 2b.
Although wide-angle X-ray diffraction give insights on the atomic structure, SixS allows to perfom grazing incidence small angle X-ray scattering measurements, which provide information on the shape, size and dispersion of nanoparticles on the substrate. In the case of Ag@Co nanoalloys, we observed that the nanoparticles average size increases from 2.6 nm (about 800 atoms) up to 4.1 nm under annealing and their average distance from 3.4 nm to 5.7 nm. This information is essential for interpreting the link between their structural, chemical and morphological reorganization.

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Extreme conditions

On the PSICHE, CRISTAL and DIFFABS beamlines, high pressure analytical techniques are available by using different press systems (see PSICHE insert p.18). Various experiments can be performed: powdered samples or single crystal, angular or energy dispersive diffraction eventually coupled with absorption spectroscopy measurements. The coupling is even more important in this field than in others, in order to obtain a full structural characterization.
These studies are often carried out as a function of temperature, which in some cases can also be extremely high or low.
However, at SOLEIL, "extreme conditions" also include the "radioactivity" issue. The MARS beamline is now authorized by the Nuclear Safety Authority (ASN) to receive samples whose radioactivity is equivalent to 15 kg of uranium isotope 238; the first experiments took place on 20 September 2013. MARS has a unique setup capable of combining diffraction and X-ray absorption measurements on the same radioactive sample, with the additional possibility of using a microbeam, particularly suited to the study of heterogeneous materials.

"Playing" with the X-ray beam characteristics

Being able to distinguish between two elements with neighboring atomic numbers and likely to occupy the same crystallographic site has been made easier by the use of the neutron diffraction technique. When using X-rays, the technique known as resonant or anomalous diffraction, requiring the fine energy tuning of the available synchrotrons beam, is often the only solution (see CRISTAL insert p.21). CRISTAL, SIXS, DIFFABS, SIRIUS, PROXIMA1 and PROXIMA2 beamlines use resonant diffraction.

Another feature of the X-ray beam is coherence, the use of which has flourished with the development of third generation synchrotrons. This gives access to both slow dynamics through the study of the speckle intensity fluctuations (as with lasers), and imaging, by solving the phase determination problem encountered in conventional diffraction techniques.

In the hard X-ray field, there have been impressive results on CRISTAL (see Rayon de SOLEIL 22, p.24) and, in weaker energy ranges, magnetic-domain imaging, based on holographic techniques, gives images with almost nanometric resolution. (cf www.synchrotron-soleil.fr/Soleil/ToutesActualites/2012/SEXTANTS-IMAGERIE).
From 2014, it will be possible to carry out tomography experiments on the nanoscale, using the coherence properties of hard x-ray beams, on the Nanoscopium beamline (see Rayon de SOLEIL 21, p.9).

Time-resolved experiments and femtoslicing

Time-resolved experiments are central to the research strategy at SOLEIL in order to "see" processes on different time scales. One of the most recent developments has been operating the storage ring in the so-called "low alpha" mode (see Rayon de SOLEIL 22, p.14), making it possible to go down to temporal resolutions of a few picoseconds; On CRISTAL this type of mode is used to study ultrafast phenomena.
By the end of 2013 the first tests will have been carried out with the so-called "slicing" technique (see Rayon de SOLEIL 20, p.11). Resolutions of the order of hundreds of femtoseconds will then be reached. SOLEIL will ensure the development of all possible complementary techniques for experiments using X-ray lasers (European XFEL, LCLS).