Tutorials

Sunday, September 4

T1

SCANNING METHODS WITH MICRO- AND NANO-FOCUSED HARD X-RAY BEAMS

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The use of small lateral size X-ray beams (in the 10 keV energy range) in studying materials lately has become of increased interest. While still benefiting of the power of various analysis methods using X-rays (be it imaging, spectroscopy, diffraction, etc.), they open now the possibility of accessing properties with lateral resolution, in a microscopy like approach. Moreover, new research applications were possible.

It is the progress in X-ray optics which enables obtaining extremely focussed and intense X-ray beams, with lateral sizes as small as 10 nm. An overview of the different X-ray focusing options available nowadays will be given, and their advantages and inconveniences will be discussed. These features are intimately interconnected to the type of the experiment intended and the experimentalist should have in mind their capabilities and limits when designing and performing an experiment, via a judicious choice involving very often compromises. Quantities like photon flux, spot size, chromaticity, coherence, etc. will have, most of the time, to be carefully balanced and prioritized. This issue is also strongly linked to the related instrument needed to perform a certain type of experiment; some characteristics and possible requirements will be also addressed.

Several uses of focused beams in scanning methods will be detailed via examples, in an attempt to show some study cases but also to highlight as well some 'tips and tricks' while performing these experiments: blocking points and ways to overcome them, choice of setup and focusing device, etc. The examples are mostly related (but not only) to materials science and X-ray diffraction (XRD) technique, a powerful and non-destructive analysis tool. It can give access to the lattice parameter of crystalline structures and thus to strain fields, with an accuracy hardly achievable by any other technique. In standard XRD experiments, large areas (square millimetres) of the sample are characterized, yielding averaged, statistical properties, contrarily to the use of microscopy approaches. It became of high interest to measure the structural properties locally while still making use of all the advantages of XRD. This is particularly valid for inhomogeneous samples, like it is the case of small sized objects (micro- and nano-structures): the "average" property might simply be meaningless in the case of complex structures and local characterization is needed in order to understand the change in physical properties, when the nanoscale is approached. Focused X-ray beams are used to localize and measure one by one single nanostructures. In a raster scanning mode, a 2-dimensional image of the sample is recorded, using the scattered signal as contrast. Then, on specific single objects, the x-ray scattered signal is recorded and analysed / modelled, to give access to the shape, strain and composition inside the object with sub-micron resolution. Several examples will be shown, starting with samples closer to "model" systems and finishing with a local probe XRD study on III-V semiconductor heterostructures for optoelectronic applications in telecommunication field: by addressing shape, strain and composition at the nanoscale, the spatially resolved micro-/nano-diffraction from low-dimensional systems is expected to play an important role in the understanding of the structure properties of nanomaterials, and provide a better control on their fabrication and functionality.

With the recent developments related to detectors and data acquisition systems (hardware), but as well to the high stability and intensity of the X-ray beams, the need of rapid data acquisition schemes emerged, in order to access either to large mapped areas (with good lateral resolution) or possibility to acquire complete datasets in short intervals of time (is situ experiments, sample annealing, ...). Such a rapid scanning approach, possibly using multiple types of detectors (and thus simultaneous multiple contrast) will be presented and detailed via several examples, related to materials science and archaeology respectively. Issues like data acquisition, handling, mining, storage and exploiting will be discussed in the context of large datasets.

T2

X-RAY IMAGING METHODS

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An usual, often implicit, assumption when investigating a sample with X-rays, is that the sample is homogeneous, and that by illuminating the whole volume the result corresponds to the one we could measure locally. This assumption is not valid when dealing with an inhomogeneous sample: in this frequent case we need to use an "imaging" type approach, i.e. measuring "locally" to produce a map where each of these "local" measurements corresponds to an homogeneous area. The required size of the "local volume" is a function of the size of the inhomogeneities. The "image" resulting from the ensemble of local measurements can exhibit contrast, making internal features distinguishable.

Two main experimental schemes, shown on figure 1, allow these "local" measurements to be performed: a) a parallel beam is used together with a pixelated detector, in such a way that each pixel corresponds to a given volume in the sample, and the spatial resolution of the image associated with the pixel size (lensless technique) b) the beam is focused on a given region of the sample, which is scanned in order to obtain a series of "local" measurements on these regions, the spatial resolution being associated with their size (X-ray lenses are required in this case). X-ray imaging started over a century ago, with the discovery of X-rays, allowing the visualization of the volume of systems opaque to other probes (in particular visible light). It was immediately noticed that these radiographs, which exploited the variation of the absorption of the X-ray beam when going through the sample, constituted an important tool for medical diagnostic. The applications of radiography to materials were also rapidly developed. For several decades, absorption radiography remained the only form of X-ray imaging.

About fifty years ago, Bragg diffraction imaging developed into practical use [1-3]. This "diffraction topographic" approach apply to single crystals and basically consist of mapping the intensity and direction of the locally Bragg diffracted beams. It reveals defects in the bulk of single crystals through their associated distortion fields and the modifications they introduce into the diffraction process. These techniques helped developing the production of large, practically perfect crystals, for the microelectronics industry. Figure 2 shows an example of an "early topograph" [4], which shows dislocations in a silicon crystal produced in the 1950's. These techniques extended beyond semiconductor crystals, and are useful for investigating induced or growth defects, domains, phases…. Their range of applications dramatically grew when SR became available to condensed matter scientists [5].

A major breakthrough was performed when the computing improvements allowed producing three-dimensional (3D) images from a series of radiographs recorded at different angles ("tomography")[6]. On the other hand

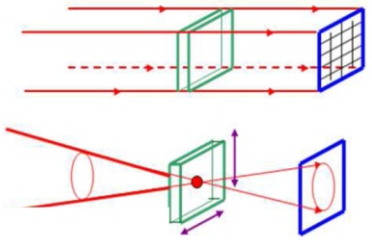

Figure 1.

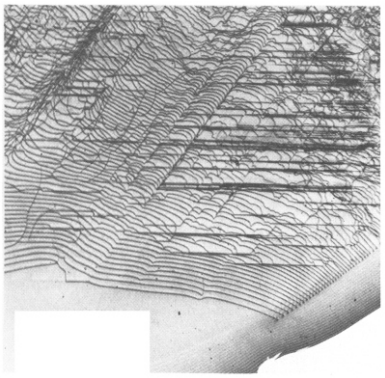

Figure 2.

interferometric devices opened the way to the first phase images.

All the X-ray imaging methods benefited from the use of synchrotron radiation (SR), the improvements being even more pronounced when considering the new features of modern, "3d generation", SR machines. Real time and high spatial resolution experiments are nowadays routinely performed, and the high coherence of the beam allows exploitation of a novel form of radiography, in which contrast arises not only from absorption differences but also from phase variations across the transmitted beam [7-10]. We will briefly describe several "parallel beam" phase contrast imaging developed over the last years ("Fresnel regime"), in particular the simple "propagation technique". When using the coherent fraction of the beam in the "Fraunhofer regime", techniques like Coherent Diffraction Imaging (CDI) and, more recently, ptychographic CDI, allow, from the recorded patterns, to reconstruct with very high resolution the local features of the sample. These "CDI techniques" constitute the topic of other lectures of the present "companion school".

On the other hand the brilliant sources of the modern SR facilities, coupled with new X-ray optics, allowed micro-beams in the μm/nm range to be achieved. This leads to scanning images, probing for instance the fluorescence or the absorption near the absorption edges, with spatial resolutions reaching the few tens of nm range. Nearly all X-ray-matter type of interactions can be exploited within the frame of what we called "imaging approach", i.e. a set of "local" measurements constituting a map or image [11]. In the present lecture the emphasis will be put on hard

X-ray (6-200 keV) images resulting from variations of absorption, phase, or Bragg diffraction, in their 2-or 3D ("microtomographic") forms. Among them, we will in particularly consider 1) the extensions of absorpt the absorption edges (angiography), and, mainly, 3D imaging with a spatial resolution in the sub-µm range (microtomography) 2) the advantages associated with phase imaging (2D and 3D) using the simple "propagation" technique and 3) Braggdiffraction imaging on single crystals (historically "X-ray topography", this being the origin of the "XTOP" of our series of Conferences) and polycrystals : as an example, figure 3 shows the reconstruction, using "Diffraction Contrast Tomography" [12], of the grains constituting the polycrystalline stainless steel sample.

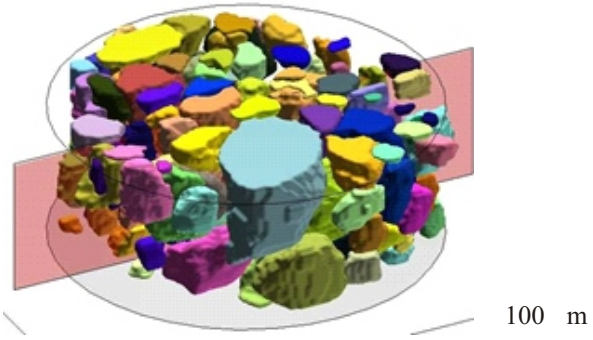

Figure 3. Reconstruction, by using the transmission and diffraction signals, in a tomographic mode, of the grain structure of a polycrystal.

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T3

LABORATORY X-RAY DIFFRACTION AND SCATTERING EXPERIMENTS

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Table-top micro-sources of brilliances comparable to second-generation synchrotron sources have proved to be extremely efficient for ex-situ experiments. The X-ray MicroImaging Laboratory (XMI-L@b) [1,2], described in this lesson, consists of a three-pinholes SAXS/WAXS camera coupled to a table-top rotating anode micro-source and equipped for SAXS (GISAXS), WAXS (GIWAXS) data acquisition and scanning SAXS microscopy. Empowered by original algorithms [3-5], the XMI-L@b has been recently used for the supramolecular and submolecular X-ray imaging of nano- and bio-materials [6-9].

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T4

X-RAY IMAGING WITH PHASE-SENSITIVE METHODS

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Hard X-ray imaging, i.e. above about 2 keV, offers the possibility to image bulk thick specimens non-destructively, and to further combine images at different incidence angles of the beam to provide 3D images of the specimen. It can also be beneficial for 2D imaging in systems that operate in a harsh environment involving gases, fluids or a bulky setup around the sample, where soft X-rays, i.e. below about 2 keV, cannot penetrate the whole system. In the case of soft X-ray microscopy, bulk setups can also prevent the optics to be placed close to the specimen. The challenge of hard X-ray imaging is that the absorption of materials is weak, offering a poor contrast for imaging.

Phase contrast is nowadays commonly exploited in synchrotron sources to image specimens with hard X-rays. As an example, propagation-based techniques [1] allow the acquisition of images with very short exposure times and hence low dose [2], enabling e.g. tomographic acquisitions in 3D in less than a second [3], or imaging animals *in vivo* [4]. Another method using grating interferometry [5] has been implemented in laboratory sources and is being developed as a potential tool for early detection of breast cancer [6]. X-ray microscopy with sub-micron resolution is considerably more difficult to achieve with hard X-rays compared to soft X-rays because of the difficulty to fabricate X-ray lenses for high energies. Nevertheless, optical microscopy schemes like Zernike phase contrast have been successfully implemented for hard X-rays [7], and propagation-based methods are also successfully used in combination with a divergent beam arising from a secondary point source [8]. Finally, coherent diffraction imaging methods offer a way to image specimens with quantitative phase contrast with a resolution which is not limited by any optics [9].

In this tutorial I present an overview of these X-ray phase contrast imaging techniques, explaining their basic principles and showing examples of their applications in different fields of research.

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T5

TUNEABLE PHOTON ENERGY METHODS

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A lecture at the XTOP 2016 Companion School will be given on methods that combine X-ray imaging with different ways of tuning the X-ray photon energy and utilising spectroscopic contrast modalities.

The main topics of the lecture are:
- Theory - Anomalous scattering and X-ray spectroscopy
- Instrumentation – Optics, detectors, software
- Research - State-of-the-art spectral imaging experiments
- Future prospects within the research filed

X-ray imaging provides a great wealth of spatial and phase information on complex structures and systems. In medical imaging, X-rays have been used since the discovery by Röntgen in 1895 and has evolved tremendously in the last decades by the development of tomographic techniques [1]. The ultimate resolution in X-ray imaging is constantly pushed in many research fields, such as Life Sciences and Materials Sciences. By using X-rays, 2D and 3D imaging of nanometer sized objects can nowadays be performed [2]. X-ray absorption- or phase-contrast imaging becomes even more powerful if spectroscopic information is included in the experiments. The use of spectroscopy as a complementary contrast mode opens up the possibility to obtain, for each pixel in the image, element specific and chemically related information on the atomic and molecular species in the sample [3, 4].

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T6

RECENT SYNCHROTRON RADIATION SOURCES, AND THE MAX IV EXAMPLE

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Synchrotrons as tools for photon science have increased in importance, capacity and scientific output over the last decades. The quality and the intensity of the radiation is developing quickly with a focus on coherence, where we tend talk about *the diffraction limited storage rings*. We currently see a new generation of synchrotrons emerge with the MAX IV facility just coming on line. Immediately before we saw PETRA III in Hamburg and the NSLS II in Brookhaven begin operation. The MAX IV will soon be followed by the SIRIUS in Brazil and among others the ESRF upgrade using similar concepts. What is the key to these new sources? What are the physics and technology breakthroughs? In a parallel field the Free Electron Lasers are now being built in the X-ray region and become an alternative path to address the coherence, intensity and extremely short pulses. Will the FELs eventually replace storage rings?

This lecture will give a view on how to relate the accelerators to the generation of light. The basic concepts, components and physics of accelerators will be introduced to give an understanding on how to generate the necessary electron beams and connect this to the generation of light in undulators. The unique features to reach higher coherence and smaller beams in the new light sources such as MAX IV, PETRA III, Sirius and the ESRF upgrade will be discussed. The perspective of special features in Free Electron lasers will be analyzed and the complementarity with synchrotrons discussed.