MS28 O1

The Development of Instrumentation for Diffraction Experiments using Sub-100-Nanometer X-ray Beams

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Keywords: Diffraction; Synchrotron Radiation, Microcrystals

The employment of x-ray micro-beams for diffraction experiments with high spatial resolution went hand in hand with the development of third generation synchrotron radiation sources offering the high brilliance input beams which are indispensable for efficient focusing. Applications of beams in the micron range have already entered routine operation in the past few years [1]. Evidently there is a strong demand for even higher resolution and thus for smaller beams. Where the production of a 50 nm beam has been demonstrated earlier at ID13 [2] the development of adapted instrumentation in order to meet the extreme requirements imposed by the nanometer scale is still ongoing. There is a whole plethora of problems to be solved, with positional stability of sample, x-ray optics and beam (e.g. thermal drift, vibrations) being only the most obvious. We will discuss an integrated concept dealing with the above mentioned problems while leaving enough modularity for a large variety of nano-prefix experimental techniques, like nano scannig-diffraction, nano-crystallography, nano-SAXS, nano-GISAXS, and more. Examples of possible applications will be proposed covering a wide range from polymer science (e.g. biopolymers) to nano-beam protein crystallography [3].

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MS28 O2

Combing Laser Tweezer and Micro-diffraction: New Possibilities for *In Situ* Manipulation

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Keywords: X-ray microdiffraction, optical manipulation, liposomes

In this presentation we show how optical trapping can be used to fix and manipulate individual micron-sized samples and at the same time investigate their internal nanostructure by X-ray microdiffraction. Multilamellar liposomes in the form of highly diluted colloidal dispersion were trapped by light tweezers in single or multiple positions in the optical path of a 1 μm X-ray beam and analyzed by scanning microdiffraction in the plane perpendicular to the X-ray beam. The validity of this technique is demonstrated for clusters of about 30 multilamellar liposomes (3 x 6 μm large cluster). The signal to background ratio shows that single liposome measurements are feasible. Moreover, this technique not only allows investigating single sample entities, but also changing simultaneously by remote control microfluidics the environmental conditions (pH, salinity, chemical potential by other interacting molecules etc.). Single particle chemistry becomes feasible. Further, multiple traps of different samples enables to induce the interaction between them, once they are brought into contact by the optical tweezers. Examples, possibilities and potential of this new technique are presented and discussed.

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MS28 O3

Strain mapping by non destructive method : Laue microdiffraction

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Keywords: X-ray diffraction, strain-stress measurements, Laue method

Scanning X-ray microdiffraction [1] combines the use of high brilliance synchrotron sources with state-of-the-art achromatic X-ray focusing optics and large area detector technology. Using either white or monochromatic beams, it allows for orientation and strain/stress mapping of polycrystalline materials with large grain size (bulk materials) using white beam and small grain size using monochromatic beam (ion beam sputtered thin films) with submicron spatial resolution.
Evolution of strain fields in materials conducted by external loading conditions (tensile or compressive testing) can be studied by performing in situ testing. White microdiffraction can then be used to study metallurgical alloys with grain size greater than the beam spot (typically 1x1 micron).
Evolution of the stress field in shape memory alloys has been characterized using white beam Laue diffraction. It allows following the evolution of the strain/stress field in a grain, surrounded by other grains and subject to external loading. When the internal load reaches a critical value, the austenitic phase transforms to martensite in order to accommodate the stress field and reduce internal load in the primary phase. With the sub-micron resolution of the microdiffraction, we are able to follow the stress field evolution in a single grain and also between the martensite variants during the whole transformation of the grain due to the external loading.
Due to the experimental, Laue microdiffraction beamlines allows changing from white to monochromatic beam without position change on the sample. So it is possible to use both techniques to characterize samples with grain size lower than the beam spot, as thin film on substrate:
Delamination mechanisms of a thin metallic film (such as gold) adherent to a substrate can be analysed in situ by applying compressive stress to the substrate covered by the thin metallic film (500nm thick) during white beam and monochromatic microdiffraction scanning. White

beam diffraction is used to characterise the substrate which is a LiF single crystal. Monochromatic diffraction is used to analyse the thin film, the grain constituting the film having a nanometric size. With the large two dimensional detector available at the beamline, partial powder rings are collected. The thin film diffraction patterns are analysed using the classical powder diffraction technique and $\sin^2\theta$ method.

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MS28 O4

Recent developments in single crystal microdiffraction at the ESRF-ID13 beamline. D. Popov*, M. Burghammer and C. Riekel *ESRF, B.P.220, F-38043 Grenoble Cedex09, France.* E-mail: popov@esrf.fr

Keywords: microdiffraction, microgoniometer, microcrystals

The aim of this contribution is to report on the state of microcrystallography at the ESRF-ID13 undulator beamline. Single crystal experiments on a wide range of samples are usually performed using a microgoniometer with beam sizes down to 5 μm.[1] A new microgoniometer has been developed for single crystal diffraction with a 1*1 μm^2 beam.[2] The sample preparation is based on Kleindiek Nanotechnik "nanomanipulator", which is installed at an Olympus BX51W1 microscope. Small unit-cell microcrystals are usually glued to glass capillaries and mounted on the microgoniometer. Protein samples are usually cryofrozen in loops. The beam is focused by parabolic compound Be-refractive lenses [3] and collimated to 5, 10 or 30μm. The 1*1 μm^2 beam system is based on KB mirrors or Fresnel lenses focusing.[2] Mechanical stability is obtained by an air-bearing rotation stage with sub micron sphere of confusion. Centering of the sample on the goniometer is done semiautomatically by a Kleindiek Nanotechnik manipulater mounted on the rotation stage. This contribution will show several case studies performed with the microgoniometer (A-amylose) and the 1*1 μm^2 beam system (Xylanase II, a new metal organic framework compound).

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MS28 O5

Selective 3D imaging of diamond/C60 growth's pellet by X-Ray Micro-Diffraction Computed-Tomography Pierre Bleuet[a], Eleonore Welcomme[b], Eric Dooryhee[c], Philippe Walter[b], Jean-Louis Hodeau[c]. [a]*ID22, ESRF, Grenoble France.* [b]*Centre de Recherche et de Restauration des Musées de France, C2RMF, Paris, France.* [c]*Institut Neel – CNRS, Grenoble, France.*
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Keywords: microdiffraction, tomography, C60, diamond

In this presentation, we present the mapping of nanocrystalline heterogeneous materials by using X-ray diffraction computed tomography (XRD-CT) with high spatial resolution.
Computed tomography is usually based on the detection during the moving (x,z) and the rotation θ of the sample of a signal such as X-ray absorption, phase contrast, Compton diffusion or X-ray fluorescence. From this data, a spatial distribution reconstruction can be performed to visualize the electron density, the mass density distribution or the distribution of all chemical elements. We have adapted the reconstruction algorithm to perform such an analysis by using the X-ray diffraction signal. Diffraction profiles are collected in parallel projections from the sample with a 2D detector. After integration of the images, selective Bragg peaks or amorphous signals are used for the reconstruction of the different phases existing in heterogeneous samples. For this development, we used a method already developed to combine the information obtained by different types of tomography (integrated tomographic technique ITT) [1].
We demonstrate the high possibilities of X-ray diffraction computed tomography (XRD-CT) by mapping different nano-crystallized diamond-like phases of a heterogeneous pellet synthesized from C60 molecules under High Pressure. On such a transformation, if compressed non hydrostatically at room temperature, the fullerene cages collapse, producing polycrystalline cubic diamond and an sp^3 amorphous carbon phases [2]. Using a 2μm X-ray beam, the XRD-CT analysis discriminates the different phases and maps their distribution in the pellet formed in the diamond HP cell. The reliability of XRD-CT is excellent, as shown by the similar reconstruction built from the different reflections of cubic diamond or sp^3 amorphous phase. Furthermore, by selecting intense pixels in the corresponding sinograms, it is possible to extract the scattering pattern of each phase even for the minor ones (less than 1%).
Using XRD-CT analysis, we have demonstrated that even in presence of amorphous phases and reflection overlaps, it is possible to discriminate, to map and also to characterize different sp^3 carbon phases with similar density. We will also illustrate the efficiency of this method on other heterogeneous samples like mixed powders or archaeological ones.

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