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X-ray focusing technique is essential for probing microscopic samples at high pressures. Compound refractive lens (CRL) is one of the most versatile devices that are capable of focusing and collimating x-rays in the high-energy range of 20 – 60 keV [1, 2]. The CRL offers many advantages: good efficiency for focusing, compactness, robustness, and easy alignment (in-line focusing element). At high-pressure x-ray diffraction beamline BL10XU of SPring-8, the x-ray focusing optics consisting two different types of CRL devices have been installed in tandem. The first CRL (16 m focal distance) made from glassy carbon (GC), which is situated at a distance of 42 m from the source, has been used incidentally to collimate the x-ray beam. The aperture of this CRL is about 1 mm, whose size matches the beam size of the undulator. Because of difficulty in focusing the x-ray beam size down to 10 μ m through the first CRL, a second focusing CRL device was placed at 0.5 m upstream to the sample. The second CRL is sets of planar cross-lenses with a quasi-parabolic profile for focusing in two directions, and were fabricated from SU-8 polymer by deep x-ray lithography at the ANKA in Germany [3]. The placement of the SU-8 CRL after the GC-CRL produces an x-ray beam with a spot size of less than 7 μ m at 30 keV. The photon flux density at the focal point is approximately 10¹⁵ photons/sec/mm². This x-ray optics allow us to collect high-quality x-ray diffraction data on materials subjected to extreme pressures of up to 400 GPa, which exceeds the condition found at the Earth's center.

[1] A. Snigirev, et al., Nature 384 (1996),49-51.

[2] Y. Ohishi, et al., Nucl. Instrum. Methods A 467-468, (2001)962-965.

[3] V. Nazmov, et al., Proc. SPIE 5539 (2004)235-243.

Keywords: high-pressure X-ray diffraction, X-ray focusing optics, synchrotron radiation optics

P01.02.37

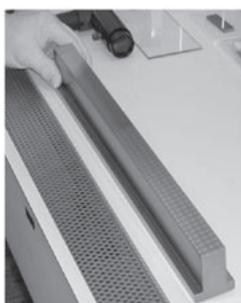
Acta Cryst. (2008). A64, C182

Total reflection and multilayer optics for synchrotrons and free-electron lasers

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We will be presenting selected aspects of simulation, preparation and characterization of total reflection and multilayer X-ray optics. The optimum coating is found by calculating the optical properties. Sophisticated improvements in manufacturing technology allow the precise realization of the specified parameters. Our capabilities for the production of multi stripe optics as well as for very long mirrors will be shown. Two applications demonstrate our possibilities: First of all we will be showing results of the development of carbon coatings as total reflection mirrors for Free-Electron Lasers. Over the past years, we have developed optimized optics for the XUV range up to 200 eV. The investigations have shown that carbon coatings offer high reflectivity >95%, high radiation stability and good uniformity both in thickness and roughness. Secondly, we will be presenting some results of the production of multi stripe optics for the



use at different energies. One example shows a three-stripe optics for a tomography beamline. A Ru/C multilayer was chosen for energies of 10-22 keV, a W/Si multilayer for energies of 22-45 keV and one stripe of the (111)-Si substrate remained uncoated.

Keywords: synchrotron optics, multilayer optics, total Reflection Optics

P01.02.38

Acta Cryst. (2008). A64, C182

Center of mass grain maps in 3D

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We would like to visualise the microstructure of crystalline materials. Diffraction based mapping techniques [1] offer a way to determine the position, orientation, size and strain state of multiple crystallites within a sample. Mixtures of different crystalline phases can also be treated, as each crystallite is indexed and fitted separately. Multiple crystallites can be indexed from a single dataset to give an orientation matrix for each one. Any small misalignment of each crystallite with respect to the diffractometer axis gives systematic shifts in the Bragg peak positions during rotation. The area detector geometry and axis direction can be fitted together with the positions of the crystallites using the ImageD11 software package [2]. The position, orientation and lattice parameters of each indexed crystallite are normally refined. Peak intensities give the volume of each grain that has been illuminated by the beam. We will show several examples of maps produced using high energy x-ray diffraction data collected on an area detector at beamline ID11 at the ESRF. Spatial resolutions for the center of mass grain position are typically somewhat better than the detector pixel size (50 micron) and cover an area as large as the beam size (up to 2 mm). Maps containing several hundred grains have been produced.

[1] Three dimensional X-ray diffraction microscopy, Mapping polycrystals and their dynamics. H F Poulsen (Springer, Berlin, 2004)

[2] <http://fable.wiki.sourceforge.net/imagid11>

Keywords: instrumentation and software, area detectors, crystallites

P01.07.39

Acta Cryst. (2008). A64, C182-183

Grazing-angle incidence hard X-ray nanoscope

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We consider in presented theoretical paper a possibility to overcome the limitations of the x-ray microscope and increase its resolution from tenth a micrometer to a nanometre or less. Proposed nanoscope would allow studies of the nanoscale structures. We foresee the

applications of a nanoscope in the set-up of hard X-ray optical data read-out device (e.g. see [1]). Other applications, for example, are envisaged in hard X-ray advanced nanolithography (chip fabrication) and in cell biology to view structural details of living cells in 3D and real time, at a previously unobtainable resolution. A set-up of grazing-angle incidence hard X-ray nanoscope (GIXN) is presented appropriate for the non-destructive high-resolution investigations of the various kinds of non-diffracting subsurface nanosize inclusions based on the grazing-angle incidence X-ray backscattering diffraction (GIXB) technique [2, 3], which takes place in the conditions of specular vacuum wave suppression phenomenon [4]. GIXN analyser is an asymmetrically cut single crystal, which is operating like an image magnifier. High-resolution X-ray diffractive optical lens (zone plate) and spatially resolving detector (CCD camera) are arranged like in classical schemes of the X-ray imaging microscopy.

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[2] Bezirganyan H.P., Bezirganyan P.H., *Phys. Stat. Sol. (a)*, 1988, 105, 345.

[3] Bezirganyan H.P., *Phys. Stat. Sol. (a)*, 1988, 109, 101.

[4] Bezirganyan H.P., Bezirganyan H.H. (Jr.), Bezirganyan S.E., Bezirganyan P.H. (Jr.), *Opt. Comm.*, 2004, 238/1-3, 13.

Keywords: X-ray imaging, grazing X-ray diffraction, X-ray back reflection

P01.07.40

Acta Cryst. (2008). A64, C183

Application of synchrotron X-ray micro tomographic microscopy at low temperature

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We report the application of synchrotron X-ray cryo-micro tomographic microscopy (SRXCTM) provided by the 'TOMCAT' beam line at the Swiss Light Source (SLS) for studying the substances such as ice and gas hydrate at low temperatures down to 150 K. Description of the precarious handling of the samples within their thermodynamic stabilities, polyamide sample holder, experimental setup, cryo-stage coupled with cooling setup, and data acquisitions were addressed. The monochromatized X-ray beam was modified by a slit system to a profile as small as of 1.4 mm² to confine the irradiation to the region of interest (ROI). The beam energy was optimized to enhance the contrast among the constituents of specimens. The X-ray beams were converted into visible light with a thin scintillator screen. Further magnifications of the projection images were done by microscope optics, which were digitalized using a high resolution CCD camera. Post processing of the reconstructed raw data was carried out using 'MATLAB'. The 3D visualization of the post processed datasets was performed with the software 'Amira' which facilitated to separate the cylindrical ROI from the raw dataset to eliminate the non reconstructed regions. Out of the SRXCTM dataset, both of ice and gas hydrate, parameters like porosity, surface area and volumes were calculated for the whole dataset as well as a single object. The imagery demonstrated the well suited applicability of SRXCTM revealing some salient microscopic features preserved in marine hydrates and atmospheric ice.

Keywords: synchrotron X-ray tomography, ice, gas hydrate

P01.07.41

Acta Cryst. (2008). A64, C183

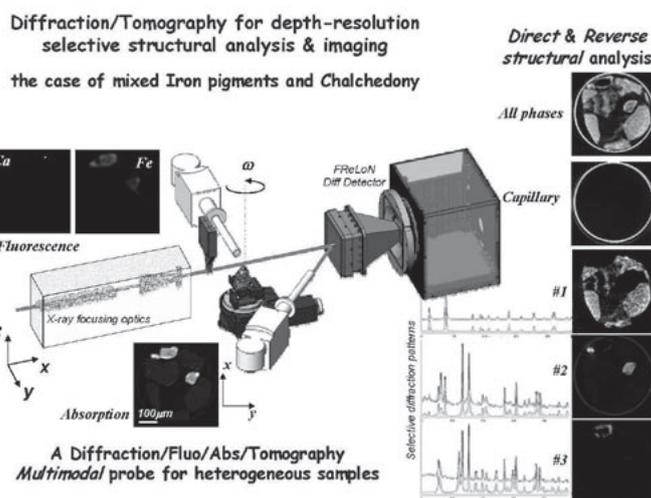
Imaging and structural analysis of heterogeneous diluted materials by diffraction tomography

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We evidence the potential of coupling pencil beam tomography with X-ray diffraction to examine unidentified phases in heterogeneous materials and to overpass the relatively low detection limit of X-ray diffraction. The demonstration is performed on a heterogeneous powder containing chalcidony and iron pigments (see figure). Furthermore we will also present the 3D phase selective reconstruction of a high-pressure pellet containing several carbon phases. The present method allows a non-invasive structural refinement with a weight sensitivity of one part per thousand. It allows the extraction of the scattering patterns of the amorphous and crystalline compounds with similar atomic densities and compositions. Furthermore, such a diffraction-tomography experiment can be carried out simultaneously with X-ray fluorescence, Compton, and absorption tomographies, allowing a multi-modal analysis of prime importance in materials science, chemistry, geology, environmental science, medical science, paleontology and cultural heritage [1].

[1] P. Bleuet, E. Welcomme, E. Dooryh e, J. Susini, J-L. Hodeau, P. Walter, *NatureMaterials* (2008) in press, URL: <http://dx.doi.org/>, DOI: 10.1038/nmat2168



Keywords: diffraction methods, tomography, diffraction imaging of heterogeneous specimens

P01.07.42

Acta Cryst. (2008). A64, C183-184

Realtime imaging in X-ray fluorescence and X-ray diffraction

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