ing, and to ventilate with various gases (He) to create non-harming atmosphere (without oxygen).

Detectors.

The advanced scattering techniques and complicated investigated structures require a spectrum of x-ray detectors. For simple measurements and scans a NaI scintillation detector is required. Reciprocal space mapping of diffraction from complex structures requires a proportional one-dimensional position sensitive detector which allows fast collection of data in angular space. This detector works usually with Ar atmosphere. Nowadays, the detector technology goes towards solid-state pixel detectors with high dynamical ranges, so we expect their availability after year 2010. In-situ techniques at extreme temperatures and at low pressure require even faster collection of data and thus a 2D CCD position sensitive detector with pixel resolution around 50 μm is necessary in order to collect the intensity at very fast times.For energy-sensitive experiments an energy-dispersive PIN diode is required.

4. User community

User community for this beamline covers users from academia and research institutions, universities or industry growing, preparing hard and soft condensed matter samples (solid state physics, supraconductivity, thin films, coatings and membranes), as well as analytical laboratories, including the Czech community “Nanoscience for society”. Among others, this will include groups from Masaryk University (Brno), Charles University (Prague), Technical university (Brno), Institute of Physics (Prague), producers of samples and nanostructures, including industry and private companies, as well as biophysics and biotechnology institutes for studying liquids, membranes, soft matter. Current experience of these Czech groups originates from experiments at ESRF, Hasylab, ANKA, LURE, BESSY, SLS.

From the central European community, the interested user community currently covers users from Slovakia and Austria (scientific institutions and universities).

A FLEXIBLE CONFIGURATION FOR COHERENT IMAGING AND TOMOGRAPHY BEAMLINE AT CESLAB

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Keywords:

tomography, phase contrast, imaging

Abstract

The state-of-the-art instrumentation and methods at the CESLAB’s Coherent Imaging and Tomography (CIT) Beamline will enable to perform research at the forefront of non-destructive materials probing. Both, academic and industry funded research will benefit from this unique imaging facility in Central Europe. The availability of several imaging techniques will trigger new groups to apply for beamtime to study materials at a different scale than they used before. We aim to build collaborative relationships across complementary disciplines and techniques. In recent years micro-tomography using synchrotron radiation became a valuable tool for the non-destructive three-dimensional investigation of specimens in fields such as medicine, biology and materials science. At CESLAB absorption- and phase-contrast techniques will be developed and applied at photon energies in the range of 200 to 30 000 eV. Due to the finite pixel size of the X-ray detector and the divergence of the source the spatial resolution of a tomogram in parallel beam geometry will be limited to about 1 μm. For resolving smaller structures in the 100 nm regime new techniques have to be developed. Apart of the standard parallel beam setup, it is foreseen to perform cone-beam tomography by creating a nanometer divergent X-ray source using a Kirkpatrick-Baez (KB) multilayer arrangement and magnifying the sample onto a two-dimensional X-ray detector.

Introduction

At CESLAB imaging methods will be performed at the two imaging beamlines: CIT and SXRM. The first end-station of CIT (CITA) is aimed to perform mostly routine experiments such as micrometer resolution tomography and laminography. The high brilliance of the third generation source and the development of faster detectors will also enable rapid time resolved tomographic studies.

The long, CTIB beamline will be optimized to perform nanoscale zoom tomography with divergent beam, coherent diffraction imaging (CDI), spectromicroscopy using both fluorescence imaging and scanning of the primary X-ray probe energy for XANES imaging with a possible extension to perform hard X-ray full-field microscopy. It will however be possible to house experiments requiring large fields of view and good coherence properties. The photon energies will range from 7 to 30 keV and the required beam size at the sample is several mm. The key strength of hard X-ray full-field microscopy is the large penetration depth of hard X-rays into matter, which allows one to image the interior of opaque objects. Combined with tomographic techniques, the three-dimensional inner structure of an object can be reconstructed without the need for difficult and destructive sample preparation. Projection microscopy and microtomography are now routinely available at synchrotron radiation sources. The resolution of these techniques is limited by that of the detector to 1 m or slightly less. X-ray images and tomograms at higher spatial resolution can be obtained by X-ray optical magnification, for example, by using Zone Plate X-ray lenses as a magnifying optics. Combining magnifying X-ray imaging with tomography allows one to reconstruct the three-dimensional structure of an object, such as a microprocessor chip, with resolution well below 1 m. In x-ray scanning microscopy, the sample is scanned through a small-diameter beam. The great advantage of scanning microscopy is that x-ray analytical techniques such as fluorescence analysis, diffraction, and absorption spectroscopy can be used as contrast mechanisms in the microscope. In combination with tomography, fluorescence analysis makes it possible to reconstruct the distribution of different chemical elements inside an object (fluorescence microtomography), while combining absorption spectroscopy with tomography yields the distribution of different oxidation states of atomic species. To achieve a smooth beam profile and preserve coherence, the beamline will have as few optical elements as possible. The experimental hutch will allow for a variable sample detector distance from almost 0 to 10 m. For cone-beam geometry, optical elements will be added to the beamline to achieve a divergent X-ray source with focal spot size of nm range.

Beamline design

A simplified general rule says that in order to achieve high contrast and high spatial resolution in phase contrast imaging, the longer the beamline is the better and the smaller the source is is better.

At the bending magnet source the collimation in horizontal direction is not preserved, i.e. is larger than the natural opening angle in the vertical direction (perpendicular to the plane of the orbit of electrons in the storage ring). However, in the straight sections, by imposing an alternating (in polarity and in space) magnetic field in the direction perpendicular to the plane of motion the collimation of emerging radiation can be preserved in both directions enhancing thus the flux/brilliance of the X-ray beam. Producing small source in both directions is crucial for 3D coherent imaging techniques. Initiated by the limited availability of straight sections at synchrotron sources, phase contrast imaging at a bending magnet source has also been demonstrated [1]. The choice to use straight section insertion devices as the source for the Coherent imaging beamline is still a privilege and a solid ground for cutting-edge 3D Coherent imaging.

Absorption based imaging emphasizes on the imaginary component of the complex X-ray refractive index ($n = 1–$ –i). The real component is primarily concerned when dealing with X-ray phase-contrast imaging (PCI). Many examples of PCI will involve a combination of phase and absorption contrast to varying degrees and while both generally decrease with increasing X-ray energy, it is important to note that varies as ^{2} whereas varies as ^{4}, in the absence of any elemental absorption edges. Hence the effects of phase contrast become progressively more dominant, relative to absorption-contrast effects, at shorter X-ray wavelengths.

The second argument for using hard X-rays for tomographic imaging is that a large number of industrial applications require tomographic imaging with spatial resolution of several micrometers and a large field of view. In order to use the full dynamic range of the CCD based X-ray detector and meanwhile keep the exposure time short, the larger and more absorbing samples imply the use of hard X-rays.

First (short) experimental end-station CITA

Located inside the experimental hall (storage ring building) this end-station would house mainly tomographic, laminographic eventually topotomographic experiments. The priority here is to keep the procedures for users as simple as possible and routinely perform experiments in absorption and in phase contrast mode. Both academic and industry related research should determine the final design of the CITA beamline. This endstation will be equipped with a furnace/calorimeter and traction device in order to study material deformation, fatigue and fracture of composites as well as annealing processes. The future upgrade of this beamline could be to mount and additional beam expansion optics in order to be able to image larger samples (>20 mm).

The long experimental end station CITB

This end-station will be optimized for phase contrast radiography and tomography with large field of view and both in parallel and divergent beam geometry. As focusing optics for the lower energies (7–12 keV) a condenser zone plate could be the right choice and for higher energies it is required to use Kirckpatric-Baez mirror system. Both would produce submicrometric 2D focal spot. The multilayer coated KB mirrors would be bent with a 3 point bending system, according to the requirements on energy and the specifications of the focal spot. The focal distances will be relatively large in order to minimize wavefront curvature (important especially for CDI). The whole system is preferably in the vacuum, to ensure thermal and mechanical stability. The sample stage would allow to perform CDI and the combination of Fluorescence imaging with tomography. The X-ray detector for CDI must be positioned on a long translation stage and the end-station building must allow to go as far as several meters away from the focal plane in the beam propagation direction and at the diffraction angle. The sample stage for the divergent beam geometry would allow to place the sample in the focus, but also a few cm out of focal plane. The divergent beam passing through the sample would be recorded with a CCD placed on the parallel beam tomographic stage. The CITB is optimized for achieving the best possible focus, thus in the nanometer range. Therefore the requirements on thermal and vibrational stability are very strict. Precise temperature control is needed. The future upgrade of this beamline can consist of adding an objective zone plate optics for full-field microscopy with hard X-rays. This would be a beamline for complementary research to the Soft X-ray microscopy beamline which operates up to 3 keV.

Experimental approaches

Several different regimes of X-ray imaging can be distinguished in function of relative detector-to-sample distance, and the overall geometry of the beam and sample stage. Absorption radiography, edge detection, holography and coherent diffraction imaging are the three basic imaging regions. Absorption imaging being the only regime not imposing any requirements on the transverse coherence length of the incident X-ray beam, it is routinely performed at several synchrotron sources and with laboratory sources as well. It is relatively easy to implement absorption based tomography on an imaging beamline, therefore we will focus here more on the planning of advanced imaging techniques.

Microtomography in parallel beam geometry (towards fast tomography)

The reconstruction algorithms based on inverse Radon transform for parallel beam 3D tomographic imaging are well established at synchrotron facilities. The spatial resolution is limited by the specifications of the X-ray detector (pixel size, point spread function) to about 1 m. Routinely used detectors have a CCD with 2048 × 2048 or 1024 × 1024 pixels, which sets the field of view that can be from 2 mm up to a few cm in the case of lower resolution imaging.

Perhaps the most spectacular difference in imaging performed with a synchrotron beam compared to laboratory sources is the interference effects resulting from the spatial coherence properties of the X-ray radiation. Coherent radiation comes from a point source, which is not the case in practice, since the dimension of the source is not infinitely small. We say therefore that the radiation is partially coherent. Applications like for instance coherent scattering or coherent diffraction imaging require to know precisely the transverse coherence properties of the beam in order to limit the beam size impinging on the object to a coherent part.

Fast-tomography

Temporal resolution is often as important as spatial resolution. Since in the parallel beam setup there is no need for additional optics in the beam path, a 3rd generation synchrotron has the capacity to provide enough flux to perform fast tomographic imaging. On these sources the speed of tomography is becoming limited by the rotation stage rather than photons flux or detector speed. This is valid for coarse resolutions, because for smaller effective pixel sizes the scintillator must be thin and has hence low efficiency. Fast-radiography is relatively easy to perform and is mostly dependent on the detector readout (or frame transfer) capacity and the flux at the given energy. For tomography it is a slightly more complicated case, here the optimization of the whole imaging system is very important, because any software or hardware delay time of a few milliseconds would sum up at the end of the tomographic acquisition.

Cone-beam projection imaging (zoom-tomography)

The resolution limit imposed by detector technology can be overcome if the beam geometry is changed to divergent and geometrical magnification is used. The nearly parallel beam is focused to a small spot (its dimensions determine the final resolution), the sample is placed in te beam small distance from the focal plane and the detector further downstream the divergent beam. The magnification is the ratio between the sample to focal plane and sample to detector distance. In this configuration the requirements on the detector pixel size are very loose, because the magnification can be of the order of 100. Radiography can be performed with spatial resolutions directly proportional to the magnification, whereas the resolution in tomographic reconstructions is dependent on several additional factors, such as the quality of the focusing optical elements and relative stability of the beam and the sample. The latter factors impose strong requirements on the laboratory expected to house nanoscale zoom-tomography experiments. For hard X-rays (20 keV) 290 nm spatial resolution has been achieved in 3D tomographic reconstructions. Going for submicrometer resolutions various image degrading phenomena can be present in the imaging system and it was shown [1] that in the case of the Kirkpatrick-Baez (KB) mirrors based focusing it is needed a correction related to the figure errors of the reflecting surfaces. They result in deviations with respect to a spherical wave of the sample illumination. An another interesting and very useful property of phase contrast imaging was pointed out by the same group, it is that from samples exceeding the field of view (e.g. by a factor of 10) quantitative 3D reconstructions in phase contrast mode are obtained. This is called local tomography mode. This kind of large are more relevant from the application point of view (for instance for in-situ deformation experiments and other dynamic studies) and they will be of particular interest in the materials science community.

Laminography

Synchrotron-radiation computed laminography (SRCL) was developed and recently successfully implemented for high-resolution non-destructive 3D imaging of regions of interest (ROIs) in laterally extended specimens and devices (such as sensors, flip-chip devices and other microsystems). This experiment has been performed with parallel beam at the ID19 beamline at ESRF. Further development of reconstruction techniques and instrumentation are expected and through the CIT beamline the CESLAB scientists will have a chance to contribute significantly to this newly emerging field of imaging.

X-ray fluorescence spectroscopy and mapping

The setup which would be used for the zoom tomography as described in can be readily modified for 2D mapping of samples and recording of fluorescence spectra at each point with an energy dispersive detector. The modification consists in placing the sample in the focal plane and the energy dispersive detector at an appropriate angle to record the fluorescence photons in the selected spectral range. The CCD camera behind the sample can still be used to combine radiographic images of the sample with the 2D fluorescence maps.

Coherent diffraction imaging

Placing the detector at the far-field region of the image plane and the sample illuminated with the focused or parallel X-rays will result in a speckle pattern. The problem of reconstructing the structure of an isolated “compact” non-periodic object from its coherent x-ray diffraction pattern alone has attracted increasing attention in recent years. Instead of lenses, algorithms are used that transform back and forth between real and reciprocal space, applying appropriate constraints in each domain. Thereby, the resolution is — at least in principle — diffraction limited only.

Topotomography

The acquisition and reconstruction from a polycrystaline sample can be performed from projection images with the detector positioned either in the diffracted-beam or in the direct-beam position. In the first case, the projection data consist of a series of integrated, monochromatic beam X-ray diffraction topographs of the grain under investigation. In the second case, the corresponding diffraction contrast in the transmitted beam may be interpreted as an additional contribution to the X-ray attenuation coefficient of the material.

Applications

Material deformation, fatigue and fracture of composites, dynamic systems such as liquid foams

For structural applications of Aluminum alloys the studies of the microstructure and its modification due to heat treatment need to be conducted on materials in the bulk form. X-ray tomography has proved to be a very suitable technique to do this for Al based alloys. Recent improvements in X-ray tomographic techniques (spatial and temporal resolution) allow to follow the structural changes of composite materials during annealing procedures [2]. For the general purpose of understanding the process of the coalescence of coarse particles at elevated temperatures experiments at submicrometric spatial resolution must be carried out.

There is a very wide and quickly growing range of applications that require fast radio and tomography in phase contrast mode with large field of view.

Relatively little is known about the dynamical properties of liquid foams, and still they are all around us in everyday life. Several theoretical predictions exists, but only recently with the emerging fast tomography techniques could some predictions be validated and some questions answered. Dry liquid foams are made of thin liquid films separating polyhedral gas bubbles. While aging, they coarsen because bubbles with a small number of faces lose their gas into bubbles with a large number of faces. Since they strongly deviate light, they are notoriously difficult to image. Using high speed 3D X ray tomography, it was recently achieved to follow the complete evolution in 3D and it was shown that it reaches a self-similar growth regime, in which the scale coarsens but the statistical distributions of topological and dimensionless geometrical quantities remains invariant in time [3]. These results also apply to grains in crystals, concentrated emulsions, and more generally in diphasic system where a continuous matrix occupies a much smaller volume than the dispersed phase. For extended structured abstracts and full papers, the text should be organized in sections.

Trace element detection with sub-micrometer spatial resolution

Spatial distribution and concentration of trace elements in tissues are important, as they are involved in some pathological conditions and in many biological functions of living organisms like metabolism and nutrition.

Microfocus hard X-rays proved to be a suitable probe for this applications [1]. The the intracellular distribution of pharmacological doses of drugs, cellular and subcellular distribution of biologically relevant elements, such as phosphorus (ATP, DNA), zinc (transcription factors), calcium (second messenger proteins) and iron is a scientific question of strongly increasing importance in biomedical and life sciences research. The X-ray beam of selected energy impinging on the sample excites secondary emission from it. The radiation emerging from the sample includes the characteristic X-ray lines of major and trace elements present in the sample. The emitted X-rays are detected using an energy dispersive detector. The intensity of the fluorescence spectra depends on the concentration of the studied elements but also on the intensity of the incoming beam.

Archeology, paleontology, arts

Before studying important specimens, fossil owners need to be guaranteed that the investigation technique is totally non-destructive. X-ray imaging techniques are therefore well adapted because they are non-invasive. Laboratory tomographs, not necessarily devoted to medical applications, have evolved over the last few years and can reach high spatial resolutions. They can reveal very small details however, despite the high quality of the data that can be obtained by some of these machines, investigation of numerous fossils remains difficult. Limitations arise from the polychromatic X-ray source spectrum of these machines and from the intrinsic nature of the fossils (highly mineralised samples, which often exhibit very low absorption contrast).

References

1. R. Mokso, P. Cloetens, E. Maire, W. Ludwig, J-Y Buffiere, *Appl. Phys. Lett.*, **90**(14), (2007), 144104.
2. E. Maire, J-Y Buffiere, R. Mokso, P. Cloetens, W. Ludwig. *Mat. Sci. Forum.*, **519**, (2006), 1367–1372.
3. R. Mokso, J. Lambert, P. Cloetens, F. Graner, *Nature.* In preparation.
4. D. Balzar & N. C. Popa, in *Diffraction Analysis of the Microstructure of Materials*, edited by E.J. Mittemeijer & P. Scardi (Berlin: Springer), 2004, pp. 125–145.