DIFFRACTION BEAMLINE AT CESLAB

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1. Introduction

Elastic X-ray scattering techniques allow to study structural properties of crystalline and non-crystalline hard and soft condensed matter. These methods cover characterization of crystalline structure with its defects and strain distribution as well as bulk, surface and interface morphology in condensed matter samples. Substrates, thin films, multilayers, laterally structured samples and nanostructures, as well as liquids can be included in these investigated materials. X-ray diffraction method can study the crystal structure perfectness, such as strain and stress in crystalline samples by recording the diffracted intensity around Bragg peaks. Reflectometry and small-angle scattering can investigate morphology of crystalline as well as amorphous samples by recording intensity around the origin of the reciprocal space. In a similar way, crystalline as well as atomic structure of (organic) layers on surfaces of liquids, liquid-liquid and solid-liquid interfaces can be characterized. The proposed diffraction beamline at Central European Synchrotron Laboratory (CESLAB) will provide experimental environments for these studies. The beamline should satisfy needs of the user community such as institutions and laboratories for growth and characterization of above-mentioned broad portfolio of samples.

The proposed diffraction beamline at CESLAB will be devoted to various methods of elastic X-ray scattering. Three diffractometers are proposed: a low-load precise diffractometer for all scattering geometries and two heavy-load ones. One of the heavy-load diffractometers will be dedicated to characterization of hard condensed matter samples at extreme conditions and the second one to characterization of soft condensed matter such as surfaces of liquids, solid-liquid and liquid-liquid interfaces. X-ray scattering techniques will be possible to perform at ambient conditions as well as in-situ or extreme conditions such as at high temperatures, high vacuum etc. The former heavy-load 6-circle goniometer should have high precision step movement, the goniometer stage will optionally hold a high temperature furnace with turbo-molecular pump, small growth chambers or other goniometer accessories. The latter heavy-duty goniometer will be equipped with a Langmuir trough with a possibility to change to another liquid sample cell. A very good anti-vibration system under the liquid sample environment, as well as possibility to rotate and translate the whole goniometer with respect to the primary beam in the horizontal and vertical plane are indispensable for studies on liquid samples. In order to detect also the chemical composition during in-situ studies, spectroscopic measurements at various energies will be also available. The beam in the hutch has to be monochromatic with tunable energies in the range 5–30 keV with high monochromaticity. All the various experimental techniques and the large spectrum of samples require various x-ray detectors such as a point, linear and area detector including an energy sensitive detector. The scattering measurements on liquid samples necessitates a possibility to change the incident beam direction in order to vary the incidence beam.

2. Materials

X-ray diffraction can be applied for study of bulk wafers (silicon, GaAs), thin films, multilayers and superlattices (1D, 2D and 3D). Nowadays, this is a standard technique which is routine utilized in laboratories for crystalline sample characterization. Synchrotron radiation is required for investigation of small objects with low scattering contrast, nanostructures, and for fast measurements or in-situ observation of growth and for specific energy selection. For example self-organized structures, quantum wires and quantum dots occupy usually small volume of the material and thus contribute to the scattering signal negligibly. In many experiments the scattering area of the sample can be smaller than 0.1× 0.1 mm and thus a very brilliant beam with possibility of focusing is necessary. These very small objects such as quantum dots, wires or just very thin layers and heterostructures are often grown on various novel pseudosubstrates.

A modern material science requires combination of different materials with different properties and various lattice parameters. Nowadays, a combination of silicon, germanium, GaAs or its oxides grown intentionally on one substrate and thus one chip is commonly used. For example silicon on insulator or silicon on nothing provides a new challenge in production of fast electronic devices. GaAs, GaN, InAs, and other III-V or II-VI compounds are currently combined in semiconductor devices, lasers and other optoelectronic devices. Also a Si/GeSi heterostructures find a wide application in CMOS (complementary metal-oxide semiconductor) technology, bipolar transistors and other fast operation devices. Such combination of materials, especially in nanostructures, induces a strain or even defects in the crystalline structure, which can be studied by X-ray diffraction or by grazing incidence diffraction. Characterisations of this type give crucial feed-back on the device quality and hints for further improvement of the production techniques.

Development of new devices finds its base not only in typical hard condensed matter semiconductors, but also in bio-materials and soft condense matter where structural studies of thin membranes, layers and their structural arrangement is required. Investigation of strain distribution or interface morphology in these materials at nanometre scales is necessary for design and development of new devices.

Studies on liquid-liquid and solid-liquid interfaces can bring new knowledge on the structure of biological membranes, and on processes at the membranes. Additionally, X-ray studies on 2D-protein crystals adsorbed to lipid monolayers open new path-way to determination of the structure of wide range of proteins which cannot be easily crystallized in 3D-crystals and thus studied by the well-established methods of X-ray crystallography. Such a research could have a high impact in pharmaceutical research and industry.

3. Beamline proposal

Diffraction beamline is a standard beamline which needs to be available at all synchrotrons. The diffraction beamline at CESLAB will be proposed for investigation of novel materials and structures using soft and hard x-ray scattering techniques with beam energies in the range 5–30 keV. The experimental techniques performed at the beamline will be: high-resolution x-ray diffraction (HRXRD), grazing incidence diffraction (GID), x-ray reflectometry (XRR), grazing-incidence X-ray scattering (GISAXS), anomalous scattering, EXAFS spectroscopy diffraction and small angle scattering on liquids with free surfaces. These experimental methods will be realized on various ambient conditions such as high temperatures up to 1500 °C, low pressure up to $2 \bullet 10^{-9}$ mbar or at low temperatures.

The scientific case thus leads to a requirement of one beamline, with three hutches equipped with a goniometer. For hard matter, one low-load goniometer for all of the methods, the other for “heavy-load” to be used with a chamber for in-situ crystal and nanostructure growth. The independent goniometers will facilitate preparation of a new experiment during a measurement on the other goniometer. The third hutch will be devoted to experiments with soft matter and liquid samples. It will have an anti-vibrational and movable goniometer (on air-pads), and a Langmuir trough.

The beamline will operate with horizontally polarized beam in the energy range from 5 to 30 keV with resolution 1 eV. Considering the choice of the insertion device, an undulator has higher intensity but only for some energies to be tuned. Undulators are normally tuned to set a certain energy, and higher harmonics can be utilized. We could consider two undulators, one optimized for lower energy, one for higher.

The design of proposed beamline is motivated by the other diffraction beamlines in the world. All the devices in all hutches will be controlled by a scriptable program like `spec`, which is currently a standard in this field.

3.1.Optics hutch proposal

The above-mentioned experimental techniques require certain parameters of the optics and the beam. Suggested beam parameters: maximum size 20×7 mm^2 (w×h) – non-focused, 0.2×0.2 mm^2 (w×h) – focused. Beam divergence < 50×50 μrad^2. The optics of the beamline will involve collimating Si bent mirror, coated on part by stripes of layers from heavier material in order to switch between low and high-energy ranges and cut high harmonics energies. Si or Ge double crystal monochromator and secondary bent mirror in order to focus beam will be positioned next. The slits before the 1st mirror and after the 2nd mirror will allow to limit the beam at an appropriate size. All the optical elements will be situated in vacuum and cooled. Automatic absorbers for measurement will help to cover high dynamical range of intensities. The particular system how the beam will be distributed between the hutches is to be defined.

For liquid/liquid interfaces the critical angle of the interface is in ranges of tens of mdeg. Incidence angle smaller than critical is required to be sensitive on the properties of the interface. Small incidence angle implicate large foot-print of the beam. On the other hand, one often faces curvature of liquid/liquid interface. To reduce undesirable effect of the surface curvature on the scattered signal reduction of the beam vertical size to 3 μm at the sample position is desirable. Thus, focusing a mirror with tunable curvature is suggested after crystal monochromators. Additional focusing X-ray optics such compound refractive lenses will be an advantage.

3.2.Experimental hutch proposal

Goniometer 1 (low load).
The scattering geometry is conditioned by 6-circle diffractometer allowing a low load sample stage with precise sample positioning. Angular accuracy of goniometer will be <0.001° (step 0.0001°) and transition accuracy in z: 1 μm and in x-y: 10 μm. Angular movements will be possible in vertical and horizontal scattering plane and the detector arm should hold stable heavy 2D detector and analyzer crystal for higher resolution in reciprocal space. The sample stage will be optionally equipped with Be dome furnace allowing to measure at all available scattering geometries at high temperatures in the high vacuum. Maximum sample size in the furnace will be 20×20 mm^2.

Goniometer 2 (heavy load).
This setup will be similar to goniometer 1, but it will be specialized for in-situ diffraction. The goniometer should allow a high load of sample stage, like growth chamber, annealing furnace, or low temperature cryostat, with x-y-z translation stage. The setup will be available for a broad range of materials according to the needs of the user community.

Goniometer 3 (heavy load for soft matter).
Specialized for diffraction on liquid surfaces and interfaces. The same parameters as goniometer 2 + whole goniometer movable to follow the beam deflected by the deflection crystal. An antivibration system on top of the horizontal goniometer stage. Gases supply to allow appropriate atmosphere for samples. Temperature control for the sample chamber – Langmuir trough with a barrier for control of the surface pressure in the film. Possibility to ventilate dangerous gasses – a fume-hood with separate circulation – not to distribute ventilated air to the surround-

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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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