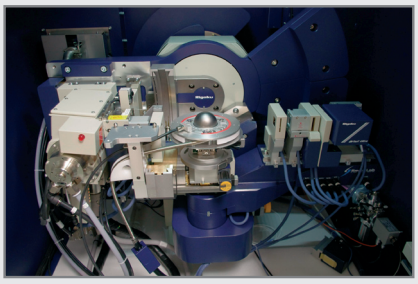
X-ray diffractometer for thin films and nano-structures characterization

Instrument description

Rigaku Smartlab 9kW is a novel high-resolution X-ray diffractometer dedicated to characterization of thin films and nano-structures. Due to its modularity, the instrument allows for large variety of X-ray diffraction and scattering techniques. The non-destructive analytical techniques reveal information about crystal structure, chemical composition, and physical properties of materials, thin films, and nano-structures.

For all techniques, the primary X-ray beam impinges on the sample surface or transmits the sample for the transmission geometry and the scattered intensity is measured as function of the incidence, in-plane exit and out-of-plane exit angles. The sample properties are then deduced from one- or two-dimensional scattering patterns based on position of interference or diffraction peaks or detailed simulations of the scattering pattern. Generally, X-ray diffraction and scattering are **sensitive to electron density fluctuations in the sample. Available X-ray techniques probe sample features on nanoscopic length scales (approx. 0.3 nm to 300 nm). The information is, however, averaged over much larger sample volume ranging from 10^{-5} mm³ to 1 mm³.**



Instrument: Rigaku Smartlab 9kW

Universal x-ray diffractometer with various high resolution, low resolution and polycapillary optics, equipped with a high temperature furnace for in-situ measurements.

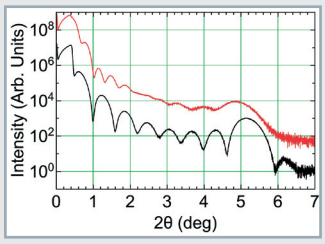
Features:

- High intensity Cu rotating anode X-ray tube (8 keV photon energy) to enhance signal from very thin layers
- Exchangeable X-ray optics for parallel, focussed or intense divergent beam allows for instrument resolution tuning
- Vertical θ–θ goniometer with an in-plane detector arm
- Horizontal sample position can be used for most of the experimental techniques without gluing the sample
- Detectors: scintillation detector and solid state linear detector D/teX Ultra
- Control software allowing for measurement automation and analysis software

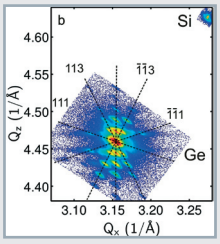
Available techniques

Technique	Typical samples	Resulting information
X-ray reflectivity and coplanar diffuse scattering	Thin films and multi-layers of inorganic or organic materials	Thickness of layers (range 1 to hundreds of nm), surface and interface roughness, and roughness lateral correlations
Coplanar X-ray diffraction (available at various resolutions)	Epitaxial hetero-structures and nano-structures	Lattice parameters, lattice strain, chemical composition, inter-layer diffusion
Wide angle diffraction	Polycrystalline thin films	Lattice parameters and strain
Pole figures measurements	Polycrystalline samples - both, bulk or thin films	Distribution of preferential crystallographic orientation (texture)
Grazing incidence X-ray diffraction (GIXRD)	Thin crystalline films	In-plane lattice parameters and preferential crystallites orientation in thin films. Depth resolved measurements
Small angle X-ray scattering and grazing incidence small angle scattering – in 1D mode	Nano-particles (in solution or thin films) and nano-porous thin films	Particles and pores size and size distribution, their mutual distance; feature size up to >≈ 100 nm
Scanning micro-diffraction	Laterally inhomogeneous samples	Information as for techniques above with lateral resolution down to 0.2 mm
X-ray diffraction and scattering during annealing	Samples for which thermally induced processes are relevant	Information as above at elevated temperatures up to 1100 °C; Environment: vacuum or air

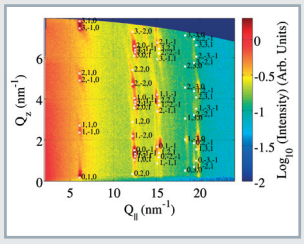
Application



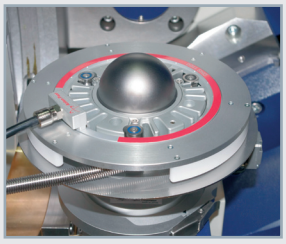
X-ray specular reflectivity on a thin film of an organic semiconductor and a similar layer with a metal electrode above.



HRXRD Reciprocal space map on facetted Si/SiGe heterostructures.



GIXRD reciprocal space map on a thin film of an organic semiconductor.

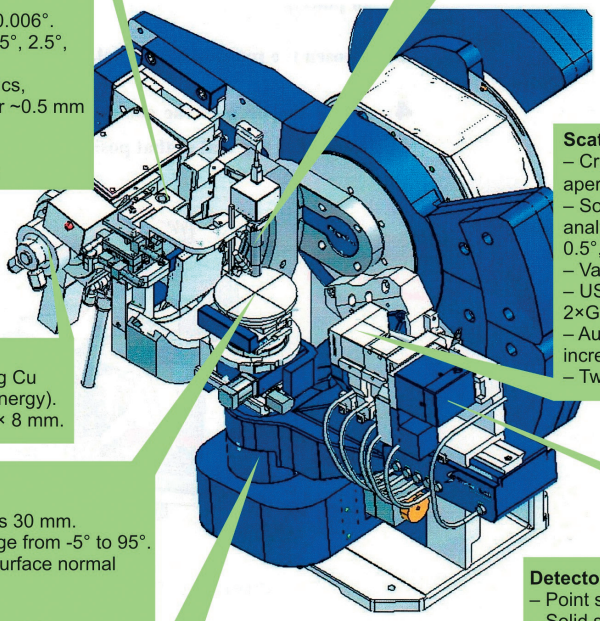


High temperature furnace for in situ experiments up to 1100 °C.

Technical Specification

Incident beam optics options
– Multilayer parabolic mirror, divergence Δθ~0.027°.
– Crystal monochromator 2×Ge(220), divergence Δθ~0.011°.
– "Bartels" monochromator 4×Ge(220),divergence Δθ~0.006°.
– Soller slits with apertures 5°, 2.5°, 0.5°, and 0.25°.
– Polycapillary focusing optics, focused beam size diameter ~0.5 mm at sample position.
– Computer controlled slit.
– Bragg-Brentano posibility.

Camera
– observing camera for sample positioning with magnification from 0.6x to 4x.



Scattered beam optics options
– Crystal analyzer 2×Ge(220), aperture Δ2θ~0.011°.
– Soller slits and/or parallel plate analyzers with apertures 5°, 2.5°, 0.5°, 0.25° and 0.112°.
– Vacuum path for SAXS.
– USAXS crystal analyzer 2×Ge(220).
– Automatic absorption filters to increase dynamical range.
– Two computer controlled slits.

X-ray source
9kW X-ray tube with rotating Cu anode (8.048 keV photon energy). Effective spot size 0.4 mm × 8 mm.

Sample environment
– Maximum sample size: 200 mm diameter, thickness 30 mm.
– Eulerian craddle with range from -5° to 95°.
– Sample rotation around surface normal (± 720°).
– motorized Z movement.
– X-Y mapping stage with range 100 mm × 100 mm.
– tilt stage with range ± 5°.
– High temperature chamber AntonPaar DHS1100 up to 1100 °C in vacuum (cca 0.1 mbar) or air.

Goniometer
Vertical θ–θ goniometer:
– scattering angle up to 155°
– Angular step 0,0001°
– In-plane arm with range up to +120°

Detectors
– Point scintillation detector SC-70.
– Solid state linear detector D/teX Ultra with 128 pixels.

Image courtesy of Rigaku Innovative Technologies

Contact

Core Facility: Structural Analysis Laboratory
Section: X-ray diffraction
Contact person: ONDŘEJ CAHA
E-mail: core.facility@ceitec.vutbr.cz
Detailed information: www.ceitec.eu

Instrument Location: Brno University of Technology
Faculty of Mechanical Engineering
Institute of Materials Science and Engineering
Technická 2896/2, 616 69 Brno
Czech Republic

EUROPEAN UNION
EUROPEAN REGIONAL DEVELOPMENT FUND
INVESTING IN YOUR FUTURE

2007–13
OP Research and Development for Innovation

www.ceitec.eu