BL13XU Surface and Interface Structures

Our mission at BL13XU [1] is to offer users friendly access to a two-dimensional crystallographic research at atomic-scale resolution. The main technique is combination of X-ray diffraction and scattering; in addition, state-of-the-art surface science facilities for analysis and growth are available. Target materials are widely spread from hard matter (such as a metal and an inorganic material) to soft matter like an organic semiconductor.
The beamline has three experimental hutches and representative facilities including a multi-axis diffractometer and three ultra-high vacuum chambers that can be independently coupled to a six-circle diffractometer.

Area of research

Atomic-scale structure analysis of a crystal surface, an ultra-thin film and a nanostructure
Surface structure analysis under thin-film growth
Analysis of nanostructures grown at a vacuum/solid, liquid/solid, and solid/solid interface

Keywords

Scientific field

Thinfilm, Surface, Crystallography, Nanostructure, Diffraction, One-dimensional structure

Equipment

Ultra-high vacuum facility, Glance-at-once observation, Grazing-incidence diffraction

Source and optics

The source is the standard SPring-8 in-vacuum undulator [2] with a 32 mm period and its number of 140. The period length gives an optimization around of the brilliance of about 8.8 keV. The gap would be opened up to 50 mm and closed down to 9.6 mm. The fundamental energy available can be correspondingly covered from 18.9 to 7 keV. A photon energy range from 7 to 32 keV is usually used.

A double-crystal monochromator with the Si 111 reflection is positioned at 50.0 m from the light source. Two mirrors are located at 54.3 and 55.7 m respectively from the source. The mirrors have two stripes of a rhodium coating and a platinum coating material. The upper-stream mirrors are for rejecting harmonics of incident photons and the down-stream one is for focusing an X-ray beam. A typical beam size that user groups use is 30 μm to 100 μm.

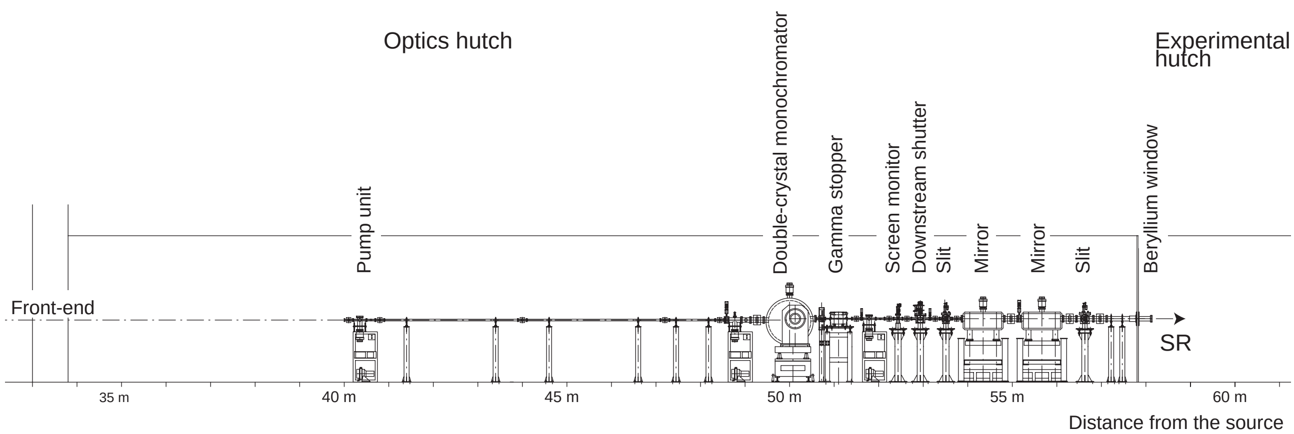


Fig.1. Schematic view of the beamline optics

Experimental stations

Multi-axis diffractometer positioned at experimental hutch 1

A multi-axis diffractometer (Kohzu-Seiki TDT-17) is designed for structural analysis of a liquid/solid and solid/solid interface. Possible techniques using the instrument are X-ray scattering in grazing incidence, crystal truncation rod measurement, and reflectivity measurements in air and liquids. The instrument can be operated in either the standard four-circle (shown in Fig. 2) or five-circle geometry. The four-circle one features 3 degrees of freedom (DOF) on a sample (axes ω, χ, and φ) and 1 DOF (a 2-θ detector arm) on an X-ray detector. An analyzer crystal can be mounted on the 2-θ arm in the geometry. A translation table in a horizontal and a rotation axis can be attached on the detector arm; (called a gamma axis) thus resulting in a five-circle geometry with 2 DOF on the detector. Smallest angular steps for the axes of the multi-axis diffractometer are listed in Table 1. A soller slit (Huber 3030-I) with an acceptance angle of 0.4 degrees can be mounted in either geometry to improve an X-ray signal/noise ratio. The instrument is mounted on a stage with rotation axis alfa along the vertical and two translation tables. An ionization chamber, a silicon PIN, a NaI scintillation detector, and a pure germanium SSD are available.

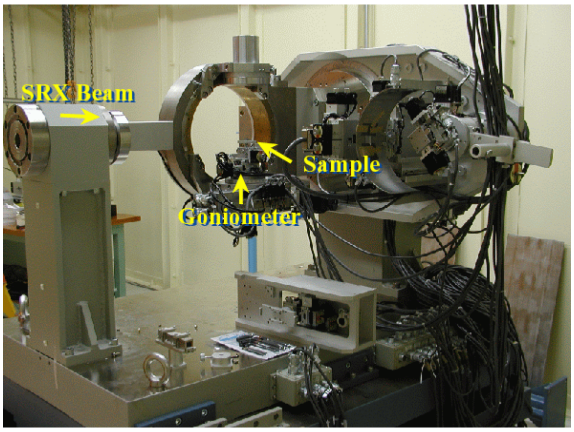


Fig. 2. Multi-axis diffractometer for structural analysis of a liquid/solid and a solid/solid interface in experimental hutch 1

Table 1. Smallest angular steps for the axes of the multi-axis diffractometer in a full step mode of motor drivers

Axis	Step (arc sec)
ω	0.09
χ	1.7289
φ	1.8
2θ	0.72
γ	3.96
α	0.144

Experimental hutch 2

A userfriendly diffractometer will be installed for thinfilm-structure analysis.

Ultra-high vacuum (UHV) chamber located at experimental hutch 3

A pack of three ultra high vacuum chambers mounted on a six-circle diffractometer (Fig.3) are available. Capabilities of the instrument include X-ray scattering studies in grazing incidence, studies of crystal truncation rods, reflectivity measurements, and X-ray standing waves in ultra-high vacuum. Examples of measurements that will be expected are the following: observation of a dynamic change in an atomic structure at a crystal surface (phase transition including order-disorder transition), analysis of a super structure at the surface. The chambers can be equipped with a low energy electron diffraction optics, a reflection high energy electron diffraction gun and its fluorescent plate, an evaporation source, a gas doser, a sputtering gun, a quartz thickness monitor, a quadrupole mass spectrometer, and a background ionization gauge. Two of sample manipulators have a sample heater and a thermocouple. In addition, one of the manipulators has a cryogenic sample holder.

Each chamber will be coupled to a diffractometer, having 2 DOF (degrees of freedom) on the sample and 2 fully independent DOF on the detector. (The diffractometer looks like a blue mammoth or 'do-hyou' for the 'sumo' game of about 3.2 m × 3.2 m × 2.3 m size.) The sample is rotated around a horizontal axis ω, which rotates about a vertical axis α. (Note, the ω axis is attached to a UHV chamber. The chamber also rotates with α) The detector is rotated around a horizontal axis δ, which rotates about a vertical axis γ. Angular resolutions per motor step are listed in Table 2.

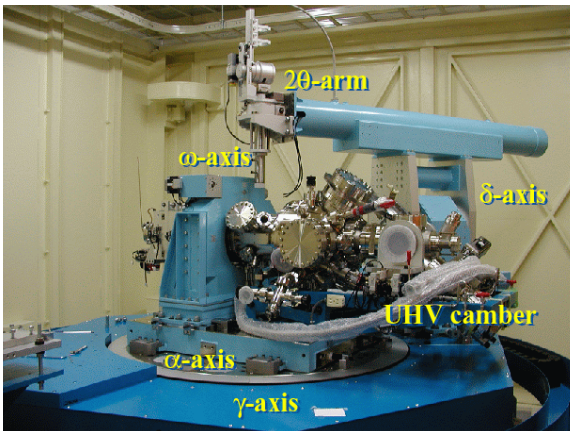


Fig. 3. The Blue 'Dohyo' diffractometer (upper) and a UHV chamber mounted on the diffractometer (lower) in experimental hutch 3

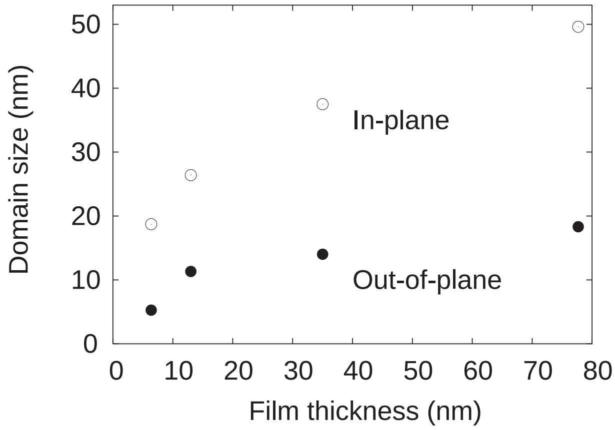
Table 2. Angular resolutions per motor step for the axes of the multi-axis diffractometer in a full step mode of motor drivers

Axis	Step (arc sec)
ω1	0.72
ω2	0.72
ω3	0.09
α	0.36
δ	0.36
γ	0.36
μ	3.6

An example of result

Growth behaviors of a NiO crystal domain both an in-plane and an out-of-plane directions were able to be explained at nano-scale resolution. They were drastically changed around 10-15 nm thin-film range. Thermodynamics factors on the nucleation and growth were dominant in an ultra-thin-film range. The step edges or terrace width of the substrates, however, limited the growth speed in a thicker film range.

Details are described in [3].



Domain sizes vs. NiO film thickness. The films were grown on ultra-smooth sapphire substrates.
(We borrowed this figure from [3])

References

[1] O. Sakata et al. Surface Review and Letters 10 (2003) 543.

[2] H. Kitamura, J. Synchrotron Rad. 7 (2000) 121-130.

[3] O. Sakata et al., Applied Surf. Sci. 221 (2004) 450-454.

Contact information

Osami SAKATA
SPring-8 / JASRI
1-1-1 Kouto, Mikazuki-cho, Sayo-gun, Hyogo 679-5198
Phone : +81- (0)791-58-2750
Fax : +81- (0)791-58-0830
e-mail : o-sakata@spring8.or.jp