BL24XU
(Hyogo ID)

1. Introduction

BL24XU, which is the Hyogo ID beamline, is a contract beamline dedicated to industrial applications constructed by the Hyogo prefecture. It is a branched beamline employing a figure-8 undulator light source, diamond (220) beam-splitting monochromator for the branched line A, and silicon (111) double-crystal monochromator (DCM) for the mainstream B, and provides the methods for local structural analysis of materials (Table 1).

BL24XU provides an effective scheme to investigate structure–function relationships of products and feedback them to material processes. Materials informatics utilizing machine learning is a promising technique to accelerate the development of materials in the trial-and-error stage. In the materials informatics approach, a number of specimens under different conditions are investigated to derive relationship among structure, function, and process. To improve both the collection efficiency and quality of data, we upgraded the mainstream optics. Hard X-ray ptychography was implemented to investigate structures at a spatial resolution better than several tens nm, which is essential for materials' function. Here, we report these upgrades and describe future prospects of implementing the materials informatics approach to synchrotron radiation analyses.

2. Beamline upgrades

2-1. Upgrades of the mainstream optics

At the end of FY2017, the mainstream optics were upgraded to improve the photon flux, coherence, and stability of monochromatic X-rays (Fig. 1). Prior to this upgrade, direct water-cooled DCM was applied, but it caused divergence of the beam and a strong astigmatism in the focusing optics due to the

Table 1. Specifications of the measurement techniques in BL24XU.

Measurement techniques	Structural Information	Spatial resolution
Projection / imaging microscope / coherent diffraction CT	2D/3D image Field of view 1 mm ~ 1 μm Absorption, refraction contrast (projection / imaging microscope) Absorption, phase contrast (coherent diffraction)	0.33 μm ~ 10 nm
Microbeam SAXS / WAXD / XRF	Periodic / aggregation structures of several hundred nm ~ angstrom Distribution of crystal grains Elemental mapping	5 ~ 0.5 μm
Bonse-Hart USAXS	Periodic / aggregation structures of 6500 ~ 16 nm	bulk
Highly-pararell microfocus diffraction, bright-field topography	Local strain, dislocation	30 ~ 0.5 μm (diffraction), 0.65 μm (topography)
Near ambient pressure HAXPES	Chemical state	30 μm

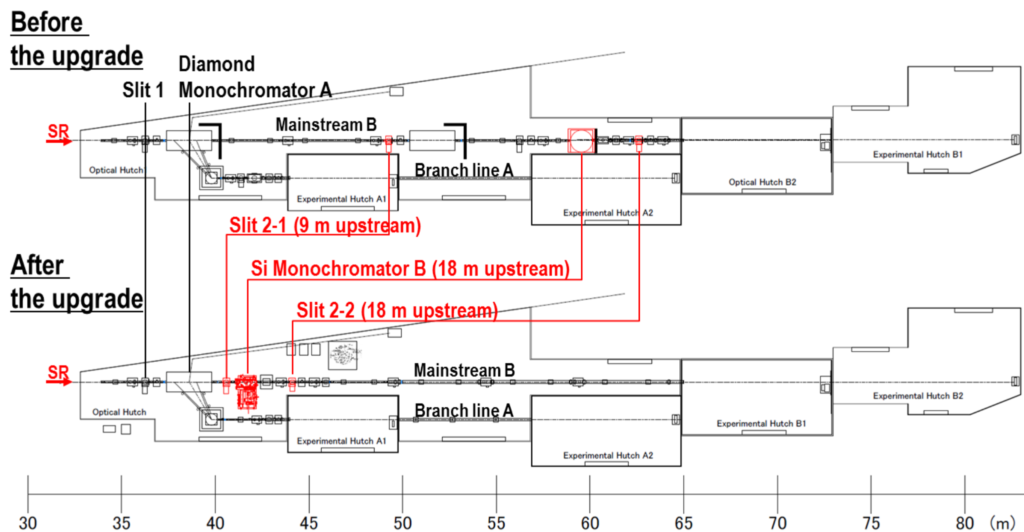

Fig. 1. Arrangement of the beamline optics before and after the upgrade.

residual heat load on the Si crystals as the emittance of the storage ring improved. The DCM and beam defining slits were too close in the experimental hutch to control the spatial coherence (Fig. 1). To address the above issues, the DCM was replaced with the standard liquid nitrogen cooled type [1] and the DCM and slits were arranged ~ 18 m upstream from the previous position (Fig. 1).

In April 2018, we commissioned each measurement technique, except the microbeam SAXS and the Bones-Hart USAXS implemented in branch line A. The asymmetric shape of the rocking curve for the previous DCM was improved to the ideal shape, even when the slits before the DCM were fully opened. The reduced beam divergence yields approximately twice the beam intensity and nearly ideal focusing for each measurement technique. Especially for the HAXPES system, the acceptance of the K-B focusing mirrors was limited due to the heat load on the previous DCM. However, after the upgrade, the K-B focusing mirrors can operate with the full acceptance without noticeable deformation of DCM crystals. This realized a ten-fold increase in photon flux at the sample position. The beam stability was also improved, as demonstrated by the successful implementation of ptychography measurements, which require a high spatial coherence and a positional stability less than 20 nm during a measurement.

2-2. Development of hard X-ray ptychography

Advances in materials science reveal the importance of structures from micro- to nanoscale for understanding and controlling their properties. Hence, the demand for visualization of nanostructures in samples that are too thick for electron microscopy is increasing. X-ray ptychography has potential to meet this demand. In ptychography, an imaging target is scanned with a spatially coherent focused probe so that the illumination areas overlap. Then far-field diffraction patterns from each illumination area are collected and subjected to a phase retrieval with constraints regarding the overlap to give a projected complex refractive index map of the target. Consequently, ptychography can achieve a spatial

resolution beyond lens-based X-ray microscopy with high contrast based on the phase shift in a sample.

We constructed the ptychographic nanoimaging system (Fig. 2 (a)) by implementing newly developed coherent illumination optics to the previously developed atmospheric coherent diffraction imaging system [2, 3]. Although ptychography is quite sensitive to drift, we achieved a spatial resolution of 20 nm/pixel for the resolution test chart fabricated on a 500-nm thick tantalum membrane (Fig. 2 (b)). Phase contrast maps obtained by ptychography display much higher contrast than the amplitude contrast (Fig. 2(b), (c)). This system will open to users in 2019A.

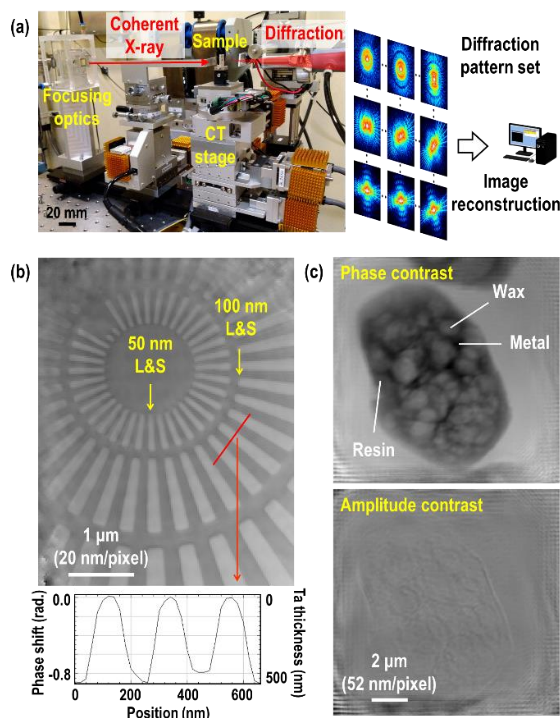

Fig. 2. X-ray ptychography at BL24XU.
(a) Photograph of the experimental setup.
(b, c) Projection maps of a 500-nm-thick tantalum resolution test chart and a black toner particle.

3. Future prospects

In FY2018, we began research projects in collaboration with industrial users utilizing materials informatics. These efforts have highlighted the importance of analyses for blind decomposition of spectroscopic or scattering data into each component to derive key structural information. It is also important to reconstruct high-quality data from noisy or imperfect experimental data using compressed sensing and deep learning approaches. In the next annual report, we will update the progress on the development of these analysis methods and their applications on materials science.

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