BL47XU
Micro-CT

1. Introduction

BL47XU, which is an X-ray undulator beamline, is dedicated to micro/nano-CT and high-speed X-ray imaging. Those experiments need a high-flux-density monochromatic beam. To handle the high heat load of the undulator, a liquid-nitrogen (LN_2) cooling system is used to cool the monochromator crystals. The available energy range is between 6 keV and 37.7 keV with a Si (111) reflection of the monochromator. To eliminate higher harmonics, a set of reflection mirrors (double bounce in the vertical direction) can be inserted.

The beamline has two experimental hutches (EH1 and EH2) located just after the optics hutch. EH1 contains experimental tables for X-ray micro/nano-CT, while EH2 contains some X-ray optics and X-ray image detectors for nano-imaging. Half of EH2 can be used as an open hutch for users who bring their own special equipment. Recently, a chamber for a condenser zone plate (CZP) and micro-loading system have been installed and used for experiments by users. The details and some results are described here.

2. Introduction of CZP chamber

We have introduced a large helium gas chamber for the X-ray nano CT with full-field X-ray microscope optics operating at BL47XU [1]. Although operation in an equivalent energy range was possible with the previous configuration, it necessitated significant setup changes when switching between energies and between projection (micro-imaging) and microscope (nano-imaging) modes. This initiative is aimed at resolving this issue, enabling continuous realization of the 7–15 keV energy range in nano-imaging and allowing seamless use of micro-imaging.

The chamber is manufactured to be vacuum-compatible and can be filled with helium gas. The X-ray windows use a 6-µm-thick SiN membrane, which can withstand atmospheric to vacuum pressure. Two sets of windows are positioned about 10 cm apart horizontally. One is for micro-imaging, and the other is for nano-imaging. The micro-imaging side has no optical elements and serves as a simple helium path. On the nano-imaging side, a CZP is mounted on a hollow rotation stage that rotates at about 4 Hz during experiments to achieve hollow cone illumination on the sample. As the CZP needs to change its position along the optical axis for each energy, it is placed on a stage about 60 cm long, along with the rotation stage. Additionally, to adjust the position after moving along the optical axis, stages for horizontal and vertical movements perpendicular to the optical axis are also installed.

The sample and the objective lens are positioned on a separate experimental table, as shown in Fig. 1. This arrangement is primarily to eliminate the effects of vibration caused by the rotational motion of the CZP.

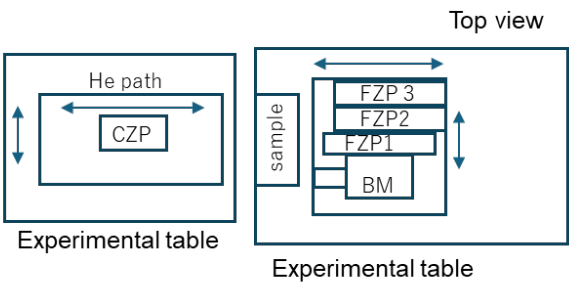

Fig. 1. Schematic illustration of the positions of optics.

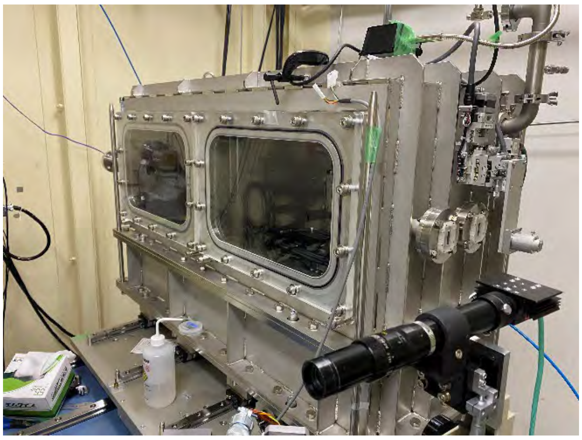

Fig. 2. Photograph of the chamber.

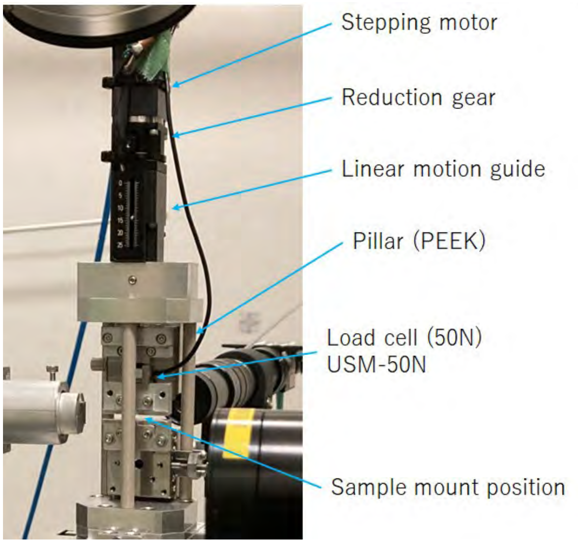

Fig. 3. Photograph of microloading system for micro/nano-tomography.

3. Introduction of micro-loading system for micro/nano-tomography

Recent equipment improvements have enabled switching between micro- and nano-modes. Additionally, depending on the conditions, CT measurements of samples can now be completed within one minute. This has made it possible to conduct *in situ* observations of sample deformation and other phenomena. However, the testing machines that can be incorporated into the beamline equipment must be small and lightweight, which is nearly impossible with commercially available products. Therefore, we attempted to create our own testing machines [2].

The basic configuration of the device consists of a stepping motor and a linear guide. It also includes a load cell for force measurement. To facilitate sample exchange, the device is supported by three PEEK rods of 6 mm diameter. With this thickness, a certain level of transmission X-rays can be maintained at 15 keV, and no missing angles occur. This has sufficient performance to pull (or compress) the sample and hold it in place during CT measurements.

As a demonstration of the device, an *in situ* observation experiment of the deformation of an aluminum-manganese alloy was conducted using the micro-loading system. Figure 4 shows the results of micro-tomography. The measurement conditions were as follows: X-ray energy was 15 keV, exposure time per projection was 5 ms, number of projections was 1000, pixel size was 508.0 nm/pixel, and the qCMOS camera (ORCA Quest, Hamamatsu Photonics) was operated in the 2×2 binning mode (2048×1152 pixels). While the total imaging time was about 60 s, the actual exposure time of the sample was 25 s.

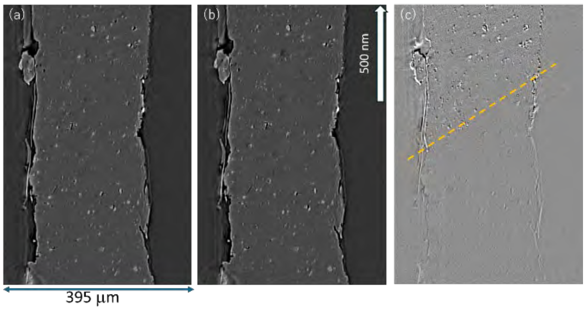

Fig. 4. (a) Initial state of Al–Mn alloy. The image shows the structure of the alloy along the rotation axis and tensile loading direction. (b) After 500 nm tensile loading. (c) Subtraction image of (b) and (a). The deformation of the upper part is larger than that of the lower part.

4. Introduction: Open Hutch

Since 2022, the beamline BL47XU has been open for users who carry in their own instruments, such as a diffractometer, to EH2 during their beamtime to perform specific experiments of their own design not utilized in the public beamlines. To support such demands, EH2 has a barrierless floor around a door system to help load/unload user's instruments (see Fig. 5). Double-bend mirrors 400 mm long in the optical hutch are also available to reject higher harmonics and focus an incident X-ray beam close to 0.1 mm in size in the vertical direction, in addition to the above-mentioned Si(111) monochromator as the standard optics.

5. Beamtime opportunities with user's instruments

To encourage users to realize flexible experimental setups, we offer the minimal hutch optics, i.e., slits, incident beam monitor, and attenuator, to allocate enough space for the user's equipment in the hutch. These are built in a shield box to reduce the background noise level (see Fig. 6) and are controlled by SPEC [3], the world standard software in diffractometry, to meet user requirements. A concise vacuum beam path and a Si PIN detector are also available for the effective use of synchrotron X-rays in a wide energy range and alignment of user instruments, respectively.

X-ray fluorescence holography (XFH) and anomalous X-ray scattering (AXS) users actively have applied for beamtime opportunities opened for instruments carried by users, as shown in Fig. 2, because these two techniques need special experimental setups using the user's own diffractometer to realize element-specific analyses. XFH requires an analyzer with wide solid-angle acceptance to collect weak signals of X-ray holograms, while AXS observes energy-resolved total scattering (with energy resolution better than 10 eV) with a crystal analyzer in two different incident X-ray energies, either one of which is close to the absorption edge of a specific element in a material.

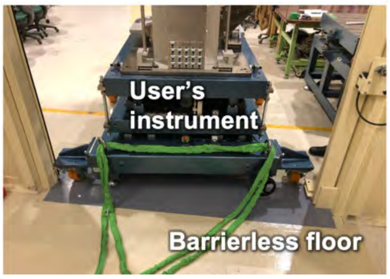

Fig. 5. Photograph of loading a user's instrument into EH2 with a barrierless floor.

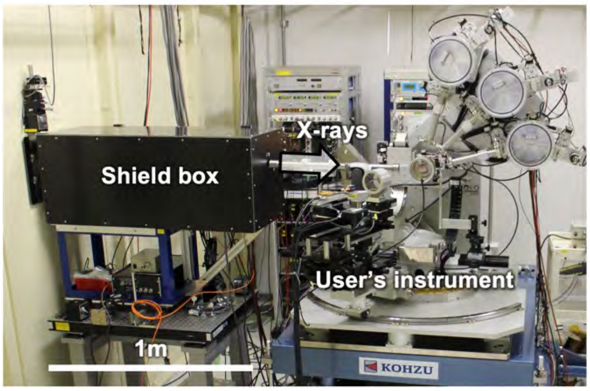

Fig. 6. Typical experimental setup for carry-in user instrument with a shield box.

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References:

[1] Uesugi, K. et al. (2023). *AIP Conf. Proc.* **2990**, 040019.

[2] Uesugi, K. et al. (2024). *Proc. SPIE*, to be published.

[3] https://certif.com/spec.html