BL13XU
(Surface and Interface Structures)

1. Introduction

BL13XU is dedicated to reveal the structures of surface layers on solids and thin films on the atomic scale via X-ray diffraction/scattering (XRD). Techniques typically include grazing-angle X-ray diffraction, crystal-truncation-rod (CTR) scattering, reflectivity, microbeam diffraction, and reciprocal-space mapping in a vacuum or in air. To investigate surface structures by the aforementioned XRD, an ultrahigh-vacuum (UHV) chamber is mounted on a diffractometer. This chamber is equipped with tools for sample preparation and surface analysis. Target materials range from hard matter (such as a metal and an inorganic material) to soft matter like an organic semiconductor. A photon energy ranging from 6 keV to 50 keV is usually used.

Recently, diffraction patterns from nanostructures such as atomic wires, nanodots, and ultrathin films have been measured. Local structures of device materials (*e.g.,* strain) have been revealed using microbeams. Not only static structure analysis of a solid surface/interface, but also *in situ* observations of a dynamic structural response of a surface such as a metal electrode through the imposition of an external field are encouraged.

In addition to the in-vacuum undulator source and the standard optics, middle-energy-bandwidth optics are available using an asymmetric double-crystal monochromator with the Si 111 reflection to supply the growing demand for a high photon flux [1]. The monochromator stabilization system and the fast tuning of the incident X-ray energy for anomalous XRD are utilized to meet users' requirements.

Here, we report technical developments and upgrades of the beamline instruments implemented in FY2018.

2. Real-time indexation of reciprocal space maps

A two-dimensional (2D) detector, especially a photon-counting 2D detector, is used in synchrotron XRD due to its low noise, high photon-detection efficiency, high count-rate, high spatial-resolution, and large active area compared to other detectors. BL13XU offers two series of 2D detectors, PILATUS-100K (Dectris) and HyPix-400 and HyPix-9000 (RIGAKU) for structural analysis of thin films and a liquid/solid interface using multi-axis diffractometer in the first experimental hutch and UHV experiments in the third hutch.

On the other hand, *spec*, which is the leading software for instrument control and data acquisition in XRD used at synchrotron facilities around the world, was adopted at BL13XU [2]. *spec* not only supports a large number of instruments, including motor controllers, detection electronics, and detectors, but also provides easy access to reciprocal space and supports easy-to-use macros for users to build up their own measurement procedures. However, one drawback is the monolithic package. This results in difficulties in sharing and/or introducing programs developed by the other systems to the spec-based measurement system.

Therefore, we applied the distributed control system, *Data collection And control system for X-ray stations Using MADOCA* (DARUMA), to the beamline with great support by Dr. Nakada [3].

Owing to the cooperation of DARUMA with *spec*, we can control the beamline instruments flexibly, including the aforementioned 2D detectors. In addition, using DARUMA, we introduced a program to index an observed XRD image using Miller index in real time, which can be controlled via *spec*.

Figure 1 shows a screen shot of the DARUMA image viewer, where the Miller index diagram is overlaid on the acquired XRD image to help identify CTR scatterings. The Miller index diagram is calculated by evaluating the so-called UB matrix from the angular information on the diffractometer,

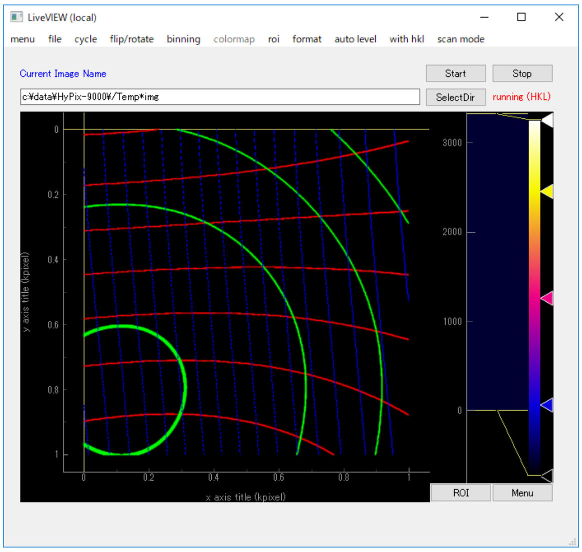

Fig. 1. DARUMA image viewer. Miller index diagram is overlaid on the acquired XRD image. Red, blue, and green lines represent integer grids of the h, k, and l in the Miller index, respectively.

crystallographic parameters of a sample, and the detector specifications.

By introducing the DARUMA system, flexible control of the existing beamline instruments is realized and additional controls can be introduced easily in the future.

3. High-resolution micro/nano-beam X-ray diffraction

The micro/nano-beam XRD technique is an excellent tool to reveal local lattice distortions in materials such as semiconductor thin films or electric devices with a high-spatial resolution. In the fourth experimental hutch at BL13XU, the micro/nano-beam XRD system is arranged with a Fresnel zone plate (FZP) and compound refractive lenses (CRL) as focusing devices (Fig. 2) [4,5]. Previously, a CCD-based camera was used as a detector, but weak diffraction signals were difficult to detect due to the readout noise.

To overcome this problem, we installed a pixel array detector (STPX-65k by Amsterdam Scientific Instruments) in 2018. The detector is based on the Timepix technology developed at CERN. The detector has features such as low noise and a high frame rate. The sensor is 1-mm-thick

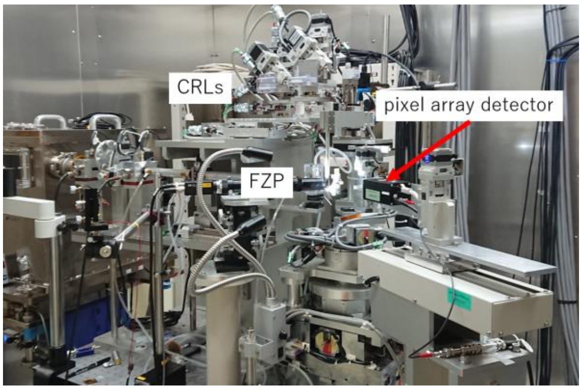

Fig. 2. Micro/nano-beam XRD system in the fourth experimental hutch at BL13XU.

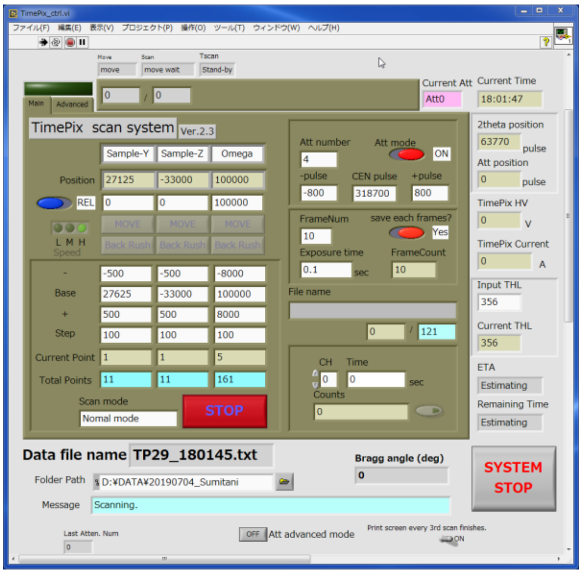

Fig. 3. Interface of the measurement software.

Si, which has a high efficiency that exceeds 80% at 8–15 keV. The pixel pitch is 55 μm. The maximum distance from a sample to the detector is about 420 mm in the system. Thus, the minimum step of the 2θ angle is about 0.0075°. The number of pixels and detection area are 256 × 256 and 14 mm × 14 mm, respectively. The maximum frame rate is 120 frames per second.

To install the detector in the micro/nano-beam XRD system, we developed a series of software for the measurements (Fig. 3), image corrections, and analyses. A mesh scan with up to three axes can be performed by the measurement software, where reciprocal lattice mapping (RLM) is performed by changing the *xz* position on the sample. To set the detector, the exposure time and the number of frames are specified so that a wide dynamic range is achieved by integrating each frame. First, images from the detector are corrected for the analyses such as the flat field correction, image rotation, and filter processing. The results of the RLM measurements are checked immediately by the analysis software. It also has a function to export RLM data to a text file for commercial graphing software.

By introducing a pixel array detector, the weak diffraction signal can be detected with a high sensitivity. The measurements are faster, allowing more effective use of the beam time.

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