# BL13XU
# Surface and Interface Structures

## 1. Introduction

The beamline BL13XU is dedicated to reveal the atomic-scale structures of surface layers on solids and thin films by X-ray diffraction/scattering (XRD). The techniques users often use are grazing-incidence X-ray diffraction, crystal-truncation-rod (CTR) scattering, reflectivity, microbeam diffraction, and reciprocal-space mapping in vacuum as well as in air. When a user investigates a surface structure by the above-mentioned XRD technique, he/she uses an ultrahigh-vacuum (UHV) chamber mounted on a diffractometer. The chamber is equipped with tools for sample preparation and surface analysis in advance. Target materials are widely spread from hard matter (such as a metal and an inorganic material) to soft matter such as an organic semiconductor. A photon energy ranging from 6 to 50 keV is often used.

Many users have recently measured diffraction from nanostructures such as atomic wires, nanodots, and ultrathin films. Local structures of device materials, such as strains, have also been revealed using a microbeam. Not only the static structure analysis of a solid surface/interface, but also the in situ observation of a dynamic structural response of a surface such as a metal electrode through the imposition of an external field is encouraged.

In addition to the in-vacuum undulator source and standard optics, we offer the middle-energy-bandwidth optics using an asymmetric double-crystal monochromator with the Si 111 reflection to supply the growing demand for 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.

The technical developments and upgrading of the beamline instruments conducted in FY2020 are described in this paper.

## 2. SXRD at ultralow temperatures

The research on low-dimensional physics has been accelerated by the recent rapid progress in the manipulation of nanometer-scale structures on crystal surfaces, where two-dimensional well-ordered structures can be obtained.

Therefore, understanding the atomic structure of a surface is a starting point for tailoring novel surface materials that meet our requirements and for understanding surface functions such as catalytic reactions. Surface X-ray diffraction (SXRD) is one of the state-of-the-art techniques of determining the constellations of atoms on crystal surfaces, including adsorbates, thin films, and relaxed layers [2].

In low-temperature physics, on the other hand, the quantum phases of He atomic layers on graphite at ultralow temperatures have been attracting much interest over decades as model systems for realizing quantum materials [3]; however, their structures are still unrevealed. That means that SXRD will provide a new opportunity for studying He quantum phases from the firm basis of its atomic constellation if we overcome difficulties in providing an ultralow-temperature environment for SXRD. Toward such a new frontier of SXRD, we developed a sample cooling system to reach around 1 K at BL13XU.

Figure 1(a) shows a photo of the ultralow-temperature cooling system that we developed, which consists of a 0.1 W cryocooler (RDK-101E, Sumitomo Heavy Industries, Ltd.) with an ICF114 standard flange, a 50 K stage, a 4 K cold head, a He pot, and a 1 K pot. At the end of the 1 K pot, we can install a sample holder for surface XRD. A temperature trend during cooling in a high-vacuum chamber is shown in Fig. 1(b). After the sufficient liquefaction of He in the 1 K pot, the evaporative cooling of the pot starts by pumping out He gas using a turbomolecular pump. Appropriate thermal anchors and insulations, such as a radiation shield and super insulation, enabled the system to reach a temperature of 1.33 K and maintain it for several hours.

Our cooling system has just been opened for public use and can accelerate the advancement of structural surface science at ultralow temperatures using synchrotron XRD. In particular, a structure analysis of He atomic layers on graphite, which is a very important target as a low-dimensional quantum model system, is currently in progress.

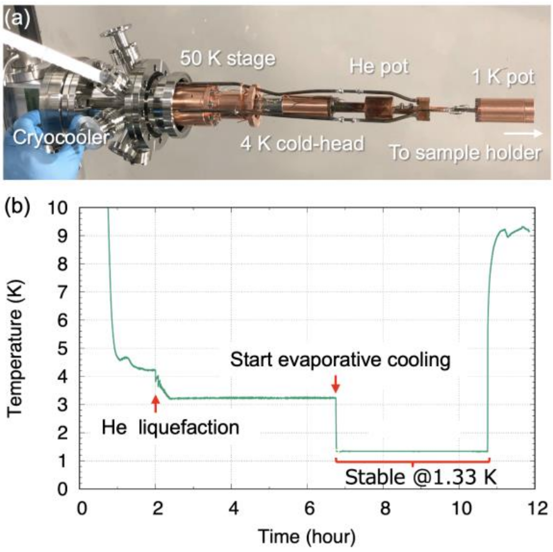


Fig. 1. (a) Photo of ultralow-temperature cooling system. (b) Temperature trend of 1 K pot.

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

Micro/nanobeam X-ray diffraction (XRD) is an excellent method of revealing local lattice distortions in materials such as semiconductor thin films or electric devices with high accuracy. In the fourth hutch at BL13XU, a micro/nanobeam XRD system is arranged with a Fresnel zone plate (FZP) and compound refractive lenses (CRLs) as focusing devices [4, 5].

To achieve a high resolution of a scattering angle for high sensitivity on the lattice distortion, the camera distance from the sample should be sufficiently large. In our system, the maximum camera distance is designed as 1000 mm with a HyPix-3000 detector produced by Rigaku Co., which has a pixel size of 100 μm ×100 μm, resulting in an angular resolution of 0.0057 degrees. However, the large distance between the sample and the detector causes the attenuation of the scattered X-rays by air. To decrease the attenuation, a beam path filled with He gas is necessary.

In 2020, we installed a He beam path system for this purpose. The schematics are shown in Fig. 2. The beam path is put in front of the detector on the two-theta axis. The aperture of the beam path is sufficiently large compared with the active area on the detector. Several types of beam path with different lengths are prepared for different camera distances. The beam path is mounted on the motorized Z stage that enables the path to escape from the optical axis. The camera distance can be changed freely from 120 mm to 1000 mm when the beam path is at the retracted position (Fig. 2(b)). This mechanism is convenient in some situations such as the search for Bragg conditions for the sample.

With the beam path, weak scattering signals are obtained more effectively, which will enhance the advanced application of the micro/nano XRD system such as time-resolved measurement with an X-ray chopper system [6].

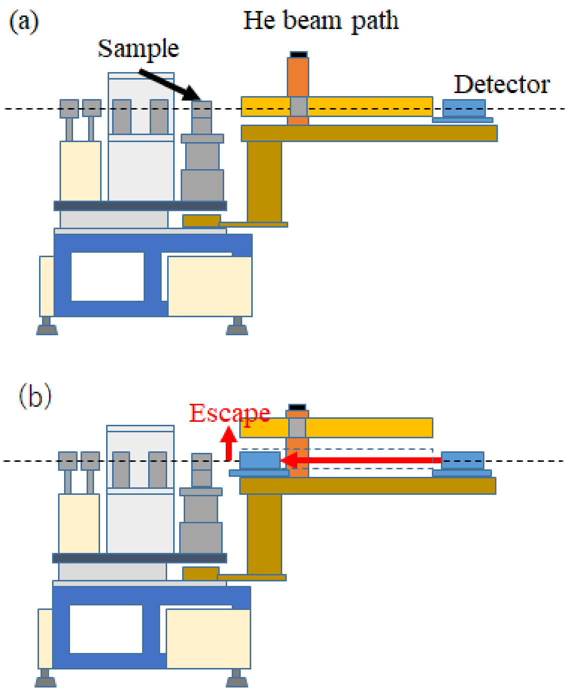


Fig. 2. Schematics of He beam path. (a) In the measurements, the beam path is put on the optical axis. (b) When the camera distance is changed, the beam path escapes above at the retracted position.

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**References:**

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