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Advanced Micro-Crystal Chemical Crystallography

A beamline for micro-crystal X-ray diffraction (μ-XRD) is a dedicated beamline designed for advanced and non-ambient crystallography of chemical crystal research. This beamline, **TPS 15A**, is scheduled to be a Phase-II beamline at Taiwan Photon Source (TPS), and its construction began in 2019.

The layout of **TPS 15A** is shown in **Fig. 1**. It consists of a tapered cryogenic undulator source (CUT18) that will generate highly brilliant X-rays in designed energy range 9–35 keV. The photon flux of this energy range is 10^{13}–10^{14} photons/s (0.1% bw), shown in **Fig. 2**. X-ray beam modes of two types, monochromatic and pink beams, are selected with a coupled double-crystal monochromator (DCM)/double-multilayer monochromator (DMM) system. The energy bandwidth of the pink beam is designed to be 3 and 5% according to the multilayer coatings Pd/B_4C and W/B_4C, respectively. Three focusing mirrors (toroidal FM, VFM and HFM) are designed to focus the X-ray beam spot, first focused with the FM, then delivered to the VFM and then the HFM, down to diameter 10 x 10 μm² (FWHM). Two endstations (ES1 and ES2) will be installed; one locates after FM and another locates at the focused beam spot. The beam spot size is adjustable in the range 200 x 200–100 x 100 μm² at ES1 and 70 x 70–5 x 5 μm² at ES2. Experiments can be conducted in either monochromatic (with the DCM) or pink (with the DMM and selected bandwidth 3 or 5%) beam mode in both endstations. A home-built diffractometer will be installed at ES1; a highly precise microdiffractometer (MD3UP) and a large-area detector (EIGER 2X CdTe 9M) will be installed at ES2, shown in **Fig. 1(a)**.

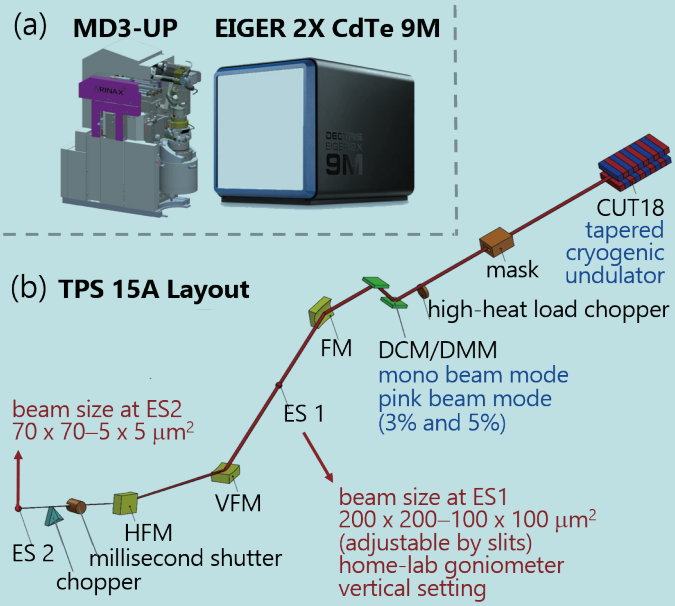

Fig. 1: (a) Microdiffractometer MD3UP and detector EIGER 2X CdTe 9M. (b) Layout of **TPS 15A**.

Single-crystal X-ray diffraction is a mature and routine technique that provides structural information from a single crystal. This information is important for the development of new materials through the relation with its physical and chemical properties. For example, graphite and diamond consist of carbon atoms, but their physical and chemical properties, such as shape, color, hardness *etc.*, differ markedly. Those differences can be explained by their structure–carbon atoms in graphite connect to each other with trigonal coordination, but in diamond all carbon atoms bind to each other with tetrahedral coordination.

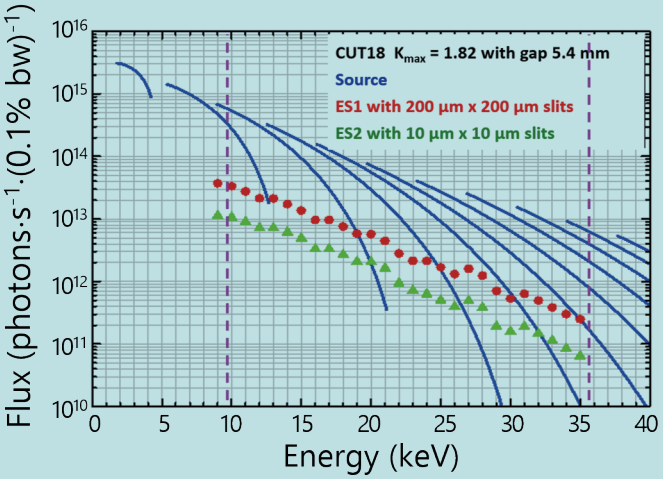

Fig. 2: Photon flux of CUT18 in designed energy range 9–35 keV.

In past decades, researchers tried diligently to synthesize novel materials of crystals with specific functionalities such as mechanical motion, gas absorption and desorption, shape memory, property change with external stimuli *etc*. To determine the crystal structures of these materials is, however, difficult in some cases for reasons such as too small crystal size, easy drying, experimental setting for external stimuli and so on.

A structural determination of even a powder sample is possible on taking advantage of second-generation synchrotron-radiation (SR) sources since the 1990s. The third-generation high-energy SR sources, such as European Synchrotron Radiation Facility (ESRF), Advanced Photon Source (APS) and SPring-8, at present provide stable beams of superior quality that allow microanalyses of many types, including the structural determination of crystals of μm size; well developed focusing mirrors apply a highly brilliant X-ray beam that enables a determination of a crystal structure from a single powder grain (< 1 μm).

Nowadays, researchers can routinely use synchrotron radiation as an X-ray source for single-crystal X-ray diffraction tasks. The continuing development of synchrotron radiation sources, detectors, software and precise positioning control are essential to establish a knowledge of the function and properties of complicated chemical systems and materials. Several advantages (according to the design of **TPS 15A**) can hence be taken of such highly brilliant X-ray sources (relative to a conventional laboratory source):

1. Monochromatic X-ray source–no contamination of $K_{\alpha 2}$;
2. Multi-wavelength X-ray source (pink beam)–Laue diffraction;
3. Tunable energy–small absorption, small extinction, high data resolution (X-ray energy available up to 35 keV), great redundancy;
4. Highly brilliant–brief duration of data collection, large ratio of signal to noise;
5. Micro-focus beam size–5 x 5 μm^2 (with a pin hole of size 5 μm);
6. Time-structure–dynamic structure and structural determination of excited states.

The great flux and fine focus of synchrotron X-rays enable the study of tiny crystals, complicated molecular structure, insufficient quality, easily decomposing crystal, which are impracticable with a conventional laboratory source. The beam of size ~5 μm not only makes it possible to study a tiny crystal and structural mapping, but also benefits the study under various non-ambient conditions, such as ultra-low temperature with gas flow from liquid helium, high pressure in a diamond anvil cell, photo-excitation crystallography *etc*. Taking advantage of the temporal structure of synchrotron radiation allows the conduct of time-resolved studies to investigate the chemical dynamics and excited-state structure. A set of X-ray beam pulse selectors will be synchronized to the storage-ring clock, which will serve to isolate a single X-ray pulse ~25 ps in FWHM) (hybrid-injection mode) and delivered to a sample at ES2. These conditions benefit the fields of chemistry, mineralogy, material science and so on. The scientific opportunities of **TPS 15A** are shown in **Fig. 3**.

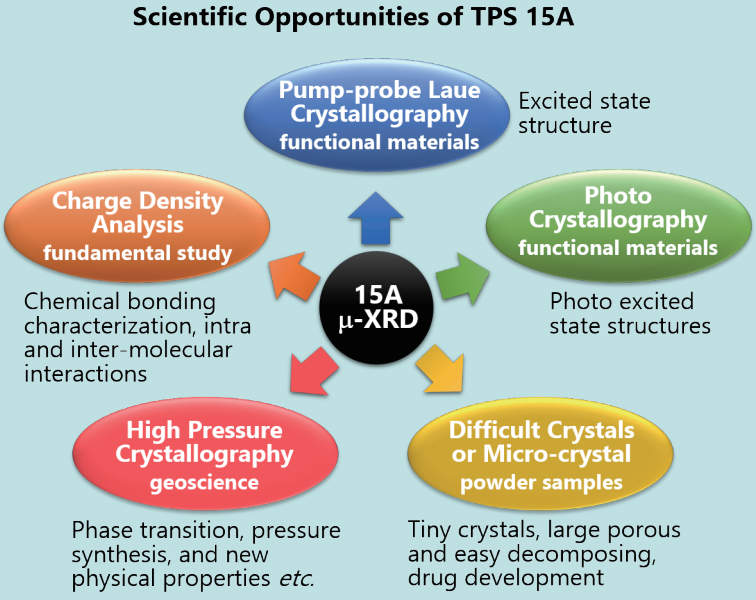

Fig. 3: Scientific opportunities of **TPS 15A**.

Year	2019	2020	2021	2022
Construction of hutch and utilities				
Design, assembly and installation of optics, vacuum components				
Design, assembly and installation of CUT18				
Installation of DCM				
Installation of mirrors (FM, VFM, HFM)				
Commissioning of beamline				
Commissioning of ES2, open to users				
Commissioning of ES1, open to users				
Commissioning of ES2 at TPS 09A2, open to users				

Fig. 4: Construction schedule of **TPS 15A**.

TPS 15A is currently under construction; the schedule appears in **Fig. 4**. The construction of a radiation safety hutch was completed at the end of 2019. In 2020, the main construction will focus on the beamline optical components and utilities. The entire beamline is expected to be commissioned at the beginning of 2022. (Reported by Lai-Chin Wu)

Soft X-ray Nano-Spectroscopy for Advanced Material Sciences

Symmetry is one of the most general and fundamental concepts in physics, yet sometimes what novelty needs is to break it. Take two-dimensional (2D) materials as an example, their reduced size and broken symmetry have led to many exotic properties. To unravel the electronic origins of these emergent properties, however, conventional spectroscopy along with microscopy is better, allowing the location and dimension of the specimen can be taken into account.

Beamline **TPS 27A** hosts two soft X-ray microscopy stations; a scanning transmission X-ray microscopy (STXM) station at the A1 branch and a photoelectron related imaging and nano-spectroscopy (PRINS) station at the A2 branch. The photon source powering **TPS 27A** is a four-meter long elliptically polarized undulator (EPU) with a

(a)

(b)

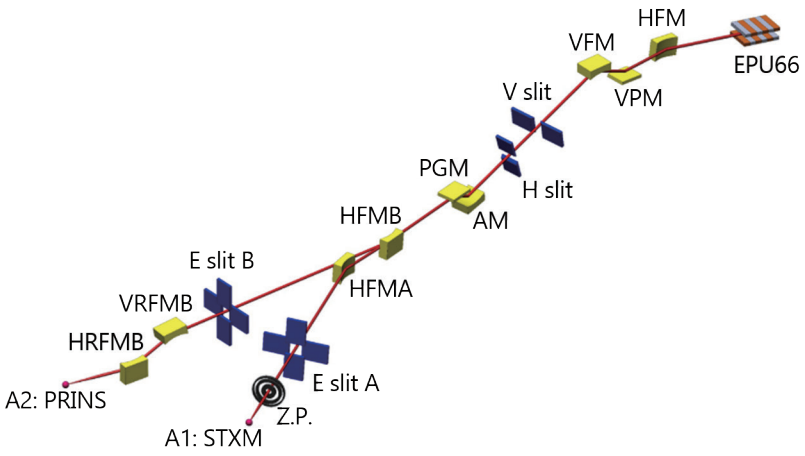

Fig. 1: (a) Mechanical frame of EPU 66. (b) Optical layout of **TPS 27A** soft X-ray nano-spectroscopy beamline.