Development of a Beamline for Coherent X-ray Scattering

Taiwan Photon Source (TPS) is one up-to-date third-generation synchrotron radiation source among others all over the world. One most remarkable feature of a third-generation source of synchrotron radiation is its production of intense beams of coherent X-ray photons. The development of coherent X-ray scattering techniques was previously restricted by the lack of a strong coherent X-ray photon source. Through the extremely brilliant and highly coherent X-ray beams provided by TPS, the opportunities to develop coherent X-ray scattering experiments such as X-ray photon correlation spectra (XPCS) and coherent diffraction imaging (CDI) have been launched. The coherent X-ray scattering (CXS) beamline, **TPS 25A**, is one of the first seven beamlines at TPS. The construction of the beamline and endstation was completed at the end of 2015. The beamline and endstation commissioning began in the first half of 2016; in the second half year of 2016 the CXS beamline was opened to public users. Until now, fifteen groups have performed their experiments including XPCS, CDI, small-angle X-ray scattering (SAXS) and wide-angle X-ray scattering (WAXS) at the CXS beamline.

The core techniques of the CXS beamline are XPCS, CDI and SAXS. In materials science the dynamics is a cardinal topic. XPCS can directly access the dynamic response function $S(Q,t)$ to obtain information about the dynamics. In practice, as the coherent X-ray photons are scattered by a disordered system, the scattering speckle patterns fluctuate temporally; these fluctuations relate to the intrinsic motions of the system. A sequence of scattering patterns is collected with time. On calculating the correlations between each scattering pattern, the embedded dynamics is extracted. One advantage of this method is to study dynamics comprised of many collective processes. XPCS has been applied in many research fields, such as diffusion of colloids, complicated fluids, polymer blends, clays, capillary fluctuation, liquid-crystal membranes, non-equilibrium dynamics, binary alloys, metal/polymer nano-composite, and charge-, spin-, orbital-, ordered domains. The progress of XPCS development is currently in both hard and soft condensed matter. There still are some limitations of XPCS; the photon flux and the frame rate of the detector are two major factors to confine the range of the correlation time. For a long-time data collection, radiation damage is another issue to the sample system.

CDI is a lensless image technique. When coherent X-ray beams illuminate a specimen, the diffraction patterns are collected. Through the phase retrieval algorithms,[1-5] the phase problem is resolved and the real-space images of the specimens are reconstructed. In the phase-retrieval algorithms, the real-space support information is required for CDI image reconstruction, which is a limitation of the CDI technique. Furthermore, to cope with a large specimen, a scanning-type CDI, ptychopgraph, can also be run in the CXS beamline. The redundancy or the overlapped region in neighboring scans is the real-space support of ptychopgraphy. The ptychographic iterative engine (PIE) and extended PIE (ePIE) algorithm[6,7] are developed for the ptychographic data processing. SAXS experiments can also be conducted.

The CXS beamline is located at TPS port 25, which is an output port of a straight section of length 12 m. The photons of the CXS beamline are irradiated from two in-vacuum undulators IU22. The photon source

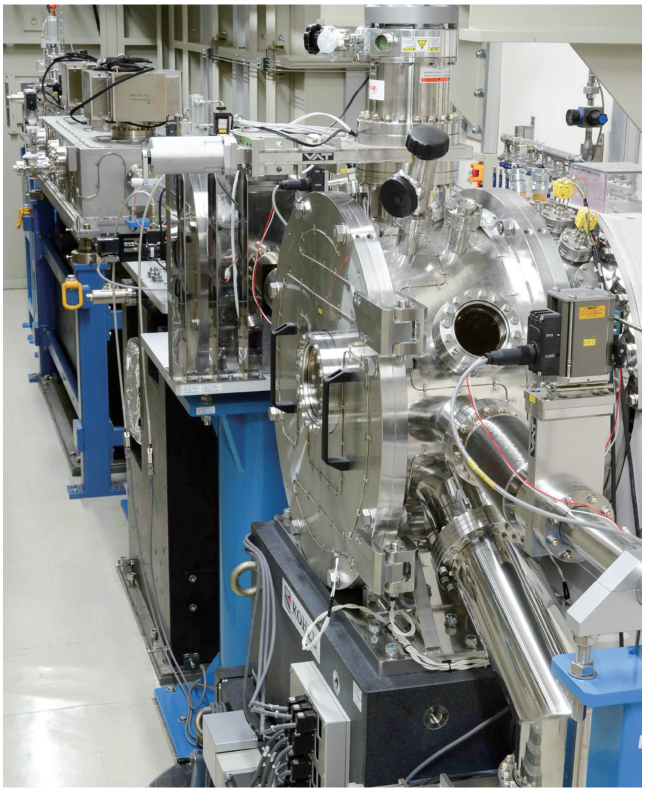

Fig. 1: Devices of the CXS beamline in the optical hutch, including beamline optics, bremsstrahlung shielding and beam diagnosis.

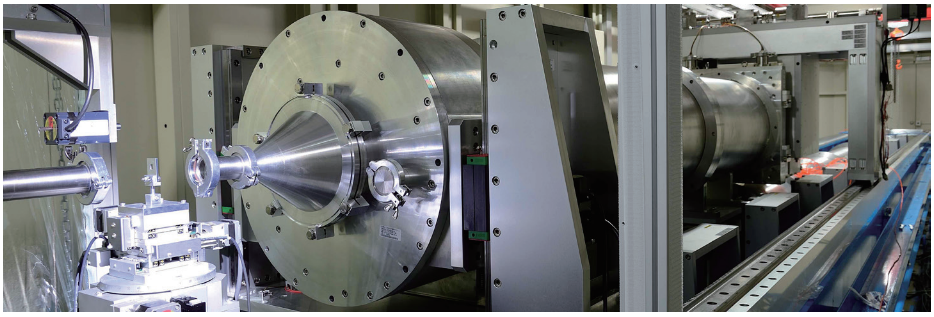

Fig. 2: Sample stage with the scattering vacuum-pipe system and area detector.

provides X-rays of energy range 5.56-20 keV. **Figure 1** shows the double-crystal monochromator (DCM), the first focusing system and some other optical components. The DCM with two sets of crystals can be operated in either a high-flux mode or high-resolution photon-energy mode. The CXS beamline has two focusing systems; the vertical compound reflective lenses (VCRL) and the first horizontal focusing mirror (HFM1) are integrated within the first focusing system. The second focusing system hosts a vertical focusing mirror (VFM), a vertical deflecting mirror (VDM) and a second horizontal focusing mirror (HFM2). In vertical, two optical components, VFM and VCRL, can be switched to achieve two beam sizes, 1 and 10 μm, at a sample position. In horizontal, two-step focusing is employed; the beam size is adjustable on altering the opening of the horizontal secondary source slit (HSS). **Figure 2** shows the sample stage and the scattering vacuum pipe with the detector system. The sample environments are still under development. A sample stage with simple sample holders is currently provided to users. For solution samples, there is a capillary tube holder; for a bulk or thin film, a stand with a rectangular frame can be used for mounting. Besides these two sample holders, a tensile stage is available. A sample holder with temperature control will become available in early 2017. A high-performance liquid chromatograph (HPLC) for bioSAXS users is under commissioning; a flow cell for liquid samples is under design. Various sample environments will be offered to our users in 2017. An important upgrade of the Bragg CDI equipment is planned to be installed during the summer shutdown in 2017.

At present the CXS beamline can be operated with beam size 2 x 10 μm^2 or 9.5 x 9.5 μm^2; the range of photon energy is 5.6-20 keV. The total photon flux at the sample position is 3 x 10^{12} photons/s; the coherent flux is 10^{10} photons/s. As the distance from sample to detector can vary from 2 to 12 m, the *q*-range is 0.0005-0.9 $Å^{-1}$. As a result of the beamtime operation in the second half of year 2016, XPCS, CDI and SAXS have been performed at the CXS beamline.

The first test of a CDI experiment was performed by Chien-Chun Chen, National Sun Yat-sen University; a gold nanoparticle (1 μm) was used to examine the performance of the beamline. The spatial resolution can achieve about 10 nm. The XPCS experiment was tested and performed by Wei-Ren Chen, Oakridge National Laboratory; he used a silica nanoparticle of diameter 100 nm. On varying the ratio of the co-solvent to alter the viscosity of the sample solution and to control the relaxation time of the system, the results showed that the rapid frame rate attains a few milliseconds. The stability of the photon beam was examined with a measurement of glassy carbon, which showed that the beam is stable within more than 2 h. The micro-beam scanning SAXS was also run to determine the orientation of a sample at this beamline.

In conclusion, based on the results of the experiments conducted in the beamtime of 2016B Cycle, the performance of the CXS beamline nearly met the design value. To cope with a variety of the requests of the experimental conditions from the uers, sample environments buildup will be prioritized in 2017. The temperature control sample holder for both liquid and solid samples will be available soon and the HPLC with a flow cell is under construction. The Bragg CDI upgrade will be installed and commissioned in late 2017. (Reported by Jhih-Min Lin)

| References |

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