BL40XU
High Flux

1. Introduction

BL40XU is dedicated to various experiments involving a high flux X-ray beam. It mainly utilizes the fundamental peak of a helical undulator radiation as a quasi-monochromatic X-ray beam without a crystal monochromator. The fundamental undulator radiation has an energy-peak width of 2%, and a flux as high as 1×10^{15} photons/s at 12 keV. Utilizing these characteristics, various experiments such as diffraction, scattering, and imaging are conducted in experimental hutch 1 (EH1), while crystallography and pump-probe experiments are performed in experimental hutch 2 (EH2).

2. EH1

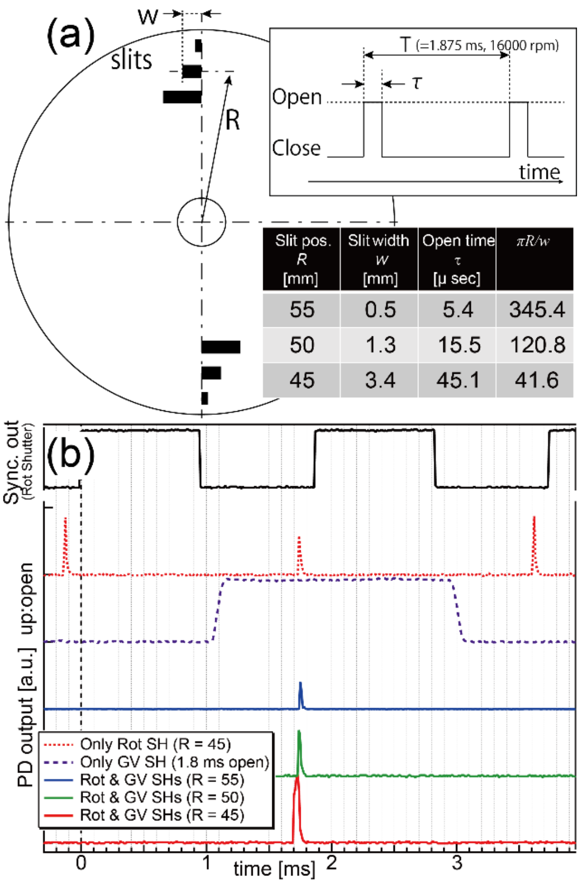
EH1 usually supports time-resolved X-ray diffraction, X-ray single-molecule measurements, and microbeam diffraction/scattering experiments on bio-soft materials. In FY2019, due to the strong demand from X-ray fast-imaging users, a microsecond X-ray shutter was constructed. Since the high-brilliance X-rays of BL40XU are used for high-speed measurements, the X-ray exposure time must be limited to prevent radiation damage to the sample. Similarly, the detector side in the case of high-speed X-ray imaging with a scintillator and high-speed/high-sensitive camera requires a limited exposure time. The intense irradiation of a signal with a longer exposure time causes stray charges to accumulate in the pixel memory, inducing signal saturation. This situation necessitated a microsecond shutter.

A microsecond-order X-ray shutter system was assembled and evaluated using an X-ray rotary (Rot) shutter and a Galvano-type (GV) high-speed X-ray shutter. The system realizes an X-ray cropping of 20–30 μs. A Rot shutter, which is permanently installed at BL40XU, is used, and a GV shutter is located downstream of the Rot shutter. These shutters are synchronized based on the rotation sync signal by the preset scaler (N-TM 105a, Tcnland) and a digital delay generator (DG645/DG535, Stanford Research System).

Figure 1(a) shows the specifications of the Rot shutter, which typically operates with a rotating speed of 16,000 rpm. The Rot shutter can extract X-ray in pulses of 5.5 μs (R=55 mm), 15 μs (R=50 mm), or 45 μs (R=45 mm), depending on the shutter position of the X-ray beam. The GV shutter is made of tantalum (1-mm thick, 4-mm wide, and 4-mm high) and is rotated by 15 degrees on a GV scanner.

Figure 1(b) shows the extracted X-ray signals by the Rot and GV shutters as a function of time. The shutter was tested in operation mode A (equally spaced 203 bunches) and the X-ray signal was detected by a PIN photodiode, which is located downstream of the GV shutter. The solid black line and the red dashed line are the synchronization signal of the Rot shutter (Sync Out) and the X-ray intensity signal (only Rot shutter) when the 45 μs (R=45 mm) Rot shutter was used, respectively. The purple dashed line shows the X-ray intensity signal for the 1.8-ms open GV shutter. About 200 μs were required to open and close the GV shutter, and a millisecond cutout was sufficient with the GV shutter. The solid blue, green, and red lines show the results of X-ray cutouts of 5.5 μs (R=55 mm), 15 μs (R=50 mm), and 45 μs (R=45 mm) in combination with Rot and GV shutters, respectively. Based on the synchronized signal of the Rot shutter, adjusting the opening time of the GV shutter extracted a microsecond-order X-ray.

Since BL40XU mainly uses quasi-monochromatic X-rays, the Rot shutter is used mostly to reduce the X-ray intensity without changing the incident X-ray energy profile. However, it is also useful for the microsecond-order X-ray shutter, which can be utilized in high-speed imaging with a high frame-rate camera (e.g., 10^7 fps).



Slit pos. R [mm]	Slit width w [mm]	Open time τ [μ sec]	$\pi R/w$
55	0.5	5.4	345.4
50	1.3	15.5	120.8
45	3.4	45.1	41.6

Fig. 1. Microsecond shutter (SH) with rotary (Rot) and Galvano (GV)-type shutters. (a) Specifications of Rot SH. (b) X-ray exposure signals from only Rot SH (dashed red line), only GV SH (dashed purple line), and Rot & GV SHs (solid lines).

3. EH2

EH2 supports single-crystal X-ray diffraction. Diffraction mapping using a focused beam and time-resolved X-ray imaging experiments are performed. In FY2013, an optical-trap sample holder was developed for single nanometer-sized particle measurements.

It is well known that structural parameters of nanometer-sized particles are affected by crystallite size and mechanical contact. However, in traditional powder diffraction, the structural parameters of nanometer-sized particles are the average values for the whole particle assemblage, which has a certain crystallite size distribution. To investigate the structural properties of a nanometer-sized particle, it is important to determine the crystal structure and crystallite size of a single particle simultaneously. Additionally, a contactless sample holder should realize precise structural measurements of a small particle without the extra strain from the sample holder.

In FY2019, a single-beam optical-trap sample holder for X-ray diffraction measurements was developed as a contactless sample holder [1]. A TEM_{00}-mode Gaussian beam emitted from an optically pumped semiconductor laser (532 nm, 1W) is focused with a non-spherical lens with a numerical aperture of 0.5 and a focal length of 8 mm. A sample particle with a high refractive index is levitated and trapped by an optical gradient force at the focal point of the laser in air without mechanical contact. The maximum particle size that our optical trap can manipulate is up to almost 500 nm.

Combining the optical-trap sample holder with focused synchrotron radiation realizes X-ray diffraction measurements of a single nanometer-sized particle. Figures 2(a) and (b) show a schematic diagram of the optical-trap sample holder and a photograph of the sample cell. In Fig. 2(b), a non-spherical lens is attached to the side face of the sample cell. The optical-trap sample holder is mounted on XYZ positioning stages and aligned by the stages to maintain the overlap between the levitating sample and the focused synchrotron radiation during a diffraction measurement.

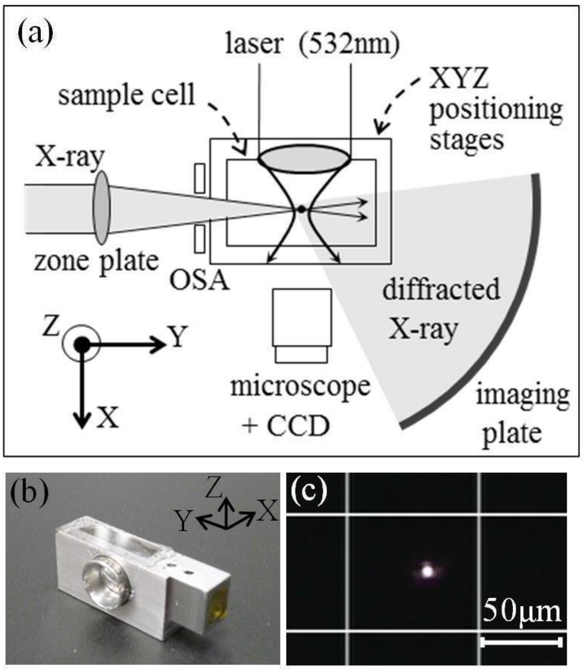


Fig. 2. (a) Experimental setup of the single-beam optical-trap sample holder and the X-ray diffractometer. (b) Photograph of the sample cell used for the optical trap. (c) Photograph of the single ZnO particle during levitation.

To evaluate the optical-trap sample holder, X-ray diffraction images of a single ZnO particle were obtained. The particle was a standard reference material, ZnO (NIST Standard Reference Material 674b) with a crystallite size of 201.4 ± 2.5 nm. In the optical-trap sample holder, the position jitter of the single ZnO particle in the X, Y, and Z directions were 0.56, 0.42, and 0.15 μm, respectively.

Figure 2(c) shows a photograph of the single ZnO particle in the optical-trap sample holder. The particle looks larger than its true size due to the blooming effect of the CCD camera. The beam sizes of the focused synchrotron radiation (15 keV) at the focal point were 3.0 μm in the X direction and 1.5 μm in the Z direction. The photon flux density was 3×10^9 photons/s/mm^2.

Fig. 3. X-ray diffraction image of the single ZnO particle.

Figure 3 shows the diffraction image of the single ZnO particle held in the single-beam optical. The image shows a Debye ring pattern, which is similar to the powder diffraction pattern of the assemblage of ZnO particles. The Debye ring pattern was mainly attributed to the irregular rotation of the particle. Unfortunately, the Debye ring pattern of the single ZnO particle had an azimuthally inhomogeneous intensity distribution, which made it difficult to apply the Rietveld method to the diffraction pattern of the single ZnO particle. The lattice parameters were determined to be a = 3.2505 ± 0.0005 Å and c = 5.207 ± 0.006 Å using the diffraction angles and Bragg's equation. The crystallite size of the single ZnO particle was determined by the Scherrer method to be 193.4 ± 26.2 nm, which is consistent with the crystallite size of the assemblage of ZnO particles (NIST 674b).

Further developments are necessary for the device to be user controllable. The single-beam optical-trap sample holder will be available for user experiments in 2021A.

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Reference:

[1] Fukuyama, Y. et al. (2020). *J. Synchrotron Rad.*, 27, 67–74 .