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Fast tomography projection x-ray microscopy and transmission x-ray microscopy beamline at TPS of NSRRC

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Fast Tomography Projection X-ray Microscopy and Transmission X-ray Microscopy Beamline at TPS of NSRRC

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Abstract. TPS 31A fast tomography and energy-resolved elemental mapping projection X-ray microscopy (PXM) and transmission X-ray microscopy (TXM) beamline of NSRRC is under construction and commissioning. This beamline will provide four operating modes including (1) PXM with white light mode, (2) PXM with double multilayer monochromator (DMM) mode, (3) PXM with double crystal monochromator (DCM) mode, and (4) TXM with double crystal monochromator (DCM) mode. All modes are capable of absorption contrast and phase contrast of images. The spatial resolution of three PXM modes is 0.5 μm. The field of view of PXM-white light, PXM-DMM, and PXM-DCM mode is 25×18 cm^2, 25×10 mm^2 and 25×3 mm^2, respectively. The temporal resolution of PXM modes can approach 1ms/frame and 45 sec/tomography. The *in operando* tomography and energy-resolved elemental mapping within 2D image as well as tomography are feasible. The spatial resolution and field of view of TXM mode is expected as 30 nm and 30 μm, respectively. The energy-resolved elemental mapping within 2D image and tomography is practical.

INTRODUCTION

Many scientific researches, such as energy storage, environmental science, medical biology, advanced materials, geology, and paleontology desire for the techniques of *in operando* tomography or/and elemental mapping microscopy to understand the mechanism of internal structure evolution of the specimen. Projection X-ray microscopy (PXM) and transmission X-ray microscopy (TXM) with absorption contrast and phase contrast are distinguished techniques for exploring the internal micron, sub-micron and nanometer morphological structure of the experimental samples. Utilizing synchrotron radiation wiggler hard X-ray source, PXM provides the vital capability of high flux for *in operando* tomography to investigate structure evolution under specific experimental environments, such as electro-magnetic field, electric current and voltage, temperature, pressure, stress, etc. The tunable X-ray energy with the range of 5 − 50 keV for PXM and 5 − 12 keV for TXM enables the energy resolved elemental mapping and nondestructive 3-dimentional (3D) tomography. Moreover, the aqueous specimen is feasible as a result of no vacuum experiment environment is required.

DESIGN AND EXPECTED PERFORMANCE OF BEAMLINE AND END-STATION

The high speed PXM tomography with temporal resolution of 0.7 − 10 ms/tomography and with spatial resolution of 10 − 38 μm have been recorded [1-3]. The high spatial resolution of TXM is able to reach 30 nm or below [4-6]. TPS 31A PXM-TXM beamline adopting wiggler source at Taiwan Photon Source (TPS) of National Synchrotron Radiation Research Center (NSRRC) is under construction for satisfying the extensive need of *in operando* tomography and energy-resolved elemental mapping. Wiggler W100 is chosen for PXM-TXM beamline, because its flux is higher than bending magnet with about 100 times at 50 keV photon energy. The energy range of 5 to 50 keV is

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designed for this beamline. The parameters of the TPS31A wiggler source W100 are summarized in Table 1.The magnetic field is 1.81 Tesla. The stored current is 500 mA. The period length is 100 mm. The Number of period is 4, and the total length of magnet is 400 mm. The critical energy is 10.8 keV. The power radiated is 0.53 kW/mrad. The total radiation power is 3.7 kW. The horizontal divergence angle can be distributed mainly within 6 mrad. The brilliance and photon flux is 4.9×10^{17}–5.8×10^{16} photons sec^{-1} $mrad^{-2}$ mm^{-2} 0.1%BW^{-1} 0.5A^{-1} and 2.6×10^{14}– 8.9×10^{12} photons sec^{-1} $mrad^{-1}$ 0.1%BW^{-1} 0.5A^{-1} for the energy range of 5 – 50 keV, respectively.

TABLE 1. Parameters of the TPS machine and wiggler W100

Photon energy (keV)	1−300
Current (mA)	500
Peak field strength (T)	1.81
Period length (mm)	100
Number of period (Number of pole)	4 (8)
Total magnetic length, L (mm)	400
Pole gap (mm)	14
Deflection parameter, k	16.91
Critical energy (keV)	10.8
Radiation power (kW/mrad)	0.53
Total radiation power (kW)	3.7

This beamline is designed to collect source horizontal divergence angle of 0.5 mrad which is limited by the thermal tolerance of the diamond filter. There are four operating modes provided in total, which are described as following. Four beamline operational modes are summarized in Table 2.

The first, PXM with white light mode as shown in Fig. 1 provides high photon flux of 1.8×10^{18} photons sec^{-1} 0.5$mrad^{-1}$ 0.5A^{-1} which is integrated from 5 keV to 300 keV. The spatial resolution and field of view (FOV) of image is 0.5 μm and 25×18 mm^2, respectively. The temporal resolution of 2-dimensional (2D) image and fast tomography can approach 1 ms/frame and 45 s/tomography, respectively. The phase contrast can be obtained by direct projection or phase retrieval methods including propagation method and grating interferometer method. A photon flux attenuator is equipped for the radiation-dosage-intolerance specimen.

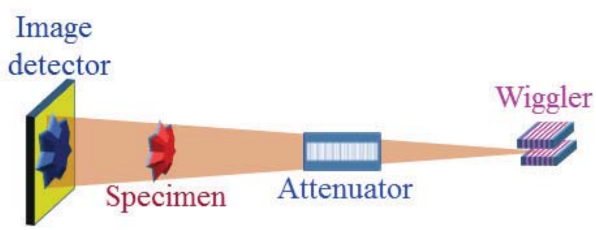


FIGURE 1. Layout of PXM with white light mode of TPS 31A PXM-TXM beamline.

The second, PXM with double [W-Si]/Si multilayer monochromator (DMM) mode as shown in Fig. 2 operates within the energy range of 5 – 50 keV. The corresponding photon flux at specimen is 3×10^{12} photons sec^{-1} 0.5$mrad^{-1}$ 0.5%BW^{-1} 0.5A^{-1}– 1.8×10^{15} photons sec^{-1} 0.5$mrad^{-1}$ 5.4%BW^{-1} 0.5A^{-1} with the energy range of 5 – 50 keV and the energy resolution ($\Delta E/E$) of 0.3% – 6.3%. The fast tomography and energy resolved chemical mapping within tomography are feasible. The spatial resolution and FOV of image is 0.5 μm and 25×10 mm^2, respectively. The temporal resolution of 2D image and fast tomography can approach 1 ms/frame and 45 s/tomography, respectively. The phase contrast can be obtained by direct projection or phase retrieval methods including propagation method and grating interferometer method. In order to deal with the high thermal load of wiggler, we analyzed the thermal temperature and slope deviation of the multilayer carefully using the method of top-side cooling [7]. The slope deviation of the multilayer is reduced to less than 17 μrad which doesn't affect the energy resolution of the multilayer.

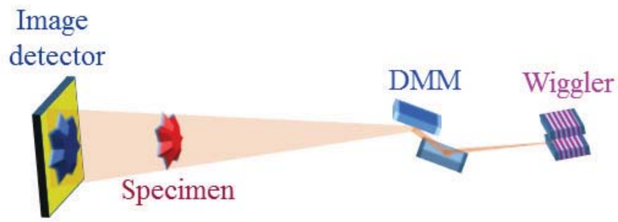


FIGURE 2. Layout of PXM with double multilayer monochromator (DMM) mode of TPS 31A PXM-TXM beamline.

The third, PXM with double Si(111) crystal monochromator (DCM) mode as shown in Fig. 3 operates within the energy range of 5 – 30 keV. One vertical collimating mirror (VCM) is located at the upstream of DCM for optimizing energy resolution. One plane mirror (PM) is located at the downstream of DCM for controlling the beam height at specimen. The corresponding photon flux at specimen is 4.5×10^{11} – 1.9×10^{12} photons sec^{-1} $0.5mrad^{-1}$ $0.013\%BW^{-1}$ $0.5A^{-1}$ with the energy range of 5 – 30 keV and the energy resolution ($\Delta E/E$) of 1.33×10^{-4}. We adopted the method of top-side cooling for VCM to reduce the slope deviation owing to thermal load to less than 0.8 μrad which doesn't influence the energy resolution of Si(111) crystal. The precise energy resolved elemental mapping within 2D image and tomography are practical. The spatial resolution and FOV of image is 0.5 μm and 25×3 cm^2, respectively. The temporal resolution of 2D image and fast tomography can reach 80 ms/frame and 5 min/tomography, respectively. The phase contrast can be obtained by direct projection or phase retrieval methods including propagation method and grating interferometer method. The precise energy resolved elemental mapping within 2D image and tomography is possible.

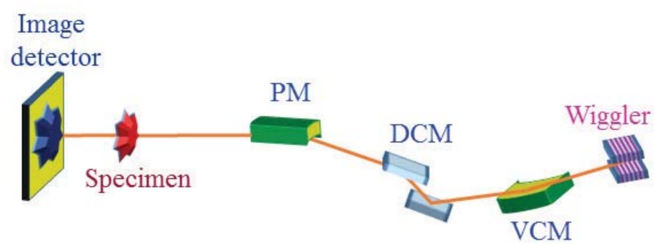


FIGURE 3. Layout of PXM with double crystal monochromator (DCM) mode of TPS 31A PXM-TXM beamline.

The fourth, TXM with double Si(111) crystal monochromator (DCM) mode as shown in Fig. 4 operates within the energy range of 5 – 12 keV. One vertical collimating mirror (VCM) is adopted at the upstream of DCM. One horizontal focusing mirror (HFM) and vertical focusing mirror (VFM) are located at the downstream of DCM to provide a secondary source with 160 μm×162 μm (FWHM) beam size for the condenser. The corresponding photon flux at focus of HFM and VFM is 7.7×10^{10} – 2.9×10^{11} photons sec^{-1} $0.5mrad^{-1}$ $0.013\%BW^{-1}$ $0.5A^{-1}$ with the energy range of 5 – 12 keV and the energy resolution ($\Delta E/E$) of 1.33×10^{-4}. One condenser is located at the downstream of VFM for refocusing the photon beam at the sample position. One zone plate set is adopted for providing excellent spatial resolution at various photon energy. One phase ring is equipped to supply phase contrast of images. The best spatial resolution and largest FOV of 2D image is designed as 30 nm and 30 μm, respectively. The temporal resolution of 2D image is 10 s/frame. The precise energy resolved elemental mapping within 2D image and tomography is feasible.

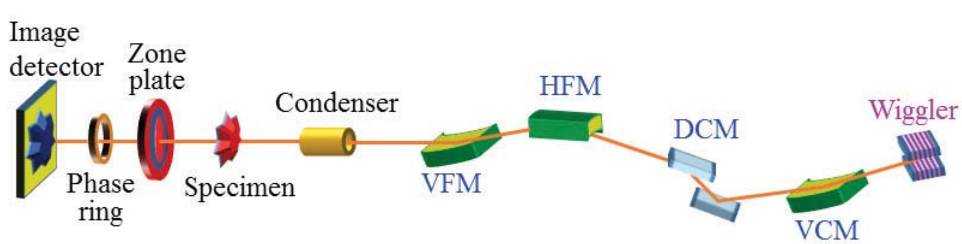


FIGURE 4. Layout of TXM with double crystal monochromator (DCM) mode of TPS 31A PXM-TXM beamline.

TABLE 2. Beamline operational modes

	PXM-white light	PXM-DMM	PXM-DCM	TXM-DCM
Energy range (keV)	5−300	5−50	5−30	5−12
Image spatial resolution	0.5 μm	0.5 μm	0.5 μm	30 nm
Image FOV	25×18 mm^2	25×10 mm^2	25×3 mm^2	30 μm
Image temporal resolution	1 ms/frame 45 sec/tomo.	1 ms/frame 45 sec/tomo.	80 ms/frame 5 min/tomo.	10 s/frame 2 hour/tomo.
Energy resolution (ΔE/E)	none	0.3%−6.5%	1.33×10^{-4}	1.33×10^{-4}
Optical component	attenuator	DMM	VCM, DCM, PM	VCM, DCM, HFM, VFM
End-station component	scintillator objective lenses CMOS detector	scintillator objective lenses CMOS detector	scintillator objective lenses CMOS detector	condenser, zone plates phase ring, scintillator, objective lenses CMOS detector

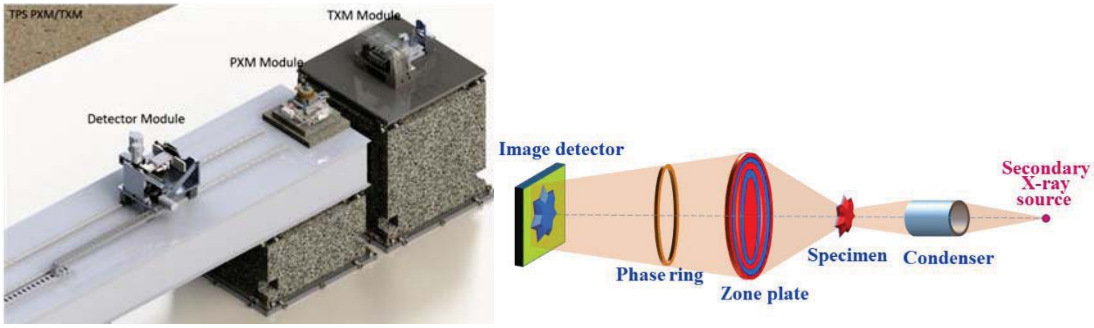
The end-station of this beamline is designed as three main parts including PXM module, TXM module and detector module as shown in Fig. 5. This PXM is aimed for direct projection, there will be no optics in this module, and the resolution depends mainly on the resolution of scintillator, which will be introduce in the detector module. The PXM module is consist of a high speed rotation stage and two linear stages. On the rotation stage, there will be a three axes positioner for the sample position adjustment. A laser interferometer is for high speed positioning and also to synchronize the position, the shutter and the detector for 2D imaging. On the top of the rotation stage, there are a three axes positioner for the sample positioning.

The TXM is a Fresnel zone plate based with capillary as the condenser. The designed energy range is from 5 keV-12 keV since the best optical efficiency of zone plate. The reasons to use the capillary as the condenser is high flux and achromatic. The TXM module includes a stopper, condenser, a zone plate set for operating different photon energy, and a phase ring for fulfilling the phase contrast of image. There can be a pinhole between condenser and sample stage if the direct beam is not completed blocked. This microscope, designed with better than 30 nm resolution, with working distance large than 3 cm at the energy of 5 keV. At the primary stage, two zone plates will be used for different performance. The zone plate specification is listed in Table 3. The distance from zone plate to detector is from 1 meter to 4.6 meters. The sample stage is consisted of a coarse stage with 3 axes in the bottom. A rotation stage on it and a three 3 axes flexure stages on the rotation stage. This stage is aimed for a high stable rotation with the sphere of confusion (SOC) of 30 nm. In this case, the 2D image from this TXM will not need the software to align.

TABLE 3. Zone plate specification of TXM module

	Zone plate 1	Zone plate 2
Outer most zone width (nm)	25	40
Center energy (keV)	8	10
Largest FOV (μm)	25	30
Number of zones	3000	1000
Focal length at 8 keV(mm)	46.5	51.6
Thick of zone (nm)	>550	>800
Outer diameter (μm)	300	160

The detector module is mainly consisted of a scintillator, three axes stages, high speed area detector, and optical objectives, which are 2×, 5×, 10×, 20×, and 50×. It is planned to be used for both TXM and PXM module. For TXM, a 20× objective is used for the X-ray magnification about 50×, which is 1000× for the detector pixel size of around 10 μm. The effective pixel size is around 10 nm, which gives a good enough over sampling for a 30 nm resolution system. For PXM system, the optical objective can be from 100× for the highest resolution, or select the other magnification for large FOV. The detector is a high speed Complementary Metal-Oxide-Semiconductor (CMOS) detector, which can be as high as 7000 frames/sec. The data rate is 8 GBytes/min. A NAC-ACS1 M60 detector with ultra-fast frame rate of 60,000 frames/sec is utilized for ultra-fast PXM. The data rate is 256 GBytes/min. The fast data stream from the detector to the storage will be also implemented.



(a) (b)

FIGURE 5. (a) Sketch of the end-station of TPS 31A PXM-TXM beamline. (b) Sketch of TXM components.

CONCLUSIONS

TPS 31A fast tomography and energy-resolved elemental mapping projection X-ray microscopy (PXM) and transmission X-ray microscopy (TXM) beamline is under construction. The construction of three PXM modes is finished and under commissioning. Whereas the TXM mode will be ready at the middle of 2023. The excellent temporal resolution of PXM with 1 ms/frame and 45 sec/tomography for *in operando* tomography as well as energy-resolved elemental mapping of PXM and good spatial resolution of TXM with 30 nm will provide new insight for academic disciplines and industrial frontiers.

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