The 3D NanoimagingBeamline at SSRF

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Abstract

Full-field transmission X-ray microscopy (TXM) is a powerful nondestructive three-dimensional (3D) imaging method with a nanoscale spatial resolution that has been used in most synchrotron facilities worldwide. An in-house-designed TXM system was constructed at the BL18B 3D Nanoimaging beamline at the Shanghai Synchrotron Radiation Facility. The beamline operates from 5 to 14 keV and enables 20 nm spatial resolution imaging. The characterization details of the beamline are described in this paper. The performances in terms of spatial resolution, nano-CT, and nano-spectral imaging of the TXM beamline are also presented in this article.

Full Text

Preamble

The 3D Nanoimaging Beamline at SSRF

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Full-field transmission X-ray microscopy (TXM) is a powerful nondestructive three-dimensional (3D) imaging method with nanoscale spatial resolution that has been implemented at most synchrotron facilities worldwide. An in-house-designed TXM system was constructed at the BL18B 3D Nanoimaging beamline at the Shanghai Synchrotron Radiation Facility. The beamline operates from 5

to 14 keV and enables imaging with 20 nm spatial resolution. This paper describes the characterization details of the beamline and presents its performance in terms of spatial resolution, nano-CT, and nano-spectral imaging.

Keywords: Transmission X-ray microscopy (TXM), Nano-CT, Synchrotron Radiation

I. Introduction

Full-field transmission X-ray microscopy (TXM) is one of the most powerful tools for observing the internal structures of objects at the nanometer scale. Utilizing the high throughput and energy tunability of synchrotron X-rays, TXM offers unique advantages such as high penetration depth and spatial resolution, making it superior to other imaging techniques (Scanning Electron Microscopy, Atomic Force Microscopy, etc.) for testing thick samples [1-3].

To provide advanced nanostructure characterization and research methods for users in materials science, energy, environmental science, and other fields, the Shanghai Synchrotron Radiation Facility (SSRF) planned to design and construct a TXM device. Table I shows the design objectives of this beamline. In December 2021, the X-ray 3D Nanoimaging beamline (BL18B) was completed.

Based on TXM, this beamline provides complementary and unique capabilities to current microscopy research methods for studying crucial scientific problems, such as structural and property changes of lithium battery materials during charge-discharge cycles and the relationship between micro- and nanoscale pores and shale gas reservoirs [4-14]. The beamline covers an energy range of 5 keV–14 keV and achieves a spatial resolution of 20 nm. The main research methods include TXM, nano-CT, and nano-spectral imaging. This paper presents the characterization and experimental methods, along with some initial operational results.

Table I. The design goals of BL18B

Parameter	Specification
Energy range (keV)	5–14
Energy resolution (ΔE/E)	2$×10^{-4}$
Flux at sample (8 keV @ 300 mA)	1.3$×10^{10}$ phs/s
Spatial resolution (TXM @ 8 keV)	20 nm

II. The Beamline

The overall design of the beamline was formulated according to the scientific goals and user requirements. Key considerations include photon flux, energy resolution, and phase-space matching.

Photon flux is a critical factor determining data quality and time resolution in imaging experiments. Therefore, maximizing photon flux at the sample while ensuring adequate energy resolution and phase-space matching is the fundamental principle of beamline design. Considering the insertion device resources at SSRF and the phase-space requirements for TXM, a bending magnet was selected for this beamline. With the light source, monochromator crystals, and condensers selected, the primary consideration becomes the transmission efficiency of the beamline optics.

The main factors affecting photon flux include the beamline's receiving angle, mirror surface coatings, beam incidence angles on the mirrors, and the condenser's reception efficiency.

Zone plate (ZP) imaging systems require monochromatic incident light sources satisfying $\Delta\lambda/\lambda \leq 1/N$ [15], where $\Delta\lambda$ is the illumination radiation spectral width, λ is the incident X-ray wavelength, and N is the zone number. Spectral imaging technology combining TXM and XANES requires an energy resolution higher than 2$×10^{-4}$. The main factors affecting energy resolution include the Darwin width of the double-crystal monochromator (DCM) crystal, the vertical divergence angle of the incident beam on the first DCM crystal, and heating deformation of the first DCM crystal. Additionally, slope errors for the mirrors and DCM must be minimized.

According to the characteristics of beam propagation in ZP imaging microscopes, the overall optical path was divided into two segments. The first segment is the beamline system from the light source to the secondary light source, where horizontal and vertical phase spaces propagate independently and approximately conserve. The second segment is the ZP imaging system from the secondary light source to the detector, which is an axisymmetric optical system where phase space is approximately conserved within a radius perpendicular to the optical axis. An important task in optical design is to assemble the beam into the desired shape according to phase-space conservation principles and deliver it to the sample.

1) Light Source

BL18B is a bending magnet (BM) beamline with a magnetic field strength of 1.27 T. To ensure that the X-ray beam reaching the TXM end station has the desired width and angle in both horizontal and vertical planes, the angular acceptance of this beamline is set to 0.4 mrad × 0.22 mrad. Table II lists the specifications of the BL18B light source. Fig. 1 [Figure 1: see original paper] shows the brilliance and flux curves of the SSRF BM sources. The estimated source brilliance is 6.48$×10^{15}$ phs/s/0.1%bw/mm^2/mrad^2 at 8 keV, with a total flux of 1.19$×10^{13}$ phs/s/0.1%bw.

TABLE II. Specifications of BL18B Light Source

Parameter	Specification
Source type	Bending-magnet
Energy	3.5 GeV
Magnetic field	1.27 T
Current	300 mA

2) Beamline

Fig. 2 [Figure 2: see original paper] shows the overall layout of the beamline, which includes two major hutches: an optical hutch and an experimental hutch. The beamline houses a collimating mirror (CM), DCM, and toroidal mirror (TM) for pre-focusing. The CM is located 20.2 m downstream of the source. After the CM, the Si(111) DCM is placed 23 m downstream of the source. Following the DCM, the vertically collimated beam passes through the TM, located 26 m from the source, and is then focused onto a slit located 39 m from the source. This slit, positioned at the experimental station, serves as the secondary source aperture (SSA).

A. Mirrors The vertical CM is the first optical component of the beamline. It receives white-beam radiation from the source and provides parallel illumination in the vertical plane for the DCM. It serves three functions: reducing the incident beam power to the DCM, rejecting higher-order harmonics from the BM spectrum, and collimating incident X-rays well below the Darwin width of the Si(111) DCM. The CM is realized by bending a plane mirror, with the incident beam collimated and reflected upward. To ensure adequate power reduction to the DCM and effective harmonic rejection for X-ray microscopy experiments, an Rh coating was used for the CM reflective surface. Water cooling is sufficient to solve the power reduction problem. The CM operates at a fixed 4.4-mrad incidence angle, leading to a cut-off energy of approximately 15 keV. Based on the vertical receiving angle of the light source (0.22 mrad) and 4.4 mrad grazing incidence angle, the optically active length of the CM is 1000 mm. To minimize diffusion of the focusing beam at the SSA and maintain mirror reflectivity above 95%, the surface roughness of the CM should be better than 3 Å. The root mean square (RMS) slope error of the CM must be less than 0.8 rad along the meridional direction (including mechanical error of 0.6 rad and gravity/deformation error of 0.2 rad), and the RMS slope error along the arc direction must be less than 5 rad.

The TM is realized by gently bending a cylindrical mirror to the required shape. It collects a monochromatic beam and delivers both horizontal and vertical foci to the SSA. The beams received by the TM are parallel in the vertical direction and divergent in the horizontal direction. Therefore, the incident beam is reflected downward and focused to 100 m (H) × 290 m (V) (full width at half maximum @ 8 keV) at the SSA position. As the TM is placed behind the DCM, it has no cooling requirements. The coating, grazing incidence angle,

surface roughness, RMS slope error, and optically active length of the TM are identical to those of the CM.

B. Double-Crystal Monochromator The DCM is placed horizontally between the two mirrors. The beam is collimated in the vertical plane before and after passing through the monochromator. Two major technical concerns for the DCM are achieving an angular stability of the diffracted beam of 0.5 rad or higher and minimizing thermally induced slope error on the first crystal. The DCM is equipped with standard Si(111) double-plane crystals to provide adjustable energy within the range of 4–15 keV and a high energy resolution better than 2$×10^{-4}$, which meets the requirements of the ZP objective in TXM. The DCM is a domestic monochromator adopting a common design used at SSRF. The two crystals have an (n–n) achromatic arrangement. During energy scanning, the position and direction of the outgoing beam are maintained constant, with a height offset between the incident and outgoing beams of 25 mm. The main axis of Bragg rotation is located in the first crystal. As the DCM performs Bragg rotation, the second crystal moves in the direction normal to the crystal surface [16]. Thermal deformation of the first monochromator crystal reduces flux, energy resolution, beam size at the SSA, and matching efficiency of the condenser in the TXM end station. The thermally induced RMS slope errors along the arc and meridional directions must be less than 5 rad. According to calculations, the power absorbed on the first crystal can be comfortably managed by water cooling.

III. TXM End Station

The experimental hutch houses the SSA, beam intensity detector system, and TXM system. The beam detector system comprises two ion chambers located downstream of the SSA to monitor incident flux. Fig. 3 [Figure 3: see original paper] shows a photograph of the TXM system comprising a monocapillary condenser, pinhole, objective ZP, sample station, and two X-ray imaging detectors.

The condenser, located approximately 2 m from the SSA, is a single-bounce ellipsoidal glass monocapillary. BL18B adopts an in-house-designed monocapillary as condenser [17,18]. The monocapillary condenser provides hollow-cone illumination to match the numerical aperture of the ZP, with a beam stop attached to its upstream end. For beamlines at third-generation synchrotron radiation sources, the beam emittance in the vertical plane is typically lower than the required microscope acceptance. This mismatch causes some sample areas to be under-illuminated or the beam angle to be insufficient to fill the objective lens. To overcome this problem, a shaker was installed under the condenser chamber to perform fast scanning and expand the illumination angle of the sample to match the numerical aperture of the ZP.

A pinhole upstream of the sample blocks direct light not focused by the monocapillary condenser. It is positioned on a motorized stage between the condenser

exit and sample. Pinholes with diameters of 100 m and 150 m are mounted on a single holder.

The sample stage comprises a high-precision xyz motor and an air-bearing rotation stage. The sample holder is placed on a breadboard mounted on the sample stage.

The microscope objective ZP produces a magnified image of the sample. The image resolution is determined by the outermost zone width of the ZP. For TXM imaging at BL18B, two sets of ZPs are used with outermost zone widths of 25/40 nm, diameters of 125/150 µm, and Ir thickness >0.7/0.9 µm (XRnanotech, Switzerland).

BL18B has two X-ray imaging detectors for high- and medium-resolution imaging. The high-resolution detector is a scintillator-coupled optical microscope with 2×, 4×, and 10× optical magnification lenses and a water-cooled 16-bit $2048\$\times 2048 camera(Zyla 4.2 PLUS sCMOS, Andor, Oxford Instruments, UK) with 6.5- \mu m square pixels. It is installed on a linear track with a travel range of 1.3 meters along the beam direction, providing$ pixels and 6.5 m/pixel (12849-1111U, HAMAMATSU, Japan) is fixed at the back end of the hutch. The two detectors can be switched according to experimental requirements.

An inline telescope is placed on the optical platform for sample positioning. Vacuum tubes are placed between the ZP and detector to minimize X-ray attenuation in air.

Each component of the TXM system is mounted on a highly stable marble optical platform with fixed relative positions. To minimize effects from acoustic vibrations, airflow, and temperature changes, a Plexiglas enclosure is placed on the platform. The enclosure has sliding doors to facilitate operations such as sample changing.

1) Imaging Technologies

The primary imaging technologies at this beamline include TXM, nano-CT, and nano-spectral imaging. Nano-CT is a non-destructive imaging protocol used to generate high-resolution 3D images comprising two-dimensional (2D) projections or "slices" of micrometer-scale samples.

Nano-spectral imaging combines TXM with X-ray absorption near-edge structure (TXM-XANES) to detect 2D and 3D chemical phase transformations in mesoscale volumes at the nanoscale [19,20]. Notably, combining CT with spectral imaging enables joint characterization of sample morphology and chemical states, facilitating better study of material properties.

A. TXM Fig. 4 [Figure 4: see original paper] shows the schematic of a TXM. A monocapillary condenser delivers a focused beam onto an object (sample). A pinhole cleans the beam and blocks direct light not focused by the capillary.

A ZP magnifies and images the sample onto a detector. TXM with nanoscale resolution can achieve a micron-level field of view with nanoscale resolution.

Based on the two detectors, the BL18B beamline TXM method has two resolution modes—high- and medium-resolution modes—enabling sub-20 nm and sub-70 nm spatial resolution [21].

B. Nano-CT Nano-CT is a valuable method for studying the fine structure of materials. Unlike micron-scale resolution experiments, nanoscale-resolved TXM samples are smaller and require more careful preparation. For ZP-based TXM CT imaging, the sample thickness should be less than the depth of focus (DOF), defined by DOF = $\pm\lambda/2(\mathrm{NA})^2$ = \$±2Δr^{2}/ \$, where λ is the incident X-ray wavelength, NA is the ZP lens numerical aperture, and Δr is the outermost zone width of the ZP [22]. When a portion of the sample lies outside the DOF, the ZP cannot focus that out-of-range portion.

For BL18B, suitably sized powder samples were gently blown onto stretched glass capillaries. Bulk samples were cut to appropriate size and firmly fixed on the tip of a needle using focused ion beam (FIB) or other fine sample preparation methods. Since sample sizes are in the micron range, an offline light microscope was used for rough examination of sample preparation. Fig. 5 [Figure 5: see original paper] shows a photograph of gold particles in a stretched glass capillary and their 2D projection. The capillary tube or needle was firmly clamped to the center of the sample holder and remained stable during CT experiments.

BL18B CT results were processed using a completely in-house-designed automatic projection alignment algorithm [23] and in-house reconstruction software, AduRecon. This automatic projection alignment algorithm is based on generated feature maps and improves the fidelity of 3D nano-CT reconstruction models. The reconstruction software incorporates several technologies for nanoimaging data processing, such as data jitter calibration, phase recovery, and ring artifact cancellation, providing strong guarantees for obtaining accurate experimental data.

C. Nano-Spectral Imaging Nano-spectral imaging can be classified into dual-energy and TXM-XANES imaging, as illustrated in Fig. 6 [Figure 6: see original paper]. Dual-energy imaging records projection images at two different energy points: before and after the X-ray absorption edge of the target element. The target element shows a significantly higher absorption edge jump than other elements in the absorption edge energy region. After differential calculation of the two images, the enhanced portion displays the distribution of the target element in the sample. Dual-energy imaging combined with CT technology can obtain the 3D distribution of the target element in a sample.

In a typical XANES experiment, the sample is placed between two ion chambers that record beam intensity during monochromator energy scanning. The absorption coefficients of target elements are obtained by recording photon counts

in both ion chambers. Accordingly, during XANES imaging, the imaging detector records the number of photons not passing through the sample. When the monochromator performs energy scanning, it records a stack of images to calculate the absorption coefficient of the target element.

To extract the absorption spectrum, the stack image undergoes several processing steps. The main processing steps for 2D XANES imaging are: (1) averaging projection and reference images to reduce vibration impact and increase signal-to-noise ratio; (2) reference correction of projection images; and (3) magnification correction and alignment of lower-energy projection images. The focal length of the ZP varies linearly with X-ray energy. During experiments, the ZP must move continuously to maintain focus at different energies. Projected images at different magnifications are recorded using the detector, so image sizes must match for subtraction of difference maps.

After image processing, an absorption spectrum is generated for each pixel within the region of interest (ROI). After normalizing the spectra, the position of the absorption edge energy of the target element is determined. The offset of the normalized absorption spectrum characterizes relative valence state changes of the elements.

XANES imaging combined with CT technology can obtain 3D valence distribution information of the target element in the sample. BL18B uses the "TXM-Wizard" software package to perform magnification and misalignment corrections, reference corrections, normalization, and other data manipulations on XANES stack data [24,25].

2) Control and Data-Acquisition System

Control and data acquisition for BL18B are performed using EPICS and in-house-designed software [26]. EPICS is a suite of open-source software tools, libraries, and applications providing a standard distributed control system architecture, communication protocol, runtime database, and software tools that support a wide range of I/O devices. The in-house-designed software communicates with the EPICS system, enabling motion control of each motor in the experimental station. Instead of using vendor-supplied software, the self-designed software integrates control of all important beamline equipment, enabling automatic data collection in TXM, CT, spectral imaging, and other experimental modes.

For TXM and dual-energy imaging, the experimental procedure involves imaging at selected energies. For CT experiments, the sample is placed at the center of rotation, and experimental energy is selected according to sample characteristics; images of sample projections are captured at different angles. In XANES experiments, users can set the energy-scanning range and steps in the software interface. As the focal length of the ZP is a function of incident X-ray energy, users must capture two clear images at different energies. The software calculates ZP coordinates corresponding to each energy point based on ZP positions

in the two images, scanning energy range, and required step size. Therefore, during XANES imaging, the ZP moves to keep the sample in focus while the monochromator rotates. Subsequently, the sample is moved in and out to enable acquisition of projections with and without the sample at the current energy. For three-dimensional XANES, the program mode collects CT data at each energy point.

VI. Commissioning Results

A. TXM

A standard Siemens star pattern (Carl Zeiss, Germany) with a smallest feature size of 30 nm was used for TXM testing in high-resolution mode. Fig. 7(a) shows an absorption contrast image captured at 8 keV with an exposure time of 30 s. To quantify TXM spatial resolution, power spectral density (PSD) analysis was performed on the image data. A 2D Fourier transform was applied to the TXM projection image to calculate the PSD. The 2D power spectral intensity was then integrated over the azimuthal direction (along the circle) at fixed radial frequency [27-29]. Fig. 7(b) [Figure 7: see original paper] shows the PSD result. The cut-off frequency is 25 m^{-1}, corresponding to a period of 40 nm, which indicates a spatial resolution of 20 nm [30].

We used a piece of dehydrated rabbit bone to test radiation damage to biological samples at low energy. Fig. 8 [Figure 8: see original paper] shows absorption contrast images of the sample obtained at 5.5 keV, taken one hour apart. Flux at the sample position was measured using an ionization chamber with a pair of 10-mm long electrodes (IC Plus 10; Oxford, UK). The ionization current was measured using a high-sensitivity picoammeter (Keithley 6485; Tektronix, USA). X-ray beam intensities were evaluated using the equation N = I_0/(1 - e^(- x)), where N is beam intensity, I_0 is measured ionization current, $_0$ is gas ionization energy, e is electron charge, E is incident X-ray energy, x is electrode length, and is absorption coefficient of the filling gas. The photon flux at the sample was approximately 1.26$×10^{10}$ photons/s. Comparing the two images, the sample showed no obvious signs of irradiation damage after one hour of exposure.

B. Nano-CT

Ni-rich $LiNi_1$ Co Mn O_2 (NCM) is a commercially available cathode material with advantages such as structural stability, low cost, and good cycling performance [31,32]. Owing to its high nickel content, high performance, and high capacity, $LiNi_{0·6}Co_{0·2}Mn_{0·2}O_2$ (NCM622), a typical high-energy cathode, has emerged as an ideal material for automotive applications [33]. In the nano-CT experiment, a 50×-cycled NCM622 particle was imaged and reconstructed in 3D. Projections were obtained using absorption contrast at 8.6 keV. The complete tomography dataset was collected in approximately 6 min with 180 projections (1 s exposure time per projection). Tomography datasets were reconstructed

using AduRecon software, and 3D reconstruction was performed using Avizo® Fire software.

It has been reported that during long-cycle, high-rate charging and discharging processes, a series of phenomena occur including battery capacity attenuation and performance degradation. Correspondingly, during early stages of charge-discharge processes, microcracks and radial cracking are observed near particle centers. With increasing voltage or cycle time, significant radial cracking inside particles becomes observable [31,32]. Fig. 9 [Figure 9: see original paper] shows a reconstructed 3D image of the NCM622 particle and its slices. The reconstruction results clearly show that the 50×-cycled particle had several cracks inside in an extended state. The CT images had a resolution of 30 nm/pixel.

C. 2D TXM-XANES

XANES imaging is suitable for investigating battery materials as it correlates mesoscale to nanoscale chemical changes within secondary particles of battery electrodes, revealing interactions between nano- and micron-scale factors affecting macroscopic behavior of battery materials [34].

Pristine and 50×-cycled NCM622 particles were used to test the performance of the 2D TXM-XANES method. The target element is Ni, and the experimental energy range is 8.2–8.6 keV. Data were processed to obtain XANES spectra for each pixel in the ROI. Fig. 10 [Figure 10: see original paper] shows pseudocolor 2D XANES chemical phase maps of Ni within the two particles. The distribution of absorption edge energy in pristine particles is relatively homogeneous, whereas for cycled particles, the absorption edge energy is lower in the inner region than in the outer region. The difference in Ni K-edge absorption energy between the two NCM622 particles is obvious. Fig. 10(c) shows Ni K-edge XANES spectra from the central portions of Figs. 10(a) and (b). The difference between absorption edges of the two particles is also evident in the spectral line plot. The TXM-XANES imaging method works well for chemical characterization of battery particles in different charging states.

V. Summary and Conclusion

A new in-house-designed TXM system was constructed for SSRF. This paper presents the characterization of this beamline and commissioning results for TXM, marker-less nano-CT, and TXM-XANES imaging. The beamline will undergo future upgrades; for instance, the X-ray beam position feedback and correction system will be upgraded for more stable spot positioning. Moreover, an angle adjustment motor will be introduced for the ZP to enable better alignment, providing an advanced experimental platform for nanostructure characterization in related disciplines.

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