

X-ray Microscopy Beamlines at SSRF - Present Status and Future Plan

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Abstract. The Shanghai Synchrotron Radiation Facility (SSRF) is a 3.5-GeV third-generation light source. The facility has been open for user experiments since May, 2009. This high-brightness x-ray source is an ideal platform for x-ray microscopy. Presently, SSRF has three beamlines related to x-ray microscopy or imaging, namely the soft x-ray spectromicroscopy beamline (STXM), the hard x-ray microfocusing beamline, and the x-ray imaging beamline. The construction of SSRF phase-II beamlines will be carried out during 2011-2017. Seven additional beamlines for x-ray microscopy or imaging will be built.

Keywords: Shanghai Synchrotron Radiation Facility, x-ray microscopy

PACS: 07.85.Qe, 68.37.Yz

OVERVIEW OF SSRF

The Shanghai Synchrotron Radiation Facility (SSRF), located in Zhangjiang High Technology Park, Shanghai, is a third-generation light source that provides powerful x-rays for users in China and other parts of the world in various fields of research. SSRF is funded jointly by the Central Government of China, the Chinese Academy of Sciences, and the Municipality Government of Shanghai. It is the biggest of the so-called “Large Science Facilities” in China.

The SSRF accelerator complex consists of a 150-MeV electron linac, a full energy booster, and a 3.5-GeV storage ring. The SSRF storage ring is designed to run at a current up to 300 mA and an emittance of 3.9 nm·rad. With advanced insertion devices, it can provide high-intensity x-rays of 10^{20} photons/(s·mm²·mrad²·0.1%BW) in maximum brilliance. The main design parameters of the storage ring are shown in Table 1.

TABLE 1. Main Design Parameters of the SSRF Storage Ring

Parameters	Specifications
Energy	3.5 GeV
Circumference	432 m
Natural emittance	3.9 nm·rad
Natural energy spread	0.1%
Coupling	1%
Beam current	200-300 mA
Straight sections	16 × 6.5 m, 4 × 12 m
Beam lifetime	>10 hours

Seven beamlines, including five insertion device beamlines and two bending magnet beamlines, were selected as phase-I beamlines; this decision was made based on extensive discussions in the Chinese scientific community. They are expected to facilitate research in a number of fields, with emphasis on structural biology, environmental studies, materials science, nano-science, and biomedical applications. Table 2 shows the phase-I beamlines of SSRF.

TABLE 2. SSRF Phase-I Beamlines

Beamline	Energy Range	Source
Macromolecular crystallography	5-18 keV	In-vacuum undulator, U25
XAFS	4-50 keV	Multipole wiggler, W79
X-ray diffraction	4-30 keV	Bending magnet
Hard x-ray microfocusing	5-20 keV	In-vacuum undulator, U25
X-ray imaging and biomedical applications	8 -70 keV	Multipole wiggler, W140
Soft x-ray spectromicroscopy	200-2000 eV	Elliptical polarized undulator, EPU100
Small-angle x-ray scattering	4-30 keV	Bending magnet

The construction of SSRF started on December 25, 2004, and completed the phase-I project in April, 2009. The SSRF has been fully operational with the seven phase-I beamlines delivering light to users since May 06, 2009. From May 6, 2009 to July 20, 2010, about 1290 proposals were received, 1178 of which have been approved by the review committee; however 3-5 times over-subscribing of beam time had been seen by each beamline. By the end of July, 2010, the seven beamlines have provided more than 30000 hours of beam time to user experiments and served more than 1660 users from 127 organizations (64 universities, 57 institutes, 11 hospitals, and 7 companies) all over China. About 830 experiments have been carried out and about 40 research papers have been published in scientific journals (including *Nature*, *Science*, and *Cell*). Some research results of important significance have already been obtained; for example, the study on the structure of the CED-4 Apoptosome [1] and *in situ* XAFS study of PtFe@SiO₂ catalyst [2].

PRESENT X-RAY MICROSCOPY BEAMLINES AND ACTIVITIES AT SSRF

SSRF is an excellent platform for the development and application of x-ray microscopy. Three of the seven SSRF phase-I beamlines are focused on x-ray microscopy or high-resolution x-ray imaging: the soft x-ray spectromicroscopy beamline (STXM), the hard x-ray microfocusing beamline, and the x-ray imaging beamline. These beamlines are playing important roles in research ranging from materials science, biology, environmental science, biomedical, agriculture, and archaeology to industrial applications. A summary of these beamlines are shown in Table 3.

TABLE 3. Present X-ray Microscopy Beamlines at SSRF

Beamline	Capabilities	Source	Key Specifications
Soft x-ray spectromicroscopy (STXM)	STXM, NEXAFS, TEY	Elliptical polarized undulator	Energy range: 200-2000 eV Spectra resolution: 18000@244 eV Spatial resolution: <30 nm Photon flux: >10 ⁸ photons/s
Hard x-ray microfocusing	μ -XRF mapping, μ -XANES, μ -XRD	In-vacuum undulator	Energy range: 5-20 keV Energy resolution: $\sim 10^{-4}$ Focal spot size: $<2 \times 2 \mu\text{m}^2$ Photon flux: >10 ¹¹ photons/s
X-ray imaging and biomedical applications	PCI, KES imaging XCT	Multipole wiggler	Energy range: 8-72.5 keV Spatial resolution: 0.5-10 μm Photon flux: $\sim 10^{10}$ photons/mm ² Maximum speed: 10 ³ frame/s

The soft x-ray spectromicroscopy beamline was designed to take x-ray images and NEXAFS spectra of samples simultaneously with high spatial resolution (better than 50 nm) and high spectra resolution. An EPU is used at this beamline to produce high brilliant x-ray beam covering an energy range of 200-2000 eV. An SX-700 type monochromator is used to provide highly monochromatic beam and for energy scan. The end station is a STXM. Transmission x-ray mode is used for measuring thin samples, and total electron yield (TEY) can be used for measuring the surface of samples. A spatial resolution better than 30 nm and spectral resolution about 18000 at 244 eV were realized (Fig. 1). This beamline is now heavily used for 2D mapping of samples with high spatial resolution and chemical sensitivity (NEXAFS). For example, Gang Pang's group from the Research Center for Eco-

Environmental Sciences, CAS, has studied the mechanism of enrichment of nanoscale oxygen bubbles on the surface of SiO₂ particles, which had been used successfully to restore the polluted water of Tai Lake. Chunhai Fan's group from the Shanghai Institute of Applied Physics, CAS, has studied the Graphene-templated formation of ultrathin two-dimensional single-crystal lepidocrocite nanosheets. One of the authors, Renzhong Tai, has studied nitrogen compounds in automobile exhaust particles [3], which is one source of environmental pollution, e.g., acid rain. Hui Wang's group from the Institute of Nutritional Sciences, CAS, has studied the antitumor activity and Fe chelation efficiency of TSC by mapping Fe distribution in liver cancer cells. Three-dimensional mapping (nano-CT) and magnetic mapping (XMD) capabilities will be developed on this beamline in the future.

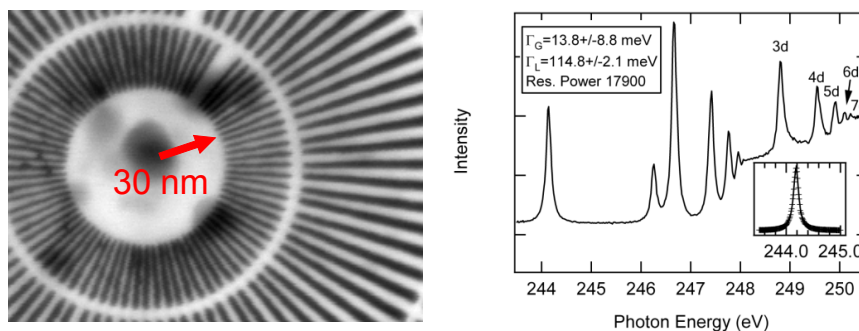


FIGURE 1. Measured STXM image of Siemens star (left) and Argon shell excitation spectrum (right).

The hard x-ray microfocusing beamline was designed for comprehensive characterization of spatially complex samples or tiny samples with high spatial resolution and high sensitivity using x-ray microbeams. An in-vacuum undulator is used to provide high brilliant x-ray beam covering an energy range of 5-20 keV for the beamline. A liquid-nitrogen-cooled double-crystal monochromator and KB mirror microfocusing system are used to produce a monochromatic micron-sized x-ray beam at the sample position. A micro-beam size of less than 2 μm has been realized (Fig. 2, left). Micro-beam size of less than 200 nm will be reached in the near future using Fresnel zone plate optics. A combination of multi-experimental techniques can be applied in the experimental station, such as micro-x-ray fluorescence analysis, scanning x-ray fluorescence microscopy (Fig. 2, middle), micro-XAFS (Fig. 2, right) and micro-XRD. Among these experimental techniques, scanning x-ray fluorescence microscopy is mostly used by users for biomedical and environmental research. For example, Wei-Yue Feng's group from the Institute of High Energy, CAS, performed an experiment on quantitative imaging of element spatial distribution in the brain section of a mouse model of Alzheimer's disease in which, for detection limits of Ca, Fe, Cu, and Zn at 2 s, the detection times were 1.15, 0.53, 0.21, and 0.20 ppm, respectively [4]. Scanning x-ray fluorescence microscopy has also been used for archaeological research. For example, the smelting process of an ancient brass sheet (about 4600 BC) was revealed by mapping the distribution of Zn and Pb in the sample; this provided important clues to the origin of metallurgy in ancient China.

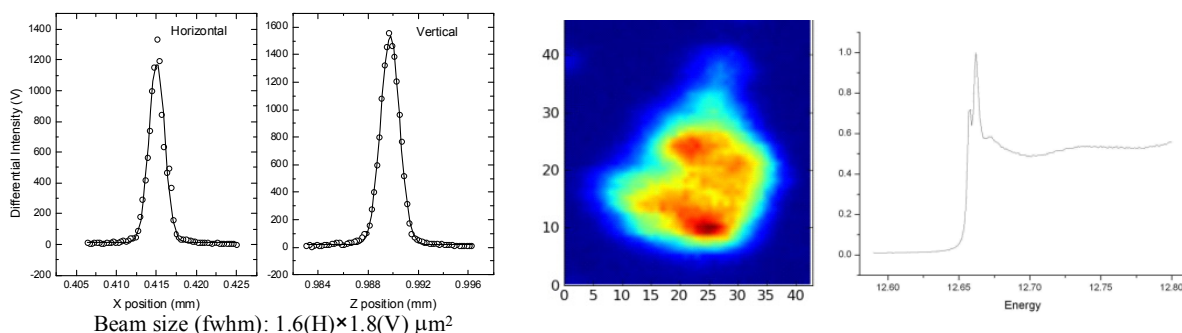


FIGURE 2. (left) Focal spot size; (middle) XRF mapping (1- μm pixel size) of Ti distribution in the TiO₂ nanoparticle exposed A549 cell; (right) micro-XAFS spectra of Se (5 ppm) in a mineral sample.

The x-ray imaging beamline was designed mainly for low-dose, nondestructive, high-resolution, Dynamic and 3-dimensional x-ray imaging for the inner microstructure of soft tissues and low-Z materials and focused on biomedical applications. The source of the beamline is a high-power multipole wiggler, which can provide intense

x-ray beam covering the required energy range of 8-72.5 keV. Unfocused monochromatic beam at a sample position with maximum beam size about 45 mm (H) \times 5 mm (V), photon flux density about 5×10^{10} phs/s/mm²@20 keV and maximum photon energy over 70 keV has been realized. The main available experimental techniques of this beamline include in-line phase-contrast imaging (PCI), K-edge absorption imaging, and microscopic computer tomography (MCT), with spatial resolution of 0.5 μ m – 20 μ m and temporal resolution of 1 ms/frame. Figure 3 is a 3D image of the blood vessels of mouse kidney with contrast medium of BaSO₄ taken at this beamline by the Med-X group from Shanghai Jiaotong University; the spatial resolution is about 20 μ m. This beamline has also been used by the users for paleontology, agriculture, and materials science research. Figure 4 shows the 3D microstructure of embryo fossil with 32 cells (about 5.8 hundred million years ago) taken by a group of researchers from Nanjing Institute of Geology & Palaeontology, CAS. Dynamic x-ray phase-contrast imaging has also been used for *in situ* observation of the growth process of tree-like crystals when Sn-Bi alloy is frozen under different experimental conditions [5].

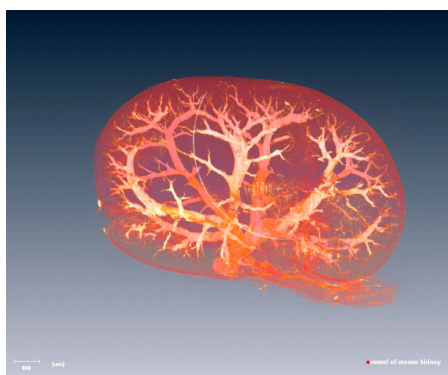


FIGURE 3. 3D image of the blood vessels of mouse kidney with 20- μ m spatial resolution.

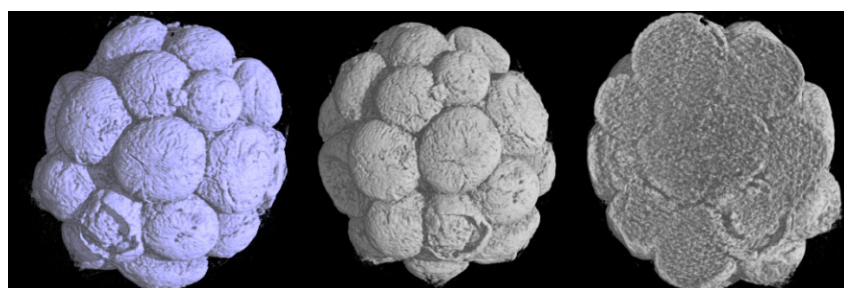


FIGURE 4. 3D microstructure of embryo fossil with 32 cells, 0.65- μ m spatial resolution.

FUTURE PLAN OF SSRF X-RAY MICROSCOPY BEAMLINES

Presently, there are 6 user beamlines (5 beamlines for protein crystallography and bioscience, 1 beamline for ARPES and PEEM) under construction at SSRF. The PEEM station will have a high spatial resolution better than 10 nm. The project of phase-II public beamlines will start in 2011 and is due to be completed in 2017. The phase-II project will construct about 24 beamlines, about 6 of which will be devoted to x-ray microscopy or imaging. When the phase-II project is completed, the SSRF beamlines will be able to cover most existing experimental methods, radiation spectrum, and application disciplines of synchrotron radiation (SR). The SSRF phase-II beamlines will be sorted by three types: one-third of the beamlines will meet the demands of a large number of the users; one-third will be dedicated to realizing higher performance for the experiments of some important scientific research fields; and one-third of the beamlines will pursue cutting-edge SR technology, such as higher time resolution, higher spatial resolution, higher energy resolution, higher photon energy, higher coherence, and so on. Table 4 lists the proposed phase-II beamlines for x-ray microscopy and imaging.

TABLE 4. SSRF Phase-II Beamlines for X-ray Microscopy

Beamline	Capabilities	Source	Key specifications
Hard x-ray nanoprobe	Scanning XRF microscopy, nano-XANES, XRD, CDI	In-vacuum undulator	5-30 keV spatial resolution <30 nm
Micro-XCT	Micro x-ray tomography, fast PCI	In-vacuum undulator	8-25keV, spatial resolution <1 μm , 106 frame/s
Coherent diffraction imaging	CDI, quantum correlation imaging	In-vacuum undulator	5-18 keV spatial resolution: 5-10 nm
Full field nano imaging	Phase contrast, absorption contrast, XANES imaging, CT	Bending magnet	2.3-15keV, spatial resolution <30 nm
Super-hard x-ray	High-energy XRD, high-energy x-ray tomography	SCW	50-300keV
Medical application	Medical imaging, microbeam therapy	SCW	20-120 keV, spatial resolution < 10 μm

The proposed hard x-ray nanoprobe beamline is representative of the SSRF effort in pursuit of x-ray microscopy with very high spatial resolution. This beamline will be used for sample characterization with high sensitivity and nanometer resolution, and will be a powerful tool for research in nano-materials, nano-devices, biology, environmental science, and x-ray optics. The x-ray nanoprobe beamline will consist of a very high-brilliance undulator source, a high stable monochromator, and focusing systems. A secondary source with slits will be used to define the source of the nanofocusing system. A very long source distance of about 100 m for nanofocusing will be used, enabling large demagnification ratio, full coherent beam. The designed minimum focal spot size is less than 30 nm and the final goal will be sub-10 nm. Various nanofocusing devices such as Fresnel zone plates, KB mirrors of multilayer Laue lenses will be tested in this beamline. Scanning XRF microscopy, nano-XANES, XRD, and CDI techniques will be used for comprehensive sample characterization.

The proposed micro-XCT beamline will be dedicated to high-speed micro-imaging and micro-CT for paleontology, materials science, biology, and phytology applications, as well as *in situ* imaging of fast processes. The available experimental techniques include absorption imaging and micro-CT, in-line phase-contrast imaging and CT, K-edge subtract imaging, and time-resolved imaging. A prominent feature of this beamline is imaging with high resolution (better than 1 μm) and high speed (10^6 frame/s); therefore a high brilliant undulator source will be used.

Another proposed phase-II beamline related to x-ray microscopy is the super-hard x-ray beamline. A 6-Tesla superconducting multipole wiggler will be used to produce intense x-ray beam up to 300 keV. The large depth of penetration of the super-hard x-ray beam will make it possible to probe the inner microstructure of bulk materials. The proposed experimental techniques will include x-ray diffraction, diffraction microscopy, and fast micro-x-ray tomography. The main purpose of this beamline is *in situ* study of engineering and industrial bulk materials or components.

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