

Introduction of the X-ray diffraction beamline of SSRF (Postprint)

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Abstract

The X-ray diffraction beamline developed at Shanghai Synchrotron Radiation Facility (SSRF) is located at the BL14B1 bending magnet port of the 3.5 GeV storage ring. The beamline optics is based on a collimating mirror, a sagittally focused double crystal monochromator and a focusing mirror. Photon flux of 4.43×10^{11} phs/s at 10 keV is obtained. The primary instrument equipped in the experimental end-station is a Huber 5021 six-cycle diffractometer. BL14B1 is a general purpose X-ray diffraction beamline and focused on material science, condensed matter physics and other relevant fields looking for structural information.

Full Text

Preamble

Introduction of the X-ray Diffraction Beamline of SSRF

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The X-ray diffraction beamline developed at the Shanghai Synchrotron Radiation Facility (SSRF) is located at the BL14B1 bending magnet port of the 3.5

GeV storage ring. The beamline optics are based on a collimating mirror, a sagittally focused double-crystal monochromator, and a focusing mirror, achieving a photon flux of 4.43×10^{11} phs/s at 10 keV. The primary instrument in the experimental end-station is a Huber 5021 six-circle diffractometer. BL14B1 serves as a general-purpose X-ray diffraction beamline focused on materials science, condensed matter physics, and other fields requiring structural information.

Keywords: Synchrotron radiation, X-ray diffraction, BL14B1, SSRF

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Introduction

X-ray diffraction is widely employed to determine the structure of crystalline materials under various conditions of temperature, pressure, electric field, and magnetic field [?]. As a Phase-I beamline at the Shanghai Synchrotron Radiation Facility (SSRF), BL14B1 is dedicated to X-ray diffraction studies in materials science, condensed matter physics, and related fields. The primary research targets include polycrystalline powders, surfaces and interfaces of films and ultra-thin films, and the microstructure of nanomaterials.

Since opening to users in 2009, BL14B1 has allocated over 20,000 hours of beamtime and executed more than 500 user proposals. The beamline and experimental end-station have undergone several upgrades to facilitate user experiments and improve operational convenience. This paper reports on the beamline optics, experimental end-station configuration, experimental methods, and commissioning results.

II. Source and Beamline Optics

SSRF comprises a 3.5 GeV electron storage ring, a 3.5 GeV booster, and a 150 MeV linac, operating with a storage ring beam current of 240 mA. The parameters of the bending magnet source for beamline BL14B1 are summarized in Table 1, and the beamline layout is illustrated in Fig. 1 [Figure 1: see original paper].

The BL14B1 beamline features three critical optical components: a collimating mirror, a sagittally focused double-crystal monochromator, and a focusing mirror, positioned 21 m, 24 m, and 30 m from the source, respectively. The collimating mirror vertically collimates the beam into parallel light to improve energy resolution. The monochromator consists of a flat water-cooled first crystal and a dynamically bent sagittal crystal that focuses the beam horizontally onto the sample. The focusing mirror further focuses the beam vertically onto the sample point.

The beamline's angular acceptance, determined primarily by the water-cooled quad-blade slit (Slit1) located before the collimating mirror, is 2.5 mrad horizontally and 0.12 mrad vertically. The energy range is mainly determined by the grazing incidence angle of the collimating mirror. Both the collimating

and focusing mirrors are half Rh-coated, Si-based cylindrically bent mirrors. A grazing incidence angle of 2.8 mrad provides a cutoff energy of approximately 22 keV, while reflection from these mirrors significantly suppresses higher-order harmonics. Three beryllium windows are installed after the collimating mirror, monochromator, and focusing mirror to maintain differential vacuum levels. A beam block positioned behind the monochromator intercepts white light and bremsstrahlung radiation reflected by the collimating mirror. Two monochromatic slits located after the focusing mirror (Slit2) and at the beamline end (Slit3) block stray light and improve angular resolution. The first monochromator crystal absorbs nearly all radiation after the collimating mirror, reaching a power density of 0.4 W/mm², necessitating water cooling.

Beamline performance was evaluated through absorption and diffraction measurements on standard inorganic powder samples. The energy range was examined at 100 mA and 200 mA using the K-edge absorption spectra of CaO (4.0385 keV) and RuCl₃ (22.117 keV). Energy resolution at 8.9 keV was estimated at 2.47×10^{-4} from the width of the Cu K-edge absorption spectrum at both currents. While this method is popular and straightforward, its precision and range are limited by natural line widths. Therefore, the 10 keV XRD pattern of NIST SRM660a LaB₆ standard powder was used to more accurately determine energy resolution, yielding an instrumental resolution function of 1.91×10^{-4} at 200 mA.

Spot size and angular divergence at the focal point were measured using a quad-blade slit and ionization chamber at 100 mA and 200 mA. Spot size is defined as the FWHM (full width at half maximum) of the intensity distribution profile, obtained by scanning the slit horizontally and vertically. Angular divergence was calculated from spot size measurements at two beamline positions. In focus mode at 200 mA, the spot size is 0.21 mm (H) \times 0.13 mm (V), with an angular divergence of 1.284×0.090 mrad². Photon flux was measured using an ionization chamber according to the formula $I = EeN/\epsilon$, where I is the current intensity, E is the X-ray energy, N is the photon number, e is the electron charge, and ϵ is the absorption efficiency. A photon flux of 4.43×10^{11} phs/s was achieved at 10 keV. Table 2 summarizes the main beamline specifications.

III. Experimental End-Station Configuration

As shown in Fig. 2 [Figure 2: see original paper], the experimental end-station is equipped with a Huber 5021 six-circle diffractometer enabling multi-dimensional sample orientation for effective measurement of ceramic, crystalline, metallic, polymeric, and other sample types. Data collection employs both a Bede point detector and an image plate detector, allowing operation in high-resolution or fast-acquisition modes. In 2012, the Mar345 image plate detector was upgraded to an Mar225 CCD with associated shutters and software, providing sub-second time resolution and significantly faster readout. A Si microstrip detector (Mythen 1K) is also available with dedicated data control software and acquisition methods. Photon flux is monitored using an online ionization cham-

ber, and a Si(111) crystal analyzer substantially improves angular resolution.

A key advantage of synchrotron-based techniques is the extremely high photon flux, which enables in situ studies impossible with conventional XRD facilities. In situ experiments provide real-time investigation of samples under non-ambient conditions that might otherwise induce changes. The beamline offers in situ capabilities through MRI TC-radiation and Linkam high-temperature chambers (Fig. 3 [Figure 3: see original paper]).

The MRI TC-radiation high-temperature chamber (Fig. 3(a)) operates in inert gas environments or vacuum. Samples can be heated to 800 °C under inert gas using a direct Mo strip heater, or to 1100 °C under vacuum using a radiation heater, making it suitable for powder and metallic samples. Samples are loaded into an Al₂O₃ crucible, and diffraction experiments are performed in reflection mode. Typical metallic sample dimensions are 10 mm × 10 mm × 1.5 mm, with temperature measured by a K-type thermocouple soldered to the sample surface. The MRI chamber can be evacuated to approximately 10⁻² Pa.

The Linkam high-temperature chamber (Fig. 3(b)) can reach 1500 °C in vacuum and is designed for transmission-mode XRD experiments on alloy and polymeric film materials. Additional capabilities include in situ electric field devices and high/low-temperature chambers for thin films. The beamline also provides a supporting laboratory equipped with a fume hood, glove box, vacuum oven, balance, and other sample preparation facilities for users.

IV. Experimental Methods and Examples

Available experimental techniques include high-resolution powder diffraction (HRXRD), X-ray reflectivity (XRR), grazing incidence X-ray diffraction (GIXRD), reciprocal space mappings (RSMs), atomic pair distribution function analysis (PDF), and total reflection X-ray fluorescence spectroscopy (TXRF).

A. High-Resolution Powder Diffraction

This method is applied to heterogeneous catalysts, hydrogen storage compounds, lithium battery electrode materials, ferroelectric compounds, thermoelectric compounds, superconductors, and other materials. Compared to conventional XRD, synchrotron-based powder XRD offers monochromatic high flux and highly collimated beams, eliminating K α ₂ diffraction peaks while providing higher signal-to-noise ratios and superior angular resolution.

Professor Chen's group utilized data collected at BL14B1 to determine crystal structures through powder XRD pattern indexing, lattice determination, space group analysis, lattice constant calculation, deconvolution of overlapped diffraction peaks, intensity determination of each diffraction peak, and calculation of related structure amplitudes [?]. Their work on synthesizing and characterizing a new hydrogen storage material—light metal ammonia borane—expands the research field of B-N related chemicals and provides theoretical foundations and

technical support for developing novel hydrogen storage materials.

B. X-ray Reflectivity

XRR employs low-angle θ - 2θ scanning (typically $2^\circ < \theta < 10^\circ$), also known as small-angle diffraction. Interface reflection arises from differences in X-ray refractive index between media layers, with the XRR signal resulting from periodic diffraction in multilayer structures. The multilayer iterative calculation method for XRR was first introduced by L. G. Parratt [?], with matrix calculation methods for non-ideal interfaces developed subsequently [?, ?]. XRR is widely applied to films, ultra-thin films, and multilayers, enabling determination of electron density, thickness, and roughness. Professor Xu Yao's group used XRR to determine thickness and optical constants of sol-gel derived polyvinylpyrrolidone/ZrO₂ films [?].

C. Grazing Incidence X-ray Diffraction

GIXRD is extensively used to study superconducting, ferroelectric, thermoelectric, and semiconducting thin films, as well as wet-epitaxially-grown organic films and polymer thin-film transistors. Films as thin as one atomic layer can be grown on single-crystal substrates and analyzed by GIXRD. At very small incidence angles, the detection cross-section increases significantly, enhancing sample signals while suppressing substrate contributions.

Using a point detector, data can be collected either in-plane or out-of-plane. By fixing the incidence angle (e.g., $\theta = 0.2^\circ$) and scanning the 2θ arm horizontally, in-plane diffraction signals displaying long-range ordering perpendicular to the sample surface can be collected. Out-of-plane signals, revealing long-range ordering parallel to the sample normal, are obtained by scanning the 2θ arm vertically. With a 2D image plate detector, both modes can be further investigated after determining the film incidence angle using the point detector.

Professor Pei Jian's group studied the structure of two thin organic films, IIDDT and IIDT [?]. GIXRD revealed four strong diffraction peaks for IIDDT but only a weak peak for IIDT, indicating that IIDDT possesses regular long-range ordering along the (001) direction while IIDT is more amorphous. Furthermore, IIDDT exhibited superior mobility and on/off ratios—critical parameters for field-effect transistors—demonstrating a clear structure-property correlation. The film structures were confirmed by AFM.

D. Reciprocal Space Mappings

Reciprocal space mapping is used to investigate domain structures, strain relaxation, orientation differences, and lattice mismatching in crystalline or film samples. Professor Yang Ping from the Singapore Light Source successfully measured BiFeO₃ thin films grown on different substrates at BL14B1 using this technique [?]. For a BiFeO₃/LaAlO₃ sample, the (103) diffraction peak ap-

peared as a triplet, indicating a monoclinic Mc phase rather than the MA or MB phases reported previously.

E. Atomic Pair Distribution Function Analysis

The atomic pair distribution function (PDF) method has attracted significant recent interest [?, ?]. By illustrating atomic pair correlation information, PDF is valuable in condensed matter physics and catalytic chemistry, particularly for nanoscale systems. PDF provides atomic correlation information up to several nanometers, complementing EXAFS (Extended X-ray Absorption Fine Structure) where correlation length is typically limited to nearest neighbors (a few angstroms).

PDF measurements at BL14B1 utilize a diffractometer for large 2θ angles and higher X-ray energies. The setup employs 18 keV X-rays with a wide 2θ scanning range ($>150^\circ$, corresponding to a Q range of 17 \AA^{-1}) for PDF studies of amorphous materials (glass or liquid) and nanomaterials.

F. Total Reflection X-ray Fluorescence Spectroscopy

Total reflection X-ray fluorescence (TXRF) spectroscopy was developed in collaboration with users. As a complementary non-destructive elemental analysis technique, TXRF is widely used to determine depth profiles of trace elements in semiconductors, thin layers on substrates, and adsorbed molecules [11-14]. Combined with XRR, TXRF enables non-destructive depth profiling of layered samples. Professor Li Wenbin's group analyzed a Si(substrate)/W/Ni/Ti/N multilayer using both reflectivity and grazing incidence fluorescence measurements, with combined data refinement yielding more precise structural parameters [?].

V. Summary and Outlook

BL14B1 is a high-resolution X-ray diffraction beamline whose optics and end-station configuration are well-suited for polycrystalline powders and film surfaces/interfaces. The beamline successfully implements diffraction techniques including GIXRD and RSM, along with specialized in situ experimental devices. These capabilities have significantly promoted user research programs, which have grown rapidly. However, global publication analysis indicates that beamlines dedicated specifically to powder diffraction, such as those at the Australian Synchrotron, Diamond I11, and Spring-8 BL02B2, demonstrate higher productivity. As a general-purpose diffraction beamline, BL14B1 at SSRF should further strengthen its focus on powder diffraction research, requiring continued beamline upgrades in this direction. The upcoming Phase-II beamline construction project will include dedicated surface diffraction and Laue microdiffraction beamlines, making it reasonable for BL14B1 to specialize in powder diffraction.

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