Next Plan of Taiwan Contract Beamlines BL12B2 and BL12XU for Materials Science and Bio-structure Research

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1. Background

The purpose of constructing Taiwan Contract Beamlines at SPring-8 was to extend synchrotron radiation-based research of National Synchrotron Radiation Research Center (NSRRC) to the spectral range of hard x-rays, complementary to the spectral range of VUV and soft x-rays most suitable for the 1.5 GeV Taiwan Light Source (TLS). The scientific programs focused on bio-structure and condensed matter physics/materials science. The SPring-8 Contract Beamline Agreement between JASRI (SPring-8) and APCST (on behalf of NSRRC) was signed on December 19, 1998. It granted NSRRC a period of 10 years from the start date for the installation and use of the Taiwan Contract Beamlines. One bending magnet port and one insertion device port were designated and subsequently NSRRC constructed BL12B2 and BL12XU, respectively according to their respective Installation Plans approved by JASRI to conduct research programs submitted along side with the Letters of Intent in April of 1998. The start dates were determined to be in June 2000 for BL12B2 and March 2001 for BL12XU. An interim review was held in November 2005. A full review was conducted in February 2010 on the accomplishment of the official 10 year period and on a future plan of the next 10 years. This future plan outlines the upgrade and new developments of beamline components for the continuation of current research programs and new initiatives.

2. Major developments in the past 10 years and current status of beamlines

2.1. BL12B2

Figure 1 presents a schematic layout of both BL12B2 and BL12XU.

2.1.1. Beamline

The BL12B2 beamline is designed to provide multiple research capabilities for bio-structure and materials research at atomic resolution. In order to accommodate a wide range of experimental requirements of x-ray users in Taiwan, this beamline includes several configurations of the optical components and can be switched between the various monochromatic and the white beam modes.

The beamline optical design follows mostly the generic SPring-8 BM beamline design. The beamline optics consists of three major components: a collimating mirror (CM) for beam collimation, higher harmonic suppression

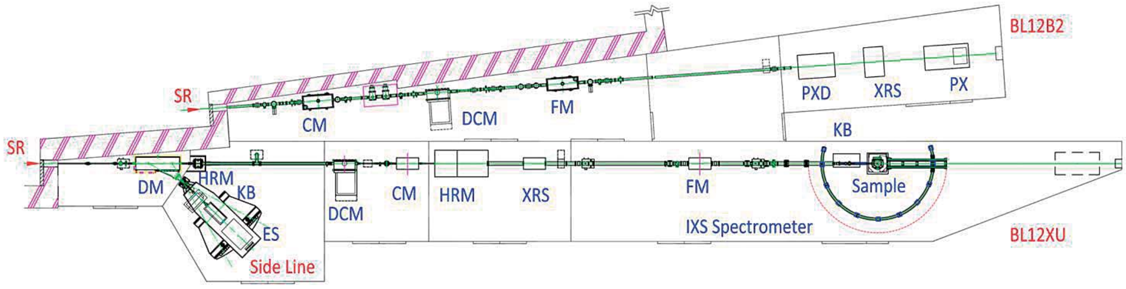

Figure 1 Schematic layout of Taiwan Contract Beamlines BL12B2 and BL12XU.

and thermal load reduction, a standard SPring-8 variable-inclined double crystal monochromator (DCM) for selecting photon energy from 5 to 70 keV, and a toroidal focusing mirror (FM) for focusing the x-ray beam at the protein crystallography (PX) end station. To maximize the utilization of BL12B2 for potential applications of Taiwan users, the beamline operation provides three different operation modes. The medium energy mono-mode for 5-25 keV is currently the main operation mode of the beamline, and provides monochromatic beam to the four experimental end stations, including the x-ray absorption spectroscopy (XAS), powder x-ray diffraction (PXD), x-ray scattering (XRS), and PX end stations, installed in tandem on the beamline. The XAS end station was decommissioned in late 2008 following the upgrade of PX end station.

2.1.2. Experimental end stations

2.1.2.1. Protein crystallography (PX) end station

So far, about 50% the user beamtime is used for protein crystallography, and is served by the PX end station installed at the end of the beamline. The PX end station was upgraded in 2008 to be compatible with RIKEN and JASRI beamlines. It has significantly reduced the time needed to conduct PX experiments. This includes the GUI based beamline control software (BSS), a fully automated sample changer robot SPACE system, auto crystal alignment, auto crystal centering and automated data collection. The old ADSC Quantum 4R CCD detector was replaced in late 2009 by a new Quantum 210r with a much faster read out and lower noise.

2.1.2.2. High-resolution x-ray scattering (XRS) end station

The other 50% user beamtime is allocated for broad demands in materials science research, among which general x-ray scattering studies take up the major part of the beamtime. This part of research program is served by a conventional Huber 6-circle diffractometer. It is now equipped with a closed cycle cryostat and heater for varied temperature experiments, and a Huber polarization-analysis goniometer. Various research subjects have been carried out at this XRS end station, such as the magnetic thin films, semiconductor quantum wells, charge density waves, semiconductor quantum dots, confined bimetallic quantum wires, etc. Diffraction spectroscopy of resonant x-ray magnetic scattering (RXMS) and diffraction anomalous fine structure (DAFS) experiments have also been carried out at this end station.

2.1.2.3. Powder x-ray diffraction (PXD) end station

Powder x-ray diffraction experiments can be performed on the beamline either using the XRS 6-circle end station or the dedicated PXD end station, which consists of an image plate setup and a Debye-Scherrer goniometer. A cryostat is also available for users to conduct low temperature PXD measurements at this end station. It now also serves for high pressure powder x-ray diffraction experiments using a diamond anvil cell (DAC) to hold a sample.

2.2. BL12XU

The BL12XU mainline is designed primarily for high-resolution inelastic x-ray scattering (IXS) here experiments with variable energy resolution from several tens to hundreds of meV to explore frontier research in correlated electron systems and electronic structure of materials under high pressure. The secondary purpose is for high Q-resolution scattering, x-ray physics and optics development. Inelastic x-ray scattering is one of a few experimental techniques that probe directly the dynamical behavior of materials, and has enormous possible applications in particular in high-pressure research. The BL12XU sideline is designed specifically for performing hard x-ray photoemission spectroscopy (HAXPES). With a much larger inelastic electron mean free path of photoelectrons compared to that of conventional VUV and soft x-ray photoemission HAXPES has the potential to probe true bulk electronic structure as well as buried interface. The overall energy resolution combining efficient x-ray optics and a high performance high energy electron energy analyzer can provide a fraction of a tenth of eV to several tenths of eV suitable for both core level and valence band photoemission.

2.2.1. Main beamline

BL12XU takes x-rays from a SPring-8 standard 4.5-m long in-vacuum undulator with a magnet period of 32 mm. The beamline consists of a sideline and a mainline. The main line begins with a Si(111) DCM, followed by a CM, a high resolution monochromator (HRM), and a FM. The DCM is of SPring-8 standard design with LN_2 cryogenic cooling. The HRM uses Si channel-cut crystals in various inline or nested 4-bounce configurations, providing variable energy resolution (~10-1000 meV) and a sufficiently wide (~keV) scanning range. At present, in-line combinations of two symmetric Si(333), Si(400) or Si(220) channel-cut crystals have been implemented, giving an energy resolution of 50, 150 or 400 meV, respectively, at 9.886 keV. Subsequently, the toroidal FM focuses the beam to the sample position of the IXS spectrometer with a spot size of 120(H) × 80(V) μm^2. It contains also the following several optional devices.

2.2.1.1. Kirkpatrick-Baez (KB) mirrors focusing system

It was designed to meet demands of high-pressure experiments to be able to routinely provide about 20 x 20 μm^2 spot size with a throughput of 72 % for 9.9 keV x-rays.

2.2.1.2. Diamond phase retarder

Our spectrometer was designed to have a horizontal arm for superior stability over a vertical arm spectrometer. However, the scattered intensity is substantially suppressed at scattering angles near 90° by a polarization prefactor for Thomson scattering in non-resonant inelastic scattering. A diamond phase retarding device was implemented in 2008 to convert the incident x-rays from being in horizontal polarization to vertical. This setup can now be routinely utilized by non-resonant IXS type of experiments.

2.2.2. Experimental end stations - Main beamline

2.2.2.1. Standard spectrometers for non-resonant and resonant inelastic x-ray scattering

The experimental end station for inelastic x-ray scattering experiments and most of the associated instrumentation are an integrated part of the beamline, and have been custom designed and built specifically to accommodate a wide range of experimental requirements for the proposed research of the beamline, in which the NSRRC personnel has played a major role. The IXS spectrometer is basically a Rowland circle instrument with a 2-m horizontal arm covering a scattering angle range of 0-140 degrees. A cryostat provides vibration-free sample cooling down to 4 K. The analyzers are Si or Ge with various crystalline surfaces for selected energy ranges covering 3*d* transition metal K-edge for resonant inelastic x-ray scattering (RIXS) or 9.9 keV for non-resonant inelastic x-ray scattering (NIXS). Multi-analyzer array can be used for NIXS type experiments to increase signal rate.

2.2.2.2. Development of 20-keV spectrometer

The currently available 10-keV x-ray is best for very low-Z samples such as H_2O and CO_2 but is not suitable for medium-Z samples such as silicon, aluminum or 3*d* transition metals because the penetration depth is too short to perform XRS in the transmission geometry. The transmission geometry is important if we need to measure low Q spectra of samples in high-pressure/high temperature environment or having only poor surfaces for reflection. Use of 20 keV x-rays is the best option in term of the penetration depth. Low resolution experiments slightly larger than 1 eV resolution are primarily considered. In the current implementation we use the forward scattering Laue geometry of a cylindrically bent triangular shape Si(660) analyzer. An energy resolution of 1.4 eV has been achieved. Source size gives a non-negligible contribution to the resolution of analyzer thus either KB mirrors focusing a small beam size onto sample or a narrow slit before sample defining source size for the analyzer is preferred. Further study is undergoing. One benefit of developing the 20-keV spectrometer is to utilize the brilliant third harmonic light from the undulator source when HAXPES (see later description) uses 6-7 keV first harmonic in the Bragg mode of diamond (111) reflection. This was in part to maximize the utilization of undulator source to meet the overcrowded user demands.

2.2.2.3. Scattering and diffraction based HUBER diffractometer

In the same experimental hutch where the HRM resides, a standard 8-circle HUBER diffractometer is installed. This end station is used for high-resolution scattering experiments, such as coherent scattering and magnetic scattering, which demands the intense coherent undulator source. The same experimental hutch and end station can be used also for x-ray physics and optics experiments.

2.2.3. Side beamline

The side line of BL12XU shares the same undulator source as the main line. It is designed specifically for HAXPES experiments. This sideline intercepts the main beam by a diamond monochromator (DM) in the first optical hutch. The DM provides single-bounce diffraction horizontally from the unchromatic light from the undulator. Its energy range 6 - 12 keV is determined by diamond (111) reflection. A rotational platform holds the rest optical elements and end station for different energies. The HRM houses a 2-bounce channel-cut crystal to reduce the bandwidth and diffracts the beam vertically. The KB mirrors focus the beam down to measured 40 × 40 microns.

2.2.4. Experimental end station - Side beamline

The HAXPES end station was developed by Professor Liu Hao Tjeng of University of Cologne, and Max-Planck-Institute for Chemical Physics of Solids (MPI-CPfS) in Dresden, Germany. The main idea is to be able to change the photoelectron detection direction between parallel to the polarization vector of incoming photons (horizontal) and perpendicular to it (vertical). The horizontal geometry yields spectra with enhanced sensitivity to the *s*-orbitals, while the vertical one was found to produce spectra with minimal *s*-contributions. These findings are crucial for valence band investigations: studies on the nature of the chemical bonding (e.g. the role of 4*s*, 5*s*, and 6*s*-orbitals) would profit from the *s*-sensitive geometry, while studies on strongly correlated electron systems (e.g. 3*d*, 4*d* and 4*f* systems) will need the *s*-insensitive geometry. A new end station is currently being designed to install two electron energy analyzers in these two geometries to be able to detect simultaneously their photoemission spectra. This installation will make our HAXPES end station unique in the world. Currently this HAXPES beamline/end station is still under commissioning.

3. Next plan for the second 10 year period

The objective of this next plan is to conduct frontier research in materials science and bio-structure science utilizing the existing beamline setup of BL12B2 and BL12XU and to upgrade them or to develop new components wherever necessary. It is also aimed to integrate the scientific programs with the upcoming 3 GeV Taiwan Photon Source (TPS) when it is scheduled to start commissioning in 2014.

3.1 Scientific direction for bio-structure research at BL12B2 and BL44XU

PX will continue to occupy 50% beamtime of BL12B2 mainly for Taiwan users. The research direction is moving toward more difficult subjects such as (1) membrane protein structures, (2) large macromolecular assemblies (>200 KDa), (3) structure based drug design, (4) drosophia brain protein structures, (5) DNA/RNA-binding proteins involved in cell defense and translational regulation and (6) functional important protein structures.

An improvement of flux transmission of BL12B2 for PX was conducted in 2009. Its flux and flux density are now comparable to those of RIKEN BM PX beamlines BL26B1 and BL26B2 of SPring-8 as well as BL13B1 of TLS. However, its flux density is still not high enough for small crystals of microns to tens of microns or crystals of large unit cells. NSRRC thus formed a collaboration relationship in 2008 with Academia Sinica in Taiwan and the Protein Research Lab of Osaka University to install a high end MAR225HE CCD detector at Osaka's high flux microfocused BL44XU of SPring-8 in late 2009 in exchange of 25% beamtime for Taiwan PX users starting 2010. This is to enhance the beamline capabilities of Taiwan Beamlines to serve higher demanding beyond current BL12B2 can offer.

3.2. Scientific direction for materials science research at BL12B2 and BL12XU

3.2.1. Structural determination and phase transitions in novel materials

X-ray scattering for novel materials using the 6-C diffractometer has been one of the most fruitful utilizations of BL12B2 and generated many high profile research papers. This end station will continue to serve users demanding high quality x-ray diffraction data on the structure of single crystal samples of novel materials at ambient pressure and low and medium high temperatures aiming at structural determination and phase transitions at high Q-resolution.

3.2.2. Structure and electronic origin of phase transitions under high pressure

High pressure experiments performed at BL12B2 has been focused on PXD and XAS using a diamond anvil cell (DAC) in transmission geometry at room temperature. The requirement of 23 keV or higher photon energies for high pressure PXD puts BL12B2 in a better position than current TLS wigglers and even future TPS bending magnet beamlines. The small size of a DAC also requires a small beam spots. Currently high pressure experiments are conducted by using small apertures to confine the beam size but it is not an efficient way. NSRRC is currently conducting a feasibility study on putting DAC near the PX sample position which provides a better focus for small size of high pressure samples as well as using the new Q210r CCD detector for PX to replace the slower image plate.

High pressure experiments performed at BL12XU has been focused on NIXS in the x-ray Raman setup on low-Z materials as well as resonant x-ray emission spectroscopy (RXES) across the 3*d* transition metal *K*-edge with 1*s*-3*p* emission and 4*f* rare-earth compounds at the *L*-edge with 2*p*-3*d* emission. We plan to study in the next phase more novel transition metal oxides, 4*f* system and 5*d* systems using RXES and valence band RIXS to explore the electronic origin of phase transitions under high pressure. There have been quite many user groups primarily from Taiwan and Japan. The new quasi-2D strip detector with multiple analyzers will be utilized to improve the detection efficiency for NIXS, as will be presented later. We will also apply the new 20 keV spectrometer in the NIXS setup to study the change of dielectric constant of materials under high pressure, especially on medium-Z samples

3.2.3. Low-energy excitations of exotic strongly correlated single crystals under ambient pressure and low temperatures

We plan in the next phase to explore the low energy valence excitations such as charge transfer and dd-excitations on more exotic single crystal strongly correlated systems by RIXS (3*d* TM *K*-edges, 5*d* TM *L*-edges, 4*f* RE *L*-edges and 5*f* actinides) under ambient pressure and low temperatures using the new quasi-2D strip detector for RIXS to substantially improve the energy resolution; see later description. Systems considered are quasi-2D cuprates, quasi-1D cuprates, as well as the new Fe-based superconductors such as pnictides and chalcogenides.

3.2.4. X-ray optics

A number of experiments have demonstrated the plausibility of x-ray resonant cavities using the temporal coherence properties of the undulator. The experiments were carried out at the 8-C diffractometer of BL12XU. This program has been conducted by Professor S.L. Chang in collaboration with Dr. Ishikawa's group at RIKEN SPring-8 Center. It is planed to continue in the next phase.

3.2.5. Bulk and interface electronic structure probed with HAXPES

We will continue to perform HAXPES research on bulk and interface electronic structure in the next phase in collaboration with Professor Liu Hao Tjeng of the Max-Planck-Institute for Chemical Physics of Solids (MPI-CPfS) in Dresden. The following topics are main targets.

(1) Novel inter-metallic compounds

Various research institutes and universities in Taiwan, as well as the Max-Planck Institute for Chemical Physics of Solids in Dresden-Germany are designing new classes of inter-metallic compounds and investigating their physical properties for interesting new phenomena. Materials include the (superconducting) skutterudites, chlatrates, and pnictides. Decomposition of the various orbital contributions to the valence band electronic structure is essential to understand the chemical bonding and to make progress in the modelling of their properties. Bulk sensitivity is crucial in view of the large unit cells and/or very different composition in the near surface region.

(2) High temperature phases of transition metal compounds

Understanding the electronic structure of materials showing metal-insulator and/or spin state transitions is a major challenge in the field of theoretical solid state physics. The availability of reliable experimental data is essential. Yet it is a fact that hardly any photoemission spectra have been reported for the high temperature phases of paradigm systems like V_2O_3, Ti_2O_3, $LaMnO_3$, and $LaCoO_3$ (all including their doping variations). This lack of data is caused by the observation that at elevated temperatures the oxide materials loose oxygen in the surface region, rendering data taken with the standard surface sensitive photoemission techniques to become useless. Bulk-sensitive HAXPES experiments are thus highly needed.

(3) High oxidation state materials

Many stoichiometric transition metal oxide materials are insulators due to the strong electron correlation effects at the 3*d* sites. CrO_2, $CaCrO_3$, $SrFeO_3$ and several of the $RNiO_3$ series seem to form the exception: they are metallic. One may argue that the metallic behavior is caused by the presence of hole carriers in the oxygen band (*p*-type metals): the oxygen-2p-to-transition-metal-3d charge transfer energy is negative due the high oxidation state of the transition metal ion. It is yet far from clear what the nature of these hole carriers are: are they the result of covalency with the transition-metal ions similar to the case of charge transfer insulators like NiO, or do they represent a new case in which the holes essentially no longer have the symmetry of the transition metal ions and in which one could speak about self-doping? What would be the consequences for the magnetic properties? On the experimental side not very much is known about the electronic structure of these high oxidation state materials. Standard photoemission is severely handicapped by the fact that the high oxidation state is not stable at the surface, resulting in substantial loss of oxygen thereby essentially altering the material properties. Bulk-sensitive HAXPES is therefore highly desired.

(4) Electronic structure at the interface between different layered compounds of strongly correlated systems

It was found recently the interface of two strongly correlated materials such as $LaTiO_3/SrTiO_3$ and $LaMnO_3/SrMnO_3$ exhibits physical properties completely from those of bulk phases. The photoemission technique probing directly the electronic structure becomes inadequate in the traditional VUV/SX range due to short inelastic electron mean free path (IMFP). The much larger IMFP in the hard x-ray range would be an ideal tool to measure the electronic structure at the interface directly, using both the horizontal and vertical geometries of the soon-to-be upgraded end station, given that the top layer is not too thick.

3.2.6. Coherent Diffractive Imaging on nano-materials and bio-samples

This proposal represents a new initiative of BL12XU as an international collaborative research program among NSRRC and Academia Sinica (AS) in Taiwan and Gwangju Institute of Science & Technology (GIST) in Korea. We explain it in more detail.

X-ray imaging with nano meter resolution is an emerging field for intense research activities using low-emittance sources of storage rings and more recently the free electron lasers. A low-emittance source allows x-ray measurements to obtain the phase information when coherent x-rays are employed in the data collection. The technique is now commonly called coherent diffractive imaging (CDI) which does not require the use of optical lenses. In the past few years, our team has carried out x-ray CDI studies using ID sources at SPring-8 (BL12XU) and APS as well as in-house electron CDI using field emission TEM. Here we are constructing a CDI station for BL12XU with some modifications made on the beamline to meet the coherence requirement.

The goal of the program is to perform CDI experiments at low resolution on BL12XU which help to prepare experiments to be carried at SPring-8 XFEL in mid 2011. Two areas of research are of our main focus: nano science and bio imaging which are briefly discussed in the following. The program will be carried as a collaborative research among Taiwan (K. S. Liang of NSRRC, T. K. Lee of Academia Sinica), Korea (D. Y. Noh, GIST), and Japan (C. Song and T. Ishikawa, RSC).

(1) Nano-Science

Direct image of nano-objects are of great interest in nano science. In the past, we have carried out extensively structural studies of multi-element nano-size objects such as catalytical particles and quantum dots using anomalous powder diffraction and grazing incidence diffraction techniques. There are many unsolved questions because the experiments only probed an average structure. The XFEL will allow the measurements to be carried on a single particle. The big challenge is to probe the strain distribution and defects in such a single nano-particle under controlled conditions in energy and environmental research.

(2) Bio-Imaging

NSRRC has continuously developed protein x-ray crystallography stations at both TLS and SPring-8 since 2000, which provide the state-of-the-art facilities for protein crystal structure research for bio-science community in Taiwan. Since a large percentage of proteins can not be crystallized, CDI has been promised to become a new tool in bio-science. NSRRC hopes to position itself in the new emerging filed for membrane protein studies which can impact to the discovery of new drugs.

3.3. Facility upgrade

The following upgrade projects are undergoing.

3.3.1. IXS upgrades for BL12XU mainline

NSRRC is testing new quasi-2D strip detector array to increase detection efficiency while keeping a similar high energy resolution for NIXS with multi-analyzer and using a single analyzer and a single strip detector for higher energy resolution for RIXS.

3.3.2. Upgrade of HAXPES end station at BL12XU sideline

The MPI-CPfS is currently building a new end station to be equipped with two MBS-A1 analyzers for simultaneously measure photoelectrons in the horizontal and vertical geometry.

3.3.3. Installation of a test station for CDI at BL12XU mainline

This end station being built will utilize the existing 8-C diffractometer to install a test station adopting Bragg scattering geometry as well as typical transmission small angle geometry.

4. Science plan beyond 2014

As TPS is a medium energy (3 GeV) storage ring its premium spectral range is below 10 keV compared to SPring-8. Therefore, we will focus on x-ray sciences with photon energy higher than 10 keV at Taiwan Beamlines when TPS becomes operational. For BL12XU high-energy IXS will be emphasized. For BL12B2 white-light and high-energy x-ray sciences, e.g. high-pressure, high-energy diffraction, and time-resolved crystallography are to be explored. It is being evaluated that the electron beam energy of TLS is to be lowered from the current 1.5 GeV to 1.0 GeV optimized at VUV spectral range. Thus TLS, TPS and Taiwan Beamlines at SPring-8 will serve users in Taiwan and in the world exploring synchrotron radiation sciences over a very broad spectral range.

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