Hard X-ray XAFS beamline, BL5S1, at AichiSR

M Tabuchi1, H Asakura1, H Morimoto1, N Watanabe1, Y. Takeda2

1Nagoya University, Synchrotron Radiation Research Center, Furocho, Chikusaku, Nagoya 464-8603, Japan
2Aichi Science and Technology Foundation, Aichi Synchrotron Radiation Center, 250-3, Minami Yamaguchicho, Seto 489-0965, Japan

m.tabuchi@nusr.nagoya-u.ac.jp

Abstract. A XAFS beamline, BL5S1, had been operated at Aichi Synchrotron Radiation Center, Japan since March 2013. The beamline was designed for the measurements in the energy range from 5 to 20 keV. The photon flux of 6 x 10^{10} at around 9 keV and beam spot size of 0.5 x 0.3 mm at sample position are as good as designed. For the standard transmission XAFS measurement, both of the step- and quick- scan modes are available. Energy resolution at around 9keV is good enough to discuss the energy shift of the order of 0.1 eV or higher even when the measurements are conducted in the quick-scan mode. With several kinds of detectors for fluorescence and/or CEY detection mode measurements, and various kinds of sample holders which are supported by the XAFS measurement software, users easily obtain spectra for their samples. Such a standard, well operated and easy to access XAFS beamline must be very important to broaden the base of the XAFS society further.

1. Introduction

Aichi Synchrotron Radiation Center (AichiSR) was built in Aichi, Japan in 2012 as a new facility to utilize the synchrotron radiation. After a short set-up and commissioning periods of the accelerators and the beamlines for about one year, it has been openly operated for synchrotron-radiation users since March 2013. A beamline, BL5S1 at AichiSR, was designed and built as a hard X-ray XAFS beamline to satisfy the potential demands both of the industrial and the academic users in the local region. In this paper, we introduce the design and initial performance of the BL5S1 with some information on the accelerator as a light source and show recent status after the two years operation for the users.

2. Aichi Synchrotron Radiation Center

The light source at AichiSR consists of three main components, a LINAC, a booster ring, and a storage ring[1]. The electron beams of 50 MeV...
from the LINAC are accelerated to 1.2 GeV by the booster ring and finally injected to the storage ring. The storage ring is usually operated in top-up mode at 300 mA. The storage ring consists of eight normal-conducting and four super-conducting bending magnets with an undulator in a linear section. The four super-conducting bending magnets of 5 T realize to provide hard X-rays of the critical energy at 4.8 keV while the normal-conducting bending magnets of 1.4 T provide soft X-rays of the critical energy at 1.3 keV. Figure 1 shows theoretically calculated photon fluxes as a function of photon energy for the AichiSR super- and normal-conducting bending magnets, comparing with those for the bending magnets and wiggler of SAGA-LS and the bending magnets of Photon Factory. As shown in Fig. 1, for the superconducting bending magnets of AichiSR, the critical energy and thus the shape of the spectrum is similar to those of SAGA-LS.

In the energy range from 5.0 to 20.0 keV, where the hard X-ray XAFS beamline BL5S1 was designed to be operated, photon fluxes of several $10^{10}$ photons/s were expected at the sample position from the calculation shown in Fig. 1.

3. Hard X-ray XAFS Beamline at AichiSR, BL5S1

Figure 2 schematically shows the optics of BL5S1 including experimental hatch. The main components of the optics are a couple of 1 m long mirrors coated with Rh and a Si (111) double-crystal monochromator. Through the mask and the first slit, the acceptance of the beam line is 2 mrad in horizontal. The mirrors are located before and after the monochromator for collimating and focusing. The grazing incidence angle of the mirrors are usually set at 3.36 mrad to realize the cut off energy of 20 keV. The first mirror is collimating the incident beam in vertical, and the second bend-cylindrical mirror is focusing the beam at sample position both in horizontal (2:1) and vertical. For the XAFS measurements around the absorption edges less than about 7 keV, the grazing angles of mirrors are changed, typically 9 mrad, to reduce the 3rd harmonics. The monochromator, provided by Kohzu Precision Co., Ltd, keeps the water-cooled first crystal on a set of curved arms with cams and control the position and the angle of the crystal mechanically to fix the out going beam position. The second crystal has no cooling system. The monochromator can be driven as fast as 100 eV per several seconds when a quick-scan mode XAFS measurement is performed.

Figure 3 shows (a) the actually measured photon fluxes at the sample position in the experimental hatch, (b) the beam spot image taken at the focusing point of the focusing mirror, and (c) measured Cu-K XANES spectra in step- and quick-scan modes. The quality of the X-ray beam at BL5S1 can be understood roughly from Fig. 3. The flux is as expected and high enough for most of the experiments.
The beam spot size is achieved by the bend-cylindrical mirror. The vertical spot size can be changed by changing the degree of the bend. However, the horizontal size is determined by the cylinder shape of the mirror. The performance of the focusing mirror is as good as designed. The position of the focused spot moves less than a few 10 μm while usual XAFS measurements in the energy range of several 100 eV. There is no problem to discuss the energy shifts of the order of 0.1 eV or higher even in the quick-scan mode in the energy resolution at around 9 keV.

In the experimental hatch, following a 200 μm-thick Be window at the end of the vacuum pipe, X-Z slits to and 3 ion chambers (S1194B1 and S1196B1, Ohken) are set on the X-ray path for the typical transmission experiments. A set of electronics consists of pre-amplifiers (S2341A, Ohken) connected to the ion chambers, V/F converters (S2341 or N2VF-1H, Tsujicon) and a counter (CT08-01E, Tsujicon) is utilized both for the standard step-scan and quick-scan mode XAFS measurement. Digital multi-meters (34410a, Agilent) to measure the outputs of the pre-amplifiers are also available as alternative choice for users. A 19-elements Ge solid state detector (CJ-9-11-116, Canberra with DXP-XMAP, XIA, LLC), a 4-elements silicon drift detector (XSSD50-07-4 with APV8004X(DSP), TechnoAP) and a 3 grid Lytle detector (EXAFS company) for the fluorescence yield measurements and a CEY(conversion electron yield) measurement chamber are available.

In order to satisfy the demands on various kinds of experiments, three types of sample holders are always stand by. The first one is a position fixed single sample holder which can keep a sample mounted.

![Fig. 3](a) Measured flux at sample position, (b) Spot size taken at focusing point, (c) XANES spectra of Cu-K edge measured at BL5S1 with step- and quick-scan modes. The spectrum of 'Quick Scan' shifted by 0.1 to upper for visibility.

![Fig. 4](The number of the shifts spent by users at BL5S1 and the occupancy rate to the available shifts for each month. AichiSR was shut down for about one month in Jan. 2014 and Apr. 2015.)
on a standard slide-film frame. The second one is a remote-controllable X-Z stage of which motion range is 10cm x 10cm wide with 1μm resolution. For the X-Z stage, a few kinds of sample-holder blades are preliminary prepared, i.e., 9 samples (3x3) holder blade, 49 samples (7x7) holder blade, and a single sample holder for rather large samples. The third one is a remote controllable four axes (X, Z, ω, θ) stage which can precisely control the alignment of a sample for some experiments like as total reflection mode measurements. The controls of the remote stages are integrated to the XAFS measurement system to make complex measurements easy.

H₂, O₂, N₂, and He gas lines controlled by digital mass flow meters are available in the experimental hatch for the users who conduct in-situ XAFS measurements with the gases. An in-situ cell for transmission measurement which can be heated up to 600°C is ready to use on the XAFS measurement stage with the gases.

The main target of the beamline BL5S1 are users who would like to use the XAFS measurement as one of the tools for their daily research and it is not expected that all the users of BL5S1 are the specialists of the XAFS measurement. Therefore, a large part of the efforts of the staffs working at BL5S1 is spent to make the beamline user-friendly. Most of the instruments at the beamline are preliminarily set up and tuned for the experiments of the day before the users' beam time starts. The XAFS measurement software is so simplified as the users need only a few clicks on the user-interface to start standard XAFS measurements. As a result, BL5S1 is easy to use even for the light users also for the specialists. Such a beamline, with the policy and easy to access for users, must be very important to broaden the base of the XAFS society further.

AichiSR provides beam time to the users in the unit of shift. 1 shift is 4 hours. Figure 4 shows transitions of the shifts spent by users and the occupancy rate to the available shifts for each month. As shown in Fig. 4, the number of the used shifts steeply increased just after the AichiSR started to be operated for public users, and it has been almost saturated at maximum for about two years. The result shows that BL5S1, which is not special but rather standard hard X-ray beamline, has been accepted by the users as one of the very important tools for their research and development.

4. Conclusions
A new hard X-ray XAFS beamline, BL5S1, built at Aichi Synchrotron Radiation Center which has been operated since March 2013 was introduced. The beamline, BL5S1, was designed for the measurements using SR in the energy range from 5 to 20 keV from a super-conducting bending magnet of 5 T. Measured photon flux at the sample position was 6 x 10¹⁰ at around 9 keV and the beam spot size of 0.5 x 0.3 mm are as good as designed. For the standard transmission XAFS measurement, both of the step- and quick-scan modes are available. Energy resolution at around 9 keV is good enough to discuss the energy shift of the order of 0.1 eV or higher even when the measurement is conducted in the quick-scan mode. With several kinds of detectors for fluorescence and/or CEY detection mode measurements, and various kinds of sample holders which are supported by the XAFS measurement software, users easily obtain spectra for their samples. Such a standard, well operated and easy to access XAFS beamline must be very important to broaden the base of the XAFS society further.

References
[1] http://www.astf-kha.jp/synchrotron/en/userguide/kougen/