Journal of Physics: Conference Series **712** (2016) 012146 doi:10.1088/1742-6596/712/1/012146

XAFS beam lines at Aichi Synchrotron Radiation Center dedicated to industrial use

Yoshikazu Takeda

Aichi Synchrotron Radiation Center, Aichi Science & Technology Foundation, Seto, Aichi 489-0985, Japan

E-mail: takeda@astf.or.jp

Abstract. Aichi Synchrotron Radiation Center was designed for industrial use following five years of discussion among academia, industry and local government in the Aichi area. Among the six beam lines constructed, those that facilitated X-ray absorption fine structure (XAFS) analysis were given first priority. In addition to the hardware, attention was given to the development of operating procedures that were quick and user-friendly. The facility entered public service in March 2013. In the year 2013, 55% of the experiments involved XAFS analysis (hard X-ray, soft X-ray and vacuum ultraviolet regions) and in 2014 it was 57%. The range of research fields is very broad, emphasizing the importance of the XAFS beam lines.

1. Introduction

The Aichi Synchrotron Radiation Center (AichiSR) was designed mainly for industrial use and entered public service on March 26, 2013. The facility is owned by Aichi Science & Technology Foundation, which cooperates with industry, academia and the Aichi Prefectural Government. The AichiSR is responsible for maintenance, operation of the facility and user services.

The AichiSR is the 8th SR facility constructed for public use in Japan. Since three general-purpose facilities already exist (Photon Factory, UVSOR and SPring-8), other facilities can be designed for specific purposes. When we first started discussing an SR facility in the Aichi area in 1991, the Photon Factory and UVSOR were already operating, and construction of the SPring-8 had begun. Therefore, our aim was to construct a compact and easily accessible facility. Based on investigations carried out in 2000 and 2001, we learned that there were a large number of active SR users in industry and universities in the Aichi area, and some of them expressed a desire to have an SR facility in this area. Considering the needs of industrial and academic researchers, working mainly in the fields of applied physics and chemistry, in 2003 we proposed a plan called “Photo-Science Nanofactory”. In this proposal, a center would be constructed that focused on SR, but also provided facilities for other high-level measurement and analysis methods such as transmission electron microscopy, scanning electron microscopy, secondary ion mass spectroscopy and nuclear magnetic resonance [1]. The technicians, scientists, and researchers working at the facility would fully support the needs of users. Research laboratories, incubation laboratories, and conference and meeting rooms would also be constructed. This plan attracted the attention of both industry and the Aichi Prefectural Government.

Based on opinions expressed by both industry and academia, the design was based on a 1.2-GeV storage ring with a booster ring for top-up operation, and four superconducting bending magnets (superbends). The working group was made up of members of Nagoya University, members of the Aichi Prefectural Government, and power users from industry and brushed up the design. A plan for implementation of the Nanofactory was completed, and the final report of the “Knowledge Hub” project was submitted to Aichi Prefecture. In 2009, Aichi Prefecture approved construction, which then started in 2010. In the summer of 2011 the accelerators were installed, and commissioning began in 2012. The first light was observed in the summer of 2012, and the opening ceremony took place on March 22, 2013. The construction costs for the facility were covered by Aichi Prefecture, donations from industry and the Japanese National Government. Therefore, from the beginning, the AichiSR facility was a common local and national asset, and is basically open to any researcher without restrictions based on region or country.

2. Facility overview

2-1. Accelerators

The accelerators consist of a 50-MeV LINAC, a 1.2-GeV booster ring and a 1.2-GeV storage ring [2]. The storage energy of 1.2 GeV was determined based on the desire to construct a compact ring (72-m circumference) that could supply hard X-rays (up to about 20 keV) into more than eight beam lines from four superbends with a magnetic field of 5 T [2,3]. The combination of compactness and the ability to produce both hard and soft X-rays was a basic requirement during the design phase. The booster ring has two purposes. First, it removes the need to ramp up the magnetic field in the bending magnets of the storage ring, thus avoiding any possible nonlinear dependence of the magnetic field in the superbends on the driving current. Second, it allows the storage ring to be run in top-up mode. Light with the same quality and brilliance is required for experimental repeatability and for checking the reproducibility of products.

2-2. Beam lines

The six beam lines currently available for use are: 1) hard X-ray absorption fine structure (XAFS) and fluorescence X-ray analysis (BL5S1: 5-20 keV), 2) soft X-ray XAFS and photoelectron spectroscopy (BL6N1: 1.75-6 keV), 3) ultra-soft X-ray XAFS, vacuum ultraviolet (VUV) and photoelectron spectroscopy (BL7U: 0.03-0.85 keV), 4) powder X-ray diffraction (BL5S2: 5-23 keV), 5) X-ray reflection, thin film and surface diffraction (BL8S1: 9.3-14.6 keV) and 6) small-angle X-ray scattering (BL8S3: 8.2 and 13.5 keV) [4]. This beam line configuration is based on user demand.

2-3. Management

The key factors that were considered with regard to industrial use were: 1) ease of use (low threshold for entry), 2) measurements under normal atmosphere, 3) repeat experiments on same topic, 4) short time from application to execution and 5) nondisclosure of results. To realize 4), applications are accepted every two months. All users pay for beam time, but the charge is much lower for academic use if results are disclosed.

3. Industrial use

In 2014, 66% of our users were from industry (including industry-university collaborations), 25% were purely academic and the remainder were from public research laboratories, as shown in Fig. 1. The research fields of these users were very diverse, as shown in Fig. 2. In 2013, the percentage of industrial users was 70%, but the beam time allocated for industrial use increased from 2976 hours in 2013 to 3732 hours in 2014.

The goal of industrial research is not to publish papers, but to produce profitable products. Therefore, evaluation of requests for beam time should not be performed based on scientific importance or impact on novel technology. Repeat experiments are also important in industry. Beam-line and beam-time assignment is carried out through negotiation between

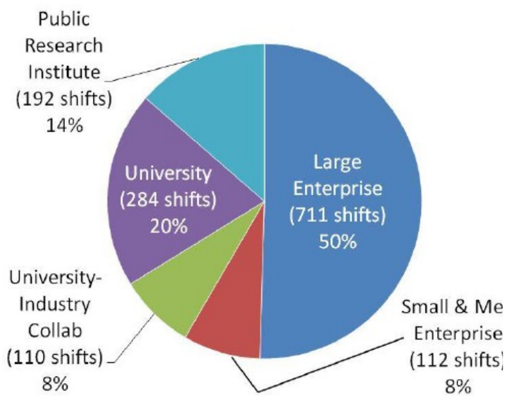

Fig. 1 AichiSR users from industry, universities and public research laboratories in 2014.

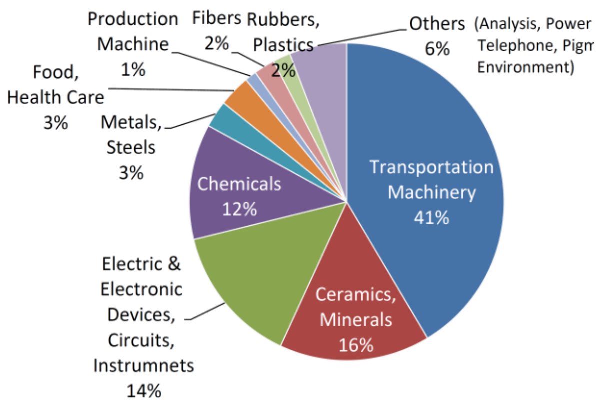

Fig. 2 Diverse research fields of AichiSR users.

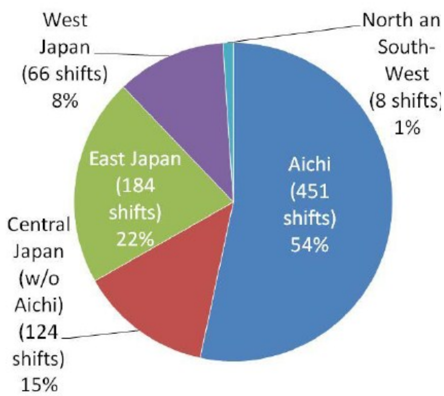

Fig. 3 Location of companies of AichiSR users.

coordinators and users. Use of multiple beam lines for one application is possible, and sometimes we actually suggest this to users.

Fig. 3 shows the locations of AichiSR user companies. As mentioned in the Introduction, many SR users existed even in 2000 and 2001 in the Aichi area, and AichiSR was constructed to meet their demands. The results shown in Fig. 3 demonstrate that a real demand exists.

4. Beam lines

4-1. XAFS beam lines

Three of the six beam lines are for XAFS measurements in the hard X-ray, soft X-ray and VUV regions. Another XAFS beam line covering the soft X-ray to VUV region is under construction. The most popular beam line is the hard X-ray XAFS beam line, which is often overbooked. For this reason, it has been decided to construct another new beam line for hard X-ray XAFS measurements. In 2013, 55% of the experiments involved XAFS (hard X-ray, soft X-ray and VUV) and this increased to 57% in 2014, as shown in Fig. 4.

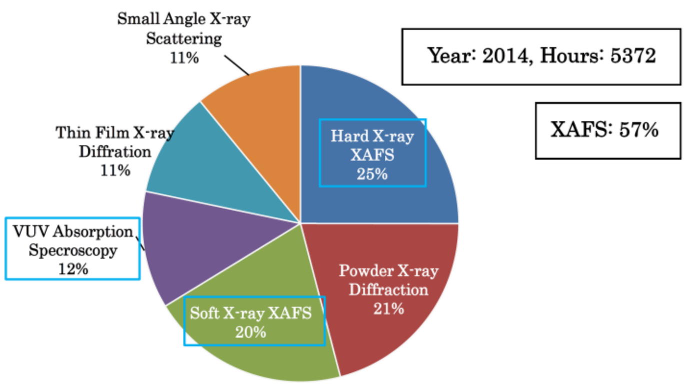

Fig. 4 The three XAFS beam lines use 57% of the beam time at AichiSR.

The most popular beam line BL5S1 is capable of operating in transmission mode, fluorescence yield mode, conversion electron yield mode, and can be used to perform quick XAFS experiments. The most common elements analyzed are Ni, Fe, Mn, Co, Ti, Zn and Cu. For example, Ni, Fe and Mn are typically found in battery electrodes. Analysis of these elements involves K-edge measurements. BL6N1 allows electron and fluorescence yield modes, either under vacuum or in a He atmosphere. The most common elements analyzed are S, Si and Ca in solids and liquids, and in particular S in catalysts and rubber. L_{III}-edge measurements of Rh, Pd, and Ag are also commonly performed. At BL7U, the electron yield mode is currently used, typically for elements such as N, C and O. Photoelectron spectroscopy can be performed using the soft X-ray and VUV beam lines.

The percentage of industrial use for BL5S1, BL6N1 and BL7U is 70%, 71% and 47%, respectively. Although this often involves analysis of heavier elements in the hard X-ray region, analysis of lighter elements is becoming more popular due to the desire for lightweight products. Even in the area of structural materials, carbon fiber reinforced polymer is increasing in importance, so that the very light element C has become a major target for measurements. Development of functional organic materials has also increased the need for analysis of C and O in the VUV region. Li in batteries can also be investigated using BL7U. Although there is a strong need for technical and scientific support for work in the VUV region, we currently have a shortage of scientists with the relevant industrial experience.

4-2. Examples of XAFS measurement

4-2-1. XAFS analysis of liquid solution

Liquid solutions can be analyzed in a He chamber at atmospheric pressure using X-rays with energies above 1 keV. At BL6N1, measurements of S K-edge and Ag L_{III}-edge spectra of such solutions have been extensively performed.

In Fig. 5 an example of XAFS signal of Ag nanoparticle dispersed in water is compared with those of Ag metal and AgNO3 powder [5].

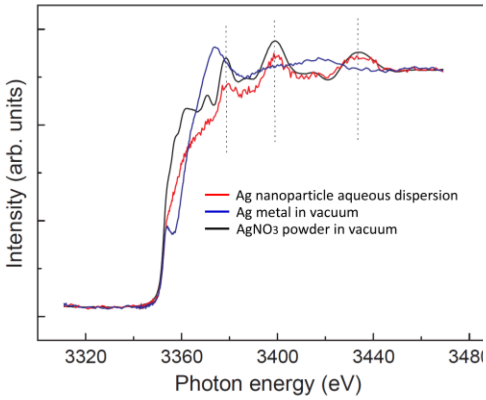

Fig. 5 XAFS signal of Ag nanoparticle in water is compared with those of metal Ag and AgNO3 powder.

4-2-2. Transfer vessel

A transfer vessel has been developed to keep samples under a high vacuum while transferring them between high-vacuum chambers at AichiSR and other member facilities of the project "Photon-Beam Platform" for different experiments at different beam lines [6].

5. Summary

After five years of discussion and planning by universities, industry, and local government in the Aichi area, the Aichi Synchrotron Radiation Center was designed for industrial use. The choice of accelerators, beam lines, and service management was determined based on discussion and feedback involving industry and academia. Of the six beam lines available, the highest priority was given to XAFS beam lines that allow analysis of elements from Li to U. In addition to the hardware, attention was given to the development of operating procedures that were quick and user-friendly. The facility became available for public use in March 2013. In 2013, 55% of the experiments involved XAFS analysis (hard X-ray, soft X-ray and VUV regions). The beam time allocated to industrial use increased from 2976 hours in 2013 to 3732 hours in 2014. The range of research fields for users of the facility was found to be very broad. Although at present there is an energy gap for the XAFS beam lines, this will be filled by the new BL1N2 beam line, which will allow analysis of Na, Mg and Al.

References

[1] Takashima Y, Yamane T, Takeda Y, Soda K, Yagi S, Takeuchi T, Akimoto K, Sakata M, Suzuki A, Tanaka K, Nakamura A, Hori M, Morita S, Seki K, Mizutani U, Kobayakawa H, Yamashita K, Katoh M 2007 *AIP Conference Proceedings(American Institute of Physics)* vol 879 pp 75-78

[2] Yamamoto N, Takashima Y, Hosaka M, Morimoto H, Takami K, Hori Y, Sasaki S, Koda S and Katoh M 2010 *Proceedings of IPAC2010 (Kyoto Japan)* pp 2567-2569

[3] Yamamoto N, TakashimaY, Hosaka M, Takami K, Mano A, Morimoto H, Hori Y, Sasaki S and Koda S, Katoh M 2011 *Proceedings of IPAC2011 (San Sebastian, Spain)* pp 2987-2989

[4] Yamamoto N, Takashima Y, Katoh M, Hosaka M, Takami K, Morimoto H, Hori Y, Sasaki S, Koda S, Ito T, Sakurai I, Hara H, Okamoto W, Watanabe N and Takeda Y 2010 *The 10th International Conference on Synchrotron Radiation Instrumentation (Melbourne, Australia, American Institute of Physics)* pp 591-594

[5] http://www.astf-kha.jp/synchrotron/en/about/greeting.html

[6] Nakanishi Y, Yagi S and Ohta T 2010 *IEEJ Electronics, Information and Systems* vol 130 pp 1762-1767 (in Japanese)