SPring-8 BL37XU Review Committee Report
on
Trace Element Analysis
(BL37XU)

Report for Director General of
Japan Synchrotron Radiation Research Institute

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October 2006

1. Introduction

This review committee meeting was held on December 19 and 20, 2006 at SPring-8. The committee members had received the "Beamline Report BL37XU (Trace Element Analysis)" and "SPring-8 Overview 2006," and had submitted individual reports before the meeting. Over the two days, four domestic members attended the meeting. The meeting began with JASRI's guidance on the beamline review, followed by their presentation on the overview of the SPring-8 facility and a tour of the BL37XU beamline. After the beamline tour, the review committee received explanations of the beamline facilities, research outcomes and future plans from the beamline staff followed by a question-and-answer session. This report was compiled on the basis of discussion among the domestic members and the report from Professor Susini.

In 2002, the Trace Element Analysis SG moved their research activities to the BL37XU beamline to rectify the situation in which the SG had shared the BL39XU beamline with the Magnetic Materials SG. The BL37XU beamline is equipped with an in-vacuum undulator of the standard type at SPring-8, and a characteristic of this beamline is that an installed double-crystal monochromator delivers X-rays in the range between 4 keV and 37 keV to branch-A, while a single-crystal monochromator provides 75.5 keV X-rays for experiments in branch-B. The main research activities at BL37XU are μ-XRF and μ-XAFS measurements using X-ray microbeams, and X-ray fluorescence analysis using K emission lines of rare-earth elements. X-ray fluorescence holography (XFH) is also performed in BL37XU. In 2004, the activities of high-brilliant XAFS were moved from the BL10XU beamline to this beamline.

2. Status of Beamline and Experimental Equipment

- The review committee strongly commends the facts that the X-ray microprobe system has been successfully installed and that the high-energy X-ray fluorescence measurement system, the main apparatus in BL37XU, has been developed during the short period of four years after commissioning the beamline.
- An X-ray optics system of the standard type at SPring-8 has been employed in BL37XU, and a new technique has been developed to adjust the system; both make the beamline facility highly reliable.
- The characteristics of the X-ray microprobe system are the use of zone plates for high-energy X-rays at 75.5 keV and that of KB mirrors for high-energy X-rays of up to 40 keV. From the practical point of view, some improvements in the stability of the X-ray microbeams and some

evaluation of the contrast, namely the tail part of the beams, are highly desirable.

- The X-ray microprobe system is available for general users. A large number of experiments have been performed using the system. No critical remarks on the apparatus appear in the report based on the users' survey. We judge that the X-ray microprobe system has been functioning well.
- The system for high-energy X-ray fluorescence measurements has been used for qualitative analysis. In the future, the installation of data-analyzing software and the preparation of standard samples for quantitative analyses will be indispensable.
- The large experimental hutches can accommodate any size of setups so as to meet a variety of users' needs. This may place additional workload on the beamline staff.
- The X-ray fluorescence holography and the hypersensitive XAFS are under development, and their feasibility studies are proceeding. We expect many applications of these techniques in the future.
- The sample preparation environment is particularly important for analytical application:
  - The kinematical mounting system for handling samples is excellent.
  - The liquid-nitrogen spraying system for cooling biological samples is functioning well.
  - For suitable research topics, the use of SEM in combination with X-rays will lead to interesting studies.

3. Research Activities

- The review committee highly evaluates the attainment of clearly separated K emission lines from rare-earth elements in X-ray spectra. In particular, the use of X-ray microbeams exemplifies the strength of the BL37XU beamline. The development of new application fields and the introduction of new users is desirable.
- The X-ray microbeam technique has been developed in BL37XU toward the use of high-energy X-rays and has been used for μ-XRF, μ-XAFS and μ-XRD measurements. Collaboration between the beamline scientists and the power users has produced many outcomes from these X-ray microbeam analyses.
- The hypersensitive XAFS technique is promising in terms of the development of new research fields. We highly evaluate the work on XFH by Hayashi and his colleagues, who have been developing the technique in Japan.
- Notable outcomes include those from a study on the interplay between Hg and Se ions in a living body, a series of studies on the hyperaccumulator in plants (particularly, high-energy XRF analyses of Cd and other trace elements), and the applications of the analytical technique

using the K emission lines from rare-earth elements.

- Taking account of the period of four years of the beamline operation, the number of publications is reasonable in comparison with other beamlines.
- It should be noted that the applications of the above techniques to forensic science have largely contributed to the research activity at SPring-8, although these results are difficult to publish.

4. Public-Use Support System

- The average proposal acceptance rate for BL37XU is 70 % and is at the same level as for other beamlines. The beamline review committee expects further expansion of users' groups since this beamline is oriented to the application of the above techniques to various research fields.
- We understand that the beamline staff have made an effort to support users' experiments, which has given much satisfaction to general users. One permanent scientist has taken care of the main apparatuses in BL37XU for μ-XRF and μ-XAFS measurements, and she has accepted a larger workload compared with the staff in similar beamlines at third-generation synchrotron radiation facilities outside Japan. From a worldwide viewpoint, the application of X-ray microbeams is one of the most competitive fields. In addition to the development of a new instrument such as a microfocusing device, the installation of a user-friendly environment for experiments is essential to maintaining the worldwide competitiveness of the beamline. For this, an increase in beamline staff is indispensable.

5. Instrumentation and Research Applications in Future

- Our expectations of BL37XU are as follows. First, we expect SPring-8 to maintain the optimal conditions of the current apparatuses for X-ray microbeams and to routinely provide the best X-ray beams for highly sensitive and accurate measurements. Secondly, we expect SPring-8 to continue upgrading the current X-ray optics and experimental equipment described in the Beamline Report. In particular, μ-XRF and μ-XAFS measurements using ~100-nm-size X-ray beams should be realized as early as possible. In addition, BL37XU will be able to represent the SPring-8 facility by the development of a new multifunctional analytical apparatus using submicron X-ray beams.
- Q-XAFS techniques, combined with X-ray microbeams or X-ray fluorescence analysis, will be more interesting if they are applied to time-resolved measurements. The idea of 3D XRF is new, and a great advance in science is expected. To achieve this goal, sufficient simulations should

be carried out beforehand.

- A continuing effort to upgrade the sample environments for X-ray microbeam experiments is desirable for the handling of various kinds of samples. The developments of a vacuum or He-filled sample chamber for the highly sensitive detection of light elements, a biological-sample cooling system for reducing radiation damage and a sample heating system for material evaluations should be considered in the future.
- Creating an opportunity to meet potential users is essential to increasing the research output of BL37XU. Public relations and other strategies should be carefully considered. The organization of a power user group is expected to take place. Collaboration with ESRF and APS should be carried out.
- Users of this beamline should focus on research using X-ray microbeams to maintain its competitiveness. The development of advanced techniques such as XFH using X-ray beams from an undulator and highly sensitive fluorescence XAFS should be discussed from the viewpoint of the whole SPring-8 facility.

6. Summary

- High-energy X-ray microbeams represent the character of BL37XU. The research outcomes have been satisfactory for the past and are also promising for the future. The future plans for upgrading the facilities in BL37XU are adequate.
- The current number of staff is too small to carry out the future plans. An increase in beamline staff should be seriously considered.
- A roadmap should be drawn up in accordance with the R&D plans and users' needs and its recommendations should be reflected in the research plan and the facility upgrade plan.