HIGH-THROUGHPUT SYSTEM FOR SYNCHROTRON X-RAY POWDER DIFFRACTOMETRY

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ABSTRACT

An automatic measurement system for powder diffraction experiments with synchrotron radiation to achieve high throughput has been developed. An automatic sample exchanger, image recognition system for automatic sample position alignment, and online detector have been developed and linked to each other by our original control program. As a result, time taken except for X-ray exposure is shortened to a single digit. The system contributes to materials science research and analytical research.

INTRODUCTION

Since the advent of the third generation synchrotron radiation facilities, diffractometry using synchrotron radiation X-ray underwent a remarkable development from a method for structural identification to a method for visualizing charge density map closely related with the properties and functions of materials. Powder diffractometry has played a key part in materials science research at SPring-8, a third generation synchrotron radiation facility in Harima Science Garden City, Hyōgo Prefecture, Japan. The high-brilliance beam has enabled us to reveal charge density map analyzing the high counting statistics diffraction data obtained even from a very small amount of powder sample [1–4]. On the other hand, there is increasing demand for analytical research of substances harmful to our health despite small quantities of such substances. First, to evaluate potential of the third generation synchrotron radiation X-ray as an analytical technique, we have performed an experiment for detecting a very small amount of asbestos, which looks like dust at a glance. As a result, 0.02 mg asbestos could be detected in 5 s [5]. However, additional time was required for sample exchange, sample position alignment, and IP exchange/readout, which takes more than ten times as long as the measurement. This point prevented us from promoting the analytical research using synchrotron radiation. To overcome this, we have developed an automatic sample exchanger, image recognition system for automatic sample position alignment, and online X-ray detector to achieve a high throughput of synchrotron X-ray powder diffraction experiment.

CONCEPT AND DESIGN OF HIGH-THROUGHPUT SYSTEM

The powder diffraction experiment at SPring-8 BL02B2 consisted of five main procedures, namely sample set, sample position alignment, Imaging Plate (IP) set, X-ray exposure, and IP read-out. To complete the high-throughput system for the experiment, these five procedures need to be automated and linked together. So far, manual exchange and position alignment of samples, and manual exchange and off-line readout of IP have been applied to the two-axis diffractometer installed at BL02B2.
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As to sample exchange, there may be two approaches to automate the sample exchange. One is to install an automatic sample exchanger into the existing diffractometer. The other is to set the exchanger apart from the diffractometer. In the end the latter was adopted since the former limits the number of samples and interferes with other accessories such as temperature changing system, etc. An overview of the automatic sample exchanger is indicated with the diffractometer in Figure 1. For easy and fast setup of the sample exchanger to the diffractometer, the handle can lift the sample exchanger staying horizontal. After that the fine alignment of the sample exchanger center to the diffractometer center is conducted using manual X, Y, and Z axes. In this way it takes within 5 min to complete the setup of the automatic sample exchanger to the diffractometer. The exchanger is composed of three parts: the body, a turntable with 36 samples, and a sample stage with electric two-axis. Figure 2 shows the sample set to the sample stage and sample exchange on the turntable using the system. The system can handle both capillary sample using transmission geometry and thin film sample using reflection geometry by replacing two attachments on the sample stage. The sample stage is equipped with only two axes for sample position alignment, although four axes are usually required. To enable sample position alignment by two axes, design of the sample holder is very important. Two kinds of sample holders were made for capillary sample and film-type sample, respectively as shown in Figure 3. The V-shaped groove can keep the capillary sample straight leading to position alignment by two axes. In addition, the sample holder with Zn and glass is available for qualitative analysis.

Sample alignment has been performed by moving the four axes of the goniometer head through the microscope with the CCD camera to date. To achieve automatic sample position alignment, the image recognition system was introduced. The system can recognize the sample image through the dedicated CCD as grayscale. We developed software to identify the sample edge by
differentiating the grayscale and command the sample stage to move the sample to the beam center position. Figure 4 shows the schematic.

![Sample holder](image1)

![Sample set to stage](image2)

![Sample exchange on turntable](image3)

![Nd magnet](image4)

![Stage for transmission geometry](image5)

![Stage for reflection geometry](image6)

Figure 2. Detail of automatic sample exchange.

The beamline has been equipped with the offline readout IP system as an X-ray detector. This is a result of adopting the curved IP, where there is no data correction following the oblique incidence, etc. However, because of the off-line readout system it takes too much time and trouble to put on, take off, and readout the IP. Here we have introduced a commercial X-ray CCD detector as the online readout X-ray detector linked with the automatic sample exchanger and the automatic sample position alignment system by our original control program. The available detection area of the CCD is only 30 × 40 mm. The size is much smaller than the area of the IP—200 × 400 mm. We are now developing an online array type X-ray detector to cover the IP area.
Figure 3. Sample holder for automatic sample exchange.

Figure 4. Schematic of automatic sample position alignment.
PERFORMANCE OF HIGH-THROUGHPUT SYSTEM

The developed high-throughput system is composed of an existing diffractometer, automatic sample exchanger, automatic sample position alignment system, and X-ray CCD detector synchronized one another using our original software written by LabVIEW. The high-throughput system can provide us with a drastic shortening of experimental time as well as a fully automatic experiment. Time taken except for X-ray exposure has now been reduced approximately from 20 to 2 min. In addition, reproducibility and precision of aligned sample position and detector position have been improved from several tens of micrometers to a few micrometers. To evaluate the performance of the system we continuously measured 36 powder patterns of the standard sample CeO₂. The results are shown in Figure 5. The powder diffraction patterns on one IP indicate that all patterns are in good agreement. The results prove that the system works well.

Figure 5. Thirty-six powder patterns on one IP using the automatic sample exchanger and position alignment system.

APPLICATION

The system is applicable and effective to analytical research, where many samples are measured under the same experimental conditions. For instance, the system is available for comprehensive analysis of toxic substances. Furthermore a combination with nanometer size beam in SPring-8 will enable hazard assessment of nano-particles such as TiO₂, which is known as the secondary asbestos in the world. The system should contribute to materials science research as well as analytical research.
SUMMARY

In the present study we have developed an automatic measurement system for powder diffraction experimentation with synchrotron radiation X-ray to achieve high throughput. As a result, time taken except for X-ray exposure is shortened to a single digit. If X-ray exposure time is short enough, the system becomes particularly a very powerful tool for synchrotron radiation diffraction experiments. The basic design can also be applied to other experiments at synchrotron radiation facilities. In the near future we will tackle the next generation advanced diffractometer combined with the high-throughput system to cover an increasingly extensive research area.

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REFERENCES