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Powder Diffractometers of J-PARC

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In the J-PARC project, we are proposing two powder diffractometers. This article describes these powder diffractometers: a versatile diffractometer with resolution of $\Delta d/d \approx 0.1\%$ for materials science and a high-resolution diffractometer with $\Delta d/d \approx 0.03\%$ for precise analysis. Each of them looks at the other side of an off-centered decoupled hydrogen poisoned moderator. Another type of high intensity powder diffractometer is also proposed separately as a total scattering instrument.

Keywords: Powder diffractometer; Materials science; High-precision analysis; High-resolution

INTRODUCTION

The current situation of neutron powder diffraction (NPD) is characterized by rapid development in both large facilities and small ones. Large facilities should provide better quality-data, which lead to overcome the previously believed limitation in NPD. In the J-PARC project, we are proposing two powder diffractometers [1]. In this article, we describe these powder diffractometers: a versatile diffractometer SuperSirius with resolution of $\Delta d/d \approx 0.1\%$ and a high-resolution diffractometer SuperHRPD with $\Delta d/d \approx 0.03\%$. Each of them is designed to look at the other side of an off-centered poisoned moderator.

STRUCTURE EVALUATION SYSTEM: A MATERIALS SCIENCE DIFFRACTOMETER WITH $\Delta d/d \approx 0.1\%$

Rapid developed high-quality X-ray powder diffractometers (XRD) are coming into market, and users are trying to extract more structural information from XRD data. Most of them are actually dealing with materials consisted of both heavy and light elements: cement, lithium-ion batteries, fuel cell materials, hydrogen absorbing materials, ferroelectric materials, other oxides, etc. This situation would be altered when we could establish a system of structural evaluation using SuperSirius in J-PARC with easy access, quick start, accepting several tens of thousands of experiments in a year. Impact on materials development would be expected.

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Currently, we have two diffractometers, Sirius [2] and Vega [3], at KENS facility and it is open for the materials scientists. The best resolution of Sirius is $\Delta d/d = 0.1\%$ for back scattering bank and it is good for materials sciences. On the other hand, the routine d-range of Sirius is limited to be 3.5 Å. This sometimes causes problems due to the lack of information. The materials scientists who need the information in longer d-range must use the Vega diffractometer with $\Delta d/d \approx 0.3\%$.

SuperSirius looks at a decoupled poisoned hydrogen moderator (36 mm, off-centered), and the incident flight path (L_1) is 25 m with a t_0-chopper, three wavelength selection choppers and a straight guide with a total length of 12.5 m. It covers in d-range $0.15 < d(\text{Å}) < 4$ with $\Delta d/d = 0.15\%$, and covers $4 < d(\text{Å}) < 60$ with gradually changing resolution. SuperSirius is planned to be an immediate diffractometer so that materials scientists can use it like the chemical analytical instruments in their materials development process. It has better resolution than GEM of ISIS or Vega and a wider Q-range than Sirius. Typical measuring time for the present "Rietveld-quality" data is several minutes with the sample size of laboratory X-ray: 0.4 cc. SuperSirius accepts various kinds of apparatus to meet users' demands: temperatures, pressures, fields, chemical cells, etc. The utilization system of SuperSirius is a most important factor for promoting potential users to utilize it like a laboratory XRD by their side. We need to establish a support system for both academic and industrial users who are willing to use neutrons but have not been familiar with neutron diffraction; tutorial courses should routinely be held, easy access to SuperSirius should be introduced followed by quick experiments and output. In addition, since several tens of thousands of experiments in a year will be carried out at most, handling samples would be a big problem; we will prepare the special sample holders, and database of samples which are relevant with users' information, safety information, experimental data information, etc. This will be especially useful for those who will use SuperSirius like chemical analyzers in their materials development process. They may visit the facility and carry out experiments by themselves, or may not come to the facility and ask for an assisting company instead. Some users also would like to use a remote access route to the facility. The combination of powder-diffraction software, structural database and visualization software should be easily utilized for the materials structural studies. Most of the know-how makes NPD analysis difficult. It depends on studies: structural study of usual oxides, ionic conductors, metals, cement, phase transition, hydrogen absorbing metals, etc. The know-how can be implemented so that users do not have to worry about it. This package of software should also be able to deal with XRD data because users prefer a single platform. Results of theoretical calculation are also directly compared with visualized results on the same platform.

EXCEED THE LIMIT OF NPD: A HIGH RESOLUTION DIFFRACTOMETER WITH $\Delta d/d = 0.03\%$

Synchrotron Radiation (SR) powder diffractometers (SR-XRD) and advanced analysis methods have developed rapidly in the past few years. SR-XRD uses high luminosity and short wavelength X-rays as well as high resolution results in the best statistic data and wide Q-range of measurements. Advanced analysis methods like the maximum entropy method enables us to extract structural information from tiny changes in diffraction patterns. The combination of these high quality data from SR-XRD and their high-precision analysis often gives us information of light elements. Then, newly developed materials with small amounts tend to be studied only by SR-XRD, which is supported by high-precision analysis methods.

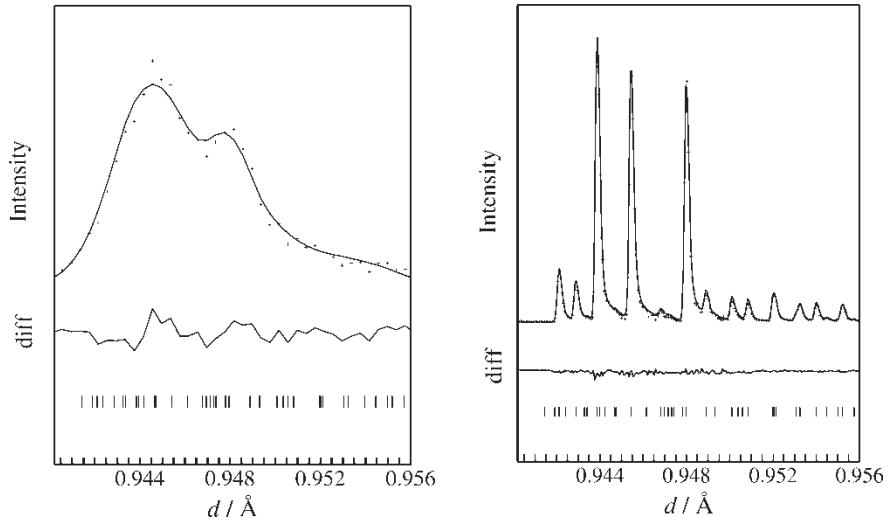

FIGURE 1 Rietveld fitting of Vega data (left) and simulated pattern with McStas together with fitting pattern (right). Tick marks indicate Bragg peak positions.

The high-precision analysis methods are in principle applicable to neutron NPD data, but the limited statistics and the instrumental resolution of existing instruments impede the breakthrough of NPD. This confinement will be removed in newly developed NPD's at ISIS, and future NPD's at ISIS, SNS and J-PARC.

We are proposing a high-resolution powder diffractometer superHRPD with $\Delta d/d = 0.03\%$, which is compatible with that of the best SR diffractometer. After careful examination with the moderator group [1], we have chosen a decoupled poisoned hydrogen moderator (25 mm, off-centered) to achieve the designed resolution within, approximately, a 100 m flight path. The incident flight path (L_1) is 92 m with three wavelength selective choppers, 32 m of curved guide, and 50 m of straight guide. Instrumental simulation for the proposed diffractometer was carried out using a program McStas [1]. Figure 1 shows a typical simulated pattern of superHRPD for an orthorhombic crystal with $24 \times 24 \times 8\,\text{Å}^3$, together with the measured diffraction pattern of Vega [4]. Because of the high resolution of superHRPD, individual intensities of Bragg reflections can be more easily identified, resulting in better reliability in obtained structure parameters. More complicated structures can only be solved with "high resolution". The proposed high-resolution powder diffractometer covers d-range $0.5 < d(\text{Å}) < 4$ with $\Delta d/d = 0.03\%$ in backward bank ($L_2 = 2$ m), and covers $4 < d(\text{Å}) < 45$ with gradually changing resolution. Other choices of d-range and resolution and intensity in combination with repetition rate would be useful for various kinds of usage. Typical measuring time for the present "Rietveld-quality" data is a couple of hours with the sample size of laboratory X-ray: 0.4 cc. The goal of the present design is to attain the best resolution with good statistics, and supply the best-quality data. We will then exceed the limit of present NPD, with the aid of the recently developing precise data analysis method. In some cases, it is advantageous to introduce nano-structural analysis methods and/or local structure analysis methods. This will lead to the holistic understanding of materials structure and their function. It should be emphasized that newly developed materials can be examined with similar amounts of samples as in the laboratory X-ray. Visualization of the results will be helpful to compare them with theoretical calculation. Those comparisons will be more and more important. Scientific cases will be reported elsewhere in detail.

REFERENCES

[1] Kamiyama, T. and Oikawa, K. "Two powder diffractometers at J-PARC", Proc. ICANS-XVI, in press.
[2] Kamiyama, T., Torii, S., Mori, K., Oikawa, K., Itoh, S., Furusaka, M., Satoh, S., Egami, T., Ikeda, S., Izumi, F. and Asano, H. (2000) "High resolution TOF neutron powder diffractometer with supermirror guide", *Mat. Sci. Forum* **321–324**, 302–307.
[3] Kamiyama, T., Oikawa, K., Tsuchiya, N., Osawa, M., Asano, H., Watanabe, N., Furusaka, M., Satoh, S., Fujikawa, I., Ishigaki, T. and Izumi, F. (1995) "A new TOF neutron powder diffractometer with arrays of one-dimensional PSD's", *Physica B* **213&214**, 875–877.
[4] Oikawa, K., Kamiyama, T. and Teshigawara, M. A structure analysis on simulated powder diffraction pattern from a proposed moderator in J-SNS, Proc. ICANS-XV, pp. 738–743, KEK Proceedings, 2000–22.