Improvement of Instrument Devices for Super High Resolution Powder Diffractometer at J-PARC

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Abstract. Super High Resolution Powder Diffractometer SuperHRPD [1] at J-PARC have suffered damage by the Great East Japan Earthquake on 11 March 2011. After temporarily restoration work, the general user program has been started since April 2012. Full-fledged restoration of the temporarily restored devices is being carried out in 2013. The absorber materials for two disk choppers were replaced to $^{10}$B$_4$C which improved quality of incident spectrum. The new detector system of 8 mm diameter position sensitive detectors was developed, and the test experiments showed excellent results.

1. Introduction

SuperHRPD accepted the first neutron beam from the pulsed source at Materials and Life Science Experimental Facility (MLF) of Japan Proton Accelerator Research Complex (J-PARC) in May 2008 and achieved the world best resolution of $\Delta d/d = 0.035 \%$ in June 2008. The beam power was operated around 4 kW at the beginning, but it is operated stably around 300 kW up to now. Several upgrading works to improve the performance of instrument such as replacement of vacuum scattering chamber, updating of the DAQ system, enhancement of detector system, etc. have also been successfully done. We have evolved the instrument with a very high S/N ratio. Coupled with the increase of the beam power, the high resolution and the high S/N ratio make it possible to carry out detailed analysis of complex crystal structures. More than 50 research proposals as general user program have been conducted at SuperHRPD and some research results have been published in various academic papers [2, 3, 4]. The Great East Japan Earthquake that occurred in March 2011 has caused very heavy damages to SuperHRPD instrument: vertical subsidence and horizontal shift of the annex building for SuperHRPD beam line, damage of the super mirror guide tube, deviation of the beam alignment, tilt of the vacuum scattering chamber, shifting of the sample center from its normal position, etc. After temporarily restoration work that took mostly over one period of the 2011 fiscal year, SuperHRPD became capable to accept the first neutron beam in April 2012. Full-fledged restoration of the temporarily restored devices is being carried out during shut down in 2013; many of beam line devices, vacuum chamber base and shielding, etc. are modified and improved by considering that the slight subsidence is continuing and the risk of future aftershock is not past. We will report them in future, and herewith report on other developments and improvements.

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2. Instrument Upgrading

2.1. Change of absorber materials for the disk chopper

SuperHRPD has about 100 m length beam line in order to achieve super high resolution. Since accelerator cycle of the MLF is 25 Hz, the wavelength window of the instrument with such long beam line would be extremely narrow. Therefore, installation of disk choppers painted with absorber materials were required in order to achieve the wide wavelength window and to suppress the frame overlap. In order to ensure the wide wavelength window of SuperHRPD, the disk choppers are usually set for the measurement at 5 Hz (1/5) and 8.33 Hz (1/3) in operation mode.

Furthermore, in order to avoid the effects of the burst and high-energy neutron, the curved guide section of 32 m in length was provided. The characteristic wavelength $\lambda^*$ calculated from equation (1) is 0.61 Å.

$$\lambda^* = \frac{2a}{\rho} \cdot \sqrt{\frac{\pi}{Nb_{coh}}} \quad (1)$$

where $a$ is the guide width (=25 mm), $\rho$ is the radius of curvature (=5 km), $N$ is the atomic density and $b_{coh}$ is the mean coherent scattering length. The value of $Nb_{coh}$ is called as the scattering length density, which denotes the value of specific materials. The supermirror guide used at SuperHRPD has three-times larger critical angle than nickel guide ($\sqrt{Nb_{coh}/\pi} = 1.73 \times 10^{-3} \text{ Å}^{-1}$). Although $\lambda^*$ is in the region where the total cross section of gadolinium decreases rapidly (absorption end), the cross section is still large value (see Figure 1). Therefore, we initially chose gadolinium oxide as absorber materials for the disk choppers. However, we learned the leakage of the short-wavelength components was large due to the excellent reflectivity of the supermirror. Then we changed the absorber materials from gadolinium oxide to boron carbide $^{10}\text{B}_4\text{C}$ ($^{10}\text{B} > 95 \text{ at%}$). The incident intensity spectra obtained using measurement of the V-Ni alloy with $^{10}\text{B}_4\text{C}$ and Gd$_2$O$_3$ disk choppers are compared in Figure 2 (left). It is evident that the extra humps were removed by the adoption of $^{10}\text{B}_4\text{C}$.

![Figure 1. Total neutron cross section of gadolinium ($^{155}\text{Gd}$, $^{157}\text{Gd}$) and boron ($^{10}\text{B}$) calculated from JENDL-3.3 code program [5]. The vertical dotted line in figure shows the energy value that converts the characteristic wavelength. A large absorption end exists in gadolinium near the characteristic wavelength. Boron shows smooth energy dependence in the same region.](image)

Although any trace of hump has been detected in the background measurements without samples, weak humps were detected when strong scattering samples are placed at the sample positions; for example, diffraction patterns of a silicon with both choppers are shown in log scale in Figure 2 (right); the extra humps hardly influenced for crystal structure analyses.
2.2. New detector system with 8 mm diameter Position Sensitive Detector

Because of shortage of initial budget and delay of development in a high spatial resolution detector system, we initially installed the 1/2 inch diameter position sensitive detectors (PSDs) that had ever used at KENS facility of KEK. This imposed limit on resolution compared with the designed one. Then we developed a new detector system based on 8 mm diameter PSDs and could improve resolution as shown below. The 16 PSDs of 8 mm in diameter were composed together into one unit and an amplifier circuit was connected to each PSD. In the new detector system, 8 amplifier circuits were built into one ASIC chip and this contributes to the more compact circuit (Figure 3).

The test measurement using the new detector system has been performed in May 2013. For the measurement, four new detector units have been arranged at the backward detector bank to cover the area about $2\theta = 157^\circ \sim 172^\circ$. A diamond powder sample enclosed into a sample holder of 6 mm in diameter was used for the measurement. We were able to obtain a very sharp diffraction peak shown in Figure 4 with $\Delta d/d = 0.0637\%$. Since the resolution of the normal sample with the 1/2 inch PSDs system is about $\Delta d/d = 0.1\%$, the improvement has been achieved by the 8 mm PSDs system. Based on the test measurement, all backward bank detectors are being replaced to the 8 mm PSDs in 2013.

Figure 2. (left) The incident intensity spectra with Gd$_2$O$_3$ and $^{10}$B$_4$C disk chopper absorber materials at 5 Hz operation. The extra humps were removed. (right) The diffraction patterns of a NIST silicon powder sample by both choppers. The extra humps existed shown by black arrows.

Figure 3. (left photograph) A new detector unit consists of 16 PSDs with 8 mm diameter. (right photograph) The conventional amplifier circuit (left) and the advanced one with an ASIC chip (right); the 16 amplifier circuits are accumulated in two ASIC chips.
3. Conclusion
SuperHRPD has been upgraded repeatedly and the high performance with the best resolution has been successfully obtained. During long-term shut down in 2013, the full-scale restoration works of re-alignment of the beam line would be carried out. The absorber materials for two disk choppers were replaced to $^{10}$B$_4$C after the earthquake. This improvement rather reduced leakage of the short-wavelength components and no extra hump was not observed in the sample data. The test experiments of the new detector system with 8 mm diameter position sensitive detectors showed excellent results, and all backward bank detectors are being replaced.

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