Automatic sample changer for IBARAKI materials design diffractometer (iMATERIA)

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Abstract. IBARAKI Materials Design Diffractometer (iMATERIA) is a high-throughput powder diffractometer. iMATERIA has a vacuum chamber, which is enclosed in a shield. Each sample is measured in 10 minutes, and must be placed in the vacuum chamber before being measured. If the vacuum in the chamber cannot be maintained while exchanging samples, the process of re-establishing the vacuum would become a bottleneck when samples are exchanged. To reduce exchange time, we developed and manufactured an automatic sample changer that can handle a large number of samples (up to 672) through both the vacuum chamber and shielding blocks.

1. Introduction

The Japan Proton Accelerator Research Complex (J-PARC) is a high-intensity proton accelerator facility. Neutron beams are produced at the Materials and Life Science Experimental Facility (MLF) from megawatt-class high-power pulsed proton beams. IBARAKI Materials Design Diffractometer (iMATERIA) [1] is built at the 20th beam line (BL20) at MLF as a versatile powder diffractometer. This diffractometer is aimed at facilitating advanced material science and is promoted by Ibaraki Prefecture—the local government. This diffractometer has a high throughput so as to handle a large number of samples at a resolution of \( \Delta d/d \sim 0.16\% \); each sample is measured in just 10 minutes. Thus, neglecting the time taken to exchange samples, it should be possible to measure about 150 samples a day. iMATERIA has a vacuum chamber in which samples are placed, and this chamber is enclosed by a shield. If a vacuum cannot be maintained in the chamber while exchanging samples, the process of re-establishing the vacuum would become a bottleneck.

Small automatic sample changers have been used in the past, e.g., the 10-sample changer used for Vega [2] and Sirius [3] at the High Energy Accelerator Research Organization (KEK). The largest, which is used for High-Pressure-Preferred Orientation (HIPPO) [4] at Los Alamos Neutron Science Center (LANSCE), can handle 100 samples simultaneously. However, these sample changers cannot add new samples or exchange stocked samples without breaking the vacuum. At minimum, we need an automatic sample changer that is capable of changing 600 samples (over 4 days), but without breaking the vacuum in the main chamber. Accordingly, we have developed and manufactured an automatic sample changer that can handle a large number of samples through both the vacuum chamber and shielding blocks.
2. Automatic sample changer

Figure 1 shows an overview of the automatic sample changer, which is organized into five parts: sample storage room, articulated robot system, elevator, pre-vacuum chamber system, and sorting system. Both the sample storage room and the articulated robot are installed on an instrument shield. The elevator, the pre-vacuum chamber system, and the sorting system are attached to the flange (port diameter 800 mm) for the main vacuum chamber, and these parts can be removed easily as a unit. The pre-vacuum chambers connect to the main vacuum chamber through gate valves, and the sorting system is set into a small chamber connected directly to the main chamber.

There are two lines from the sample storage to the sorting mechanism (two elevators and two pre-vacuum chambers). The pressure in each line is independently controlled between atmospheric pressure and vacuum. Thus, samples can be transported to the main chamber while other samples are being measured. Moreover, new samples can be set in place in the sample storage area while one set of measurements is being made. Thus, the sample-exchange time is substantially reduced.

We also developed a new sample holder.
for this automatic sample changer. This sample holder is made of vanadium (0.1 mm thick) and has a collar. The samples are packed into this sample holder, which has an Al cap and an In seal as shown in Fig. 2. A radio frequency identification (RFID) tag is placed on the top of the holder cap (made of poly oxy methylene) with two collars for handling. Each sample is controlled using RFID.

Fig. 4. Articulated robot.

To allow the addition of new samples, or the replacement of stocked samples, during measurement, the sample storage room is located outside the instrument shield. In the sample storage room, there is a shelf for samples (see Fig. 3). The shelf rotates, and the front side of the shelf presented is automatically changed according to need. The sample storage system can position 168 trays, each storing 4 samples. Thus, a maximum of 672 samples can be stored in this room.

A sample access point is located to the side of the sample storage room on the instrument shield as shown in Fig. 1. We can position only one tray (4 samples) in this access point at a time. Information on samples (name, weight, height, ID number, position in the tray, etc.) is held in a sample database, and RFID is used to identify samples. After checking the RFID information, the tray is put in the sample storage room by the articulated robot. Sample handling uses the tray (4 samples) as a unit. When we want to retrieve a sample after measurement, the articulated robot picks up the tray containing the required sample.

According to a user-defined schedule, the articulated robot picks up a tray from the sample storage room and places it on the sample stage for transport to the elevator as shown in Fig. 4. The sample stage moves...
horizontally to the elevator. The elevator picks up sample holders from the tray, and carries these sample holders vertically through through-holes in the shielding blocks to the pre-vacuum chamber, as shown in Fig. 5. The empty tray then returns to the sample storage room.

The pre-vacuum chamber has a volume of approximately 50 L. A half-round opening provides a pathway for the samples, with a gear-like stock holder at the bottom, as shown in Fig. 6. This stock holder can store 12 samples. Accordingly, the articulated robot and the elevator transports 3 trays (or a total of 12 samples) to the pre-vacuum chamber.

The vacuum in the main vacuum chamber is maintained. However, the pressure in the pre-vacuum chamber is controlled between atmospheric pressure and vacuum. In addition, the top side of the pre-vacuum chamber is at atmospheric pressure, while the bottom side is at a vacuum. Two gate valves are positioned above and below the pre-vacuum chamber. In addition, there are two pre-vacuum chambers in this system, and there is a stock holder for 12 samples in each pre-vacuum chamber. We can control the pressure for up to 12 samples during the measurement process. Thus, no time need be spent re-establishing a vacuum, except when first evacuating the chamber.

After evacuating the chamber, the underside gate valve opens, and the gear-like stock holder moves from the pre-vacuum chamber into the main vacuum chamber as shown in Fig. 7.

The sorting system is situated within the main vacuum chamber. A sample is picked from the gear-like stock holder (containing 12 samples) and is moved to the neutron beam center as shown in Fig. 7. Two collars are needed on the sample holder at this point: one for holding, and the other for handling. The sample height can be changed for each sample. Optionally, the sample can be rotated during measurement.

We manufactured an automatic sample changer with the capacity to handle more samples than any previous design. We have already installed this automatic sample changer, and it has passed an operational check. Currently, we are developing control software in order to work with a data-acquisition system. The sample exchange time is substantially reduced using this automatic sample changer.

References
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