# BL14B2
# Engineering Science Research II

## 1. Introduction

BL14B2 is a bending magnet beamline at SPring-8 dedicated to research by industrial users conducting X-ray absorption spectroscopy (XAS) measurements. Various measurement systems have been developed to realize easy and high-throughput operations of XAS measurements. In FY2020, we installed a seven-element silicon drift detector and developed a system for the efficient preparation of pellet samples for XAS measurements.

## 2. Silicon drift detector

Fluorescence-yield X-ray absorption spectroscopy (F-XAS) measurements are conducted to characterize low-concentration samples. In BL14B2, F-XAS measurement systems using a multielement solid-state detector (SSD) and digital signal processors (DSPs) are provided for industrial users. There are many requests to measure the K-edge of 3d transition metal elements by F-XAS. Therefore, we are working on improving the throughput of F-XAS measurement in the low-energy region of hard X-rays. For this purpose, we introduced a new portable seven-element silicon drift detector (SDD) and DSPs in FY2020.

Figure 1 shows a photograph of the portable SDD and DSPs. The main feature of this SDD is that its dynamic range and available solid angle for detecting fluorescence signals are larger than those of existing SSD systems previously used. Furthermore, this SDD is easy to combine with order-made equipment developed by industrial users because of its compact size and light weight. Parameters, such as course gain and region of interest (ROI), can be controlled online through DSPs.

We investigated the dependence of the count rate of fluorescence signals on the incident X-ray intensity to estimate detector deadtime. The ROI was set so as to accept the Cu Kα fluorescence, and the incident X-ray intensity was controlled by changing the thickness of attenuators (Al foil) located upstream of the ion chamber. Figure 2 shows the output count rate of the SDD as a function of input count rate. The deadtime was calculated as described in a previous report [1]. The results indicated that the deadtime τ is 0.53 μs, which is lower than that of existing F-XAS measurement systems using the SSD (τ =1.71 μs).

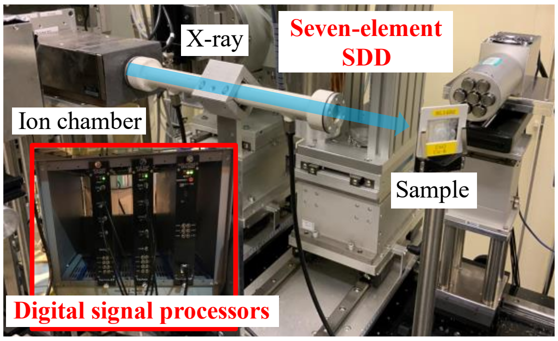


Fig. 1.Photograph of the SDD and DSPs introduced.

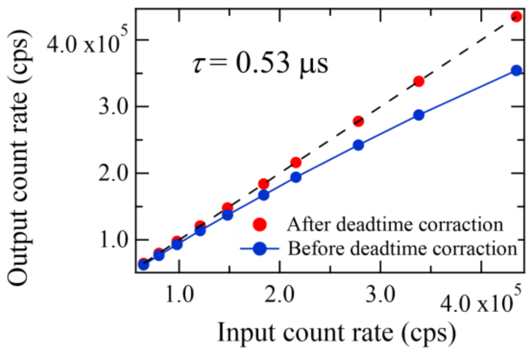


Fig. 2. Typical count rate characteristics of SDD and DSPs.

### 3. Efficiency of pellet sample preparation for XAS measurement

The typical procedure of preparing the sample for XAS measurement is as follows.

(1) Measure appropriate amounts of the sample and boron nitride (BN) powder using a scale.

(2) Mix the sample and BN powder using a pestle in a mortar.

(3) Form the mixed sample into a pellet using the tablet molding machine.

Usually, it takes 40–50 min to make one pellet sample. This procedure is a time-consuming task for users to prepare many samples, especially in the case of XAS measurement using an auto–sample changer [2]. Therefore, we developed a system for efficient sample preparation to reduce the burden of preparing samples for XAS measurement.

First, we introduced disposable tools to eliminate the need to clean laboratory equipment such as mortars and the tablet molding machine. Figure 3(a) shows a disposable bottle and a stirrer to mix the sample and BN powders. These powders are placed in the bottle with the stirrer and mixed by rotating the bottle as explained below (see Fig. 5). Figure 3(b) shows a disposable sample holder and a dice to make the pellet sample. The mixed sample is poured into the cylindrical part of the sample holder and then the dice is placed in it. By pressing the dice, the sample pellet is formed in the cylindrical part of the sample holder. The sample holder can be directly attached to the cassette holder of the auto–sample changer.

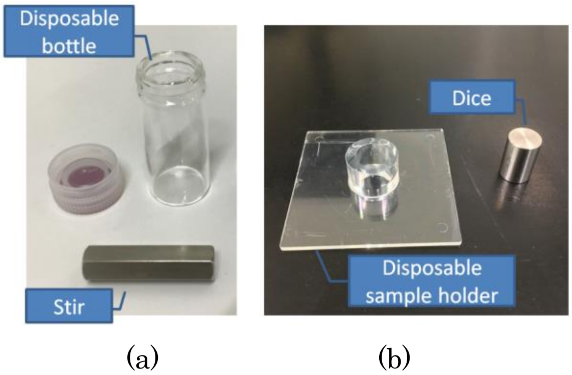


Fig. 3. (a) Disposable bottle and stirrer. (b) Disposable sample holder and dice.

Next, we introduced automated systems to (1) measure BN powder in the disposable bottle, (2) mix the sample and BN powder in the disposable bottle, and (3) form the mixed sample into a pellet. Figure 4 shows the automated powder dispensing system (made by METTLER TOLEDO). The system can automatically dispense BN powder into up to 30 bottles. It takes about 1 min to dispense 100 mg of BN powder into all 30 bottles. By using this system, we can save the time required to measure BN powder. Figure 5 shows the mix rotator that rotates the disposable bottles containing the powder and the stirrer to mix the powder. This mix rotator can rotate multiple bottles simultaneously. This procedure for mixing takes 30–40 min. Thus, we can reduce the time for mixing to about 2 min per sample. Figure 6 shows the automated pressing system for making pellets. On the positioning stage, a maximum of 30 sample holders containing the mixed sample and dices can be set as shown in Fig. 6. This system automatically presses the samples in the sample holders sequentially. This procedure takes 20–30 s per sample. With the use of these automated systems, the preparation time per sample can be reduced to 1/10 that of the manual procedure.

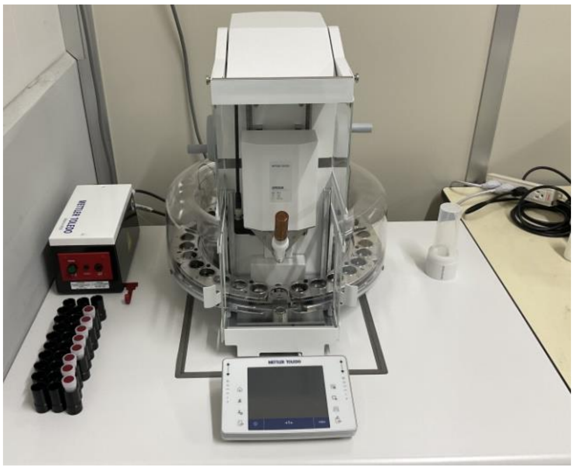

Fig. 4. Automated powder dispensing system (METTLER TOLEDO).

Fig. 5. Mix rotator.

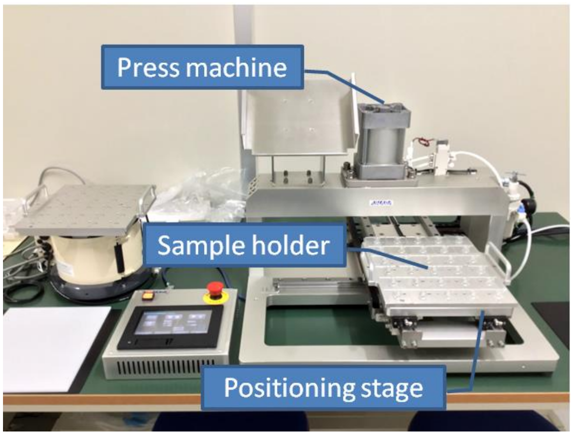


Fig. 6. Automated pressing system for making pellets.

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**References:**

[1] https://bl01b1.spring8.or.jp/tech/techinfo/SSD_DeadTime_Manual_010125.pdf

[2] Watanabe, T. Ofuchi, H. & Honma, T. (2020). *SPring-8/SACLA Annual Report FY2019*, 38–39.