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 FY2021, we developed an automatic pellet sample preparation system for XAS measurements.

2. Fully automatic pellet sample preparation for XAS measurement

In FY2021, we developed an automatic pellet sample preparation system for XAS measurement (Fig. 1). In FY2020, we improved the efficiency of pellet sample preparation for XAS measurement by installing an automatic powder dispensing system for preparing an appropriate amount of boron nitride (BN) powder as a binder (Fig. 2(a)), a mix rotator for mixing the sample and BN powder (Fig. 2(b)), and an automatic pressing system (Fig. 2(c)) for molding the mixed powder sample into pellets [1]. In FY2021, we arranged each device as a coherent system for automatic pellet sample preparation (Fig. 2) by combining them with a robot (Fig. 2(d)) and some ancillary equipment.

Figure 2 shows a schematic diagram of the automatic pellet sample preparation system for XAS measurement. The devices that compose this system are as follows.

(a) Automatic BN powder dispensing system

(b) Mix rotator

(c) Automatic pressing system

Fig. 1. Photograph of the automatic pellet sample preparation system for XAFS measurement.

(d) Robot

(e) Sample vial supplying device (instrument with 60 sample vials)

(f) Stirring rod supplying device (instrument with 60 stirring rods for mixing powder sample and BN powder)

(g) Sample vial cap supplying device (instrument with 60 caps of sample vials)

(h) Sample vial cap attaching device

(i) Sample vial cap detaching device

(j) Positioning stage supplying device (instrument for feeding three positioning stages equipped with 60 sample holders in total)

(k) Press rod setting device (instrument with 60 press rods for pressing the powder)

(l) Container for collecting the used cap of sample vial

(m) Container for collecting the used press rod

(n) Positioning stage collecting device

(o) Sample cartridges of an auto sample changer [2] for collecting the sample holders that have completed press molding

(p) Robot hands for various tasks

The outline of the operation sequence of the automatic sample preparation system is as follows.

(1) The robot sets the sample vials in the automatic BN powder dispensing system.

<Dispensing system operation>

(2) After dispensing, the robot takes out the sample vial, inserts the stirring rod, and caps the sample vial.

(3) The robot arranges the capped sample vials on the mix rotator.

<Mix rotator operation>

(4) The robot places the positioning stage with sample holders on the vibration device.

(5) After stirring, the robot removes the cap of the sample vial and transfers the sample to the sample holder on the positioning stage.

<Vibration device operation>

(6) After shaking, the robot ejects the sample vial including the stirring rod.

(7) The robot inserts the press rod into the sample holder and sets the positioning stage in the automatic pressing system.

<Pressing system operation>

(8) After pressurization, the robot takes out the press rods and arranges the sample holders that are fixed in the sample cartridges.

(9) The robot ejects the positioning stage.

One operation sequence explained above allows the preparation of any number of samples up to 20. The next series of operations could be performed in parallel using the waiting time during each series of operations, and a maximum of three sets could be advanced in parallel. Each device and instrument were arranged around the robot so that this series of operations can be performed in one place (Fig. 2).

The user only needs to put the required amount of powder sample for XAS measurement in a sample vial and to set the sample vial cap, stirring rod, and so forth to initiate the operation of the automatic pellet sample preparation system. The sample holder is then placed in the sample cartridges. The time required to pelletize 60 samples was less than 6 hours (less than 6 minutes per sample on average). The preparation time per sample has been reduced to 1/10 of that by the conventional method. In addition, the user can perform other tasks (e.g., experiment and analysis) while the automatic pellet sample preparation system is running.
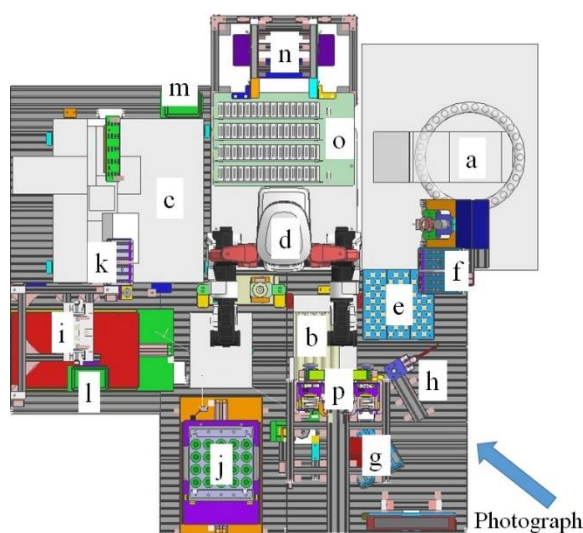

Fig. 2. Schematic illustration of the automatic pellet sample preparation system for XAFS measurement. The arrow indicates the viewpoint of Fig. 1.

Ofuchi Hironori and Honma Tetsuo

Industrial Application and Partnership Division, JASRI

References:

[1] Ofuchi, H. Watanabe, T. & Honma, T. (2021). *SPring-8/SACLA Annual Report FY2020*, 44–46.

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