

BL37XU

Trace Element Analysis

1. Introduction

BL37XU is a hard X-ray undulator beamline for trace element analysis and chemical/elemental imaging dedicated to various X-ray spectroscopy methods such as scanning X-ray micro-spectroscopy, full-field spectroscopic imaging, depth-resolved XAFS, and ultratrace element analysis. Using these methods, research is actively conducted to elucidate the properties and functions of materials through analyses of the morphology, element distribution, chemical state, and local structure. In FY2019, BL37XU operated smoothly and almost all users completed their user time as scheduled. To activate the use of high-energy X-rays, which is one of the strengths of BL37XU, a high-energy detector was installed and the instrument performance was upgraded as a scanning micro-spectroscopy system using a high-energy nano-focused beam in FY2019.

2. X-ray fluorescence detector system

The Kirkpatrick-Baez (KB) mirror focusing system was upgraded in FY2018 to enable the use of high-flux nano-focusing beams in the high-energy region^[1]. However, the conventional Ge solid-state detector (SSD) could not fully utilize the high-flux beams because of the increase of dead-time due to the increase of the counting rate. In FY2019, a high-counting Ge SSD and a high-counting digital signal processor (DSP) were installed to realize X-ray fluorescence analysis using a high-energy nano-focusing beam. Figure 1 shows the current layout of the scanning X-ray micro-spectroscopy measurement system.



Fig. 1. Layout of the scanning microscopic XRF/XAFS measurement system.

3. Ge SSD

The Ge SSD (EGX10-06-CP5-PLUS-WC; Mirion Technologies) is equipped with a CUBE preamplifier and has a high counting rate. The Ge SSD is cooled by electrical cooling (Cryo-Pulse 5 Plus), which does not require a liquid-nitrogen supply. All the waste heat from the detector is discharged to the chiller outside the hutch along with water cooling, improving the thermal stability in the experimental hutch. In addition, the Ge SSD can be used together with the 100-nm focused beam as there is no vibration associated with the air-cooling fan.

Figure 2 compares the spectrum of NIST SRM612 (a standard reference material consisting of soda-lime glass with about 40 ppm of various trace elements) to Ge SSD and the silicon drift detector (SDD). Both Ge SSD and SDD had similar detection sensitivities up to about 20 keV. The detection sensitivity of SDD decreased above 20 keV, whereas the Ge SSD had a high detection sensitivity, even above 30 keV. The increases in the

sensitivity and efficiency of scanning X-ray micro-spectroscopy measurements are attributed to the installation of the Ge SSD.

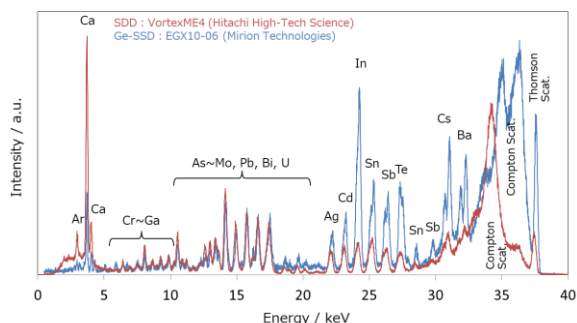


Fig. 2. XRF spectra of NIST SRM612 obtained by SSD (red) and GeSSD (blue).

4. DSP

The DSP with a pileup separator system (APU8011S; TechnoAP) was installed as the readout processor for the Ge SSD. Although dead time and signal pileup occur in the high-count region, the pileup signal is separated in real time by the pileup separator processor installed in the hardware, yielding correct energy information. As a result, high-counting measurements can be achieved.

By combining these systems with the existing on-the-fly scan system, high-speed 2D XAFS/XRF can measure not only the region of interest (ROI) but also the full X-ray fluorescence spectrum.

Kiyofumi Nitta and Oki Sekizawa

Spectroscopic Analysis Group I, Spectroscopy and Imaging Division, Center for Synchrotron Radiation Research, JASRI

Reference:

- [1] K. Nitta and O. Sekizawa, *SPring-8/SACLA Annual Report 2018*, 64 (2019).