BL28XU
Advanced Batteries

1. Introduction

BL28XU is managed and operated by Kyoto University for realizing technological innovations in rechargeable batteries. It was renamed “Advanced Batteries” from “RISING2” in FY2021. The RISING2 project ran from FY2016 to FY2020 as a contract research project of the New Energy and Industrial Technology Development Organization (NEDO) to promote technology development for practical uses of storage batteries. The project exclusively used the beamline for this purpose. In FY2021, the RISING3 project was launched as a successor to RISING2. The project focuses on the two types of post-lithium-ion battery (LIB) system: (1) fluoride batteries, which show great potential in terms of both energy density and safety, and are based on highly original technologies developed in Japan, and (2) zinc-anode batteries, which offer significant safety advantages and cost benefits. Since FY2021, the RISING3 project has used most of the beamtime of BL28XU for the research and development of these battery systems.

The main subjects of the current and previous projects that are being conducted in the beamline are as follows: (1) the elucidation of reaction distribution generation factors, (2) the analysis of active material reactions and nonequilibrium behaviors, (3) the elucidation of electrode/electrolyte interface phenomena, (4) the elucidation of the formation mechanism of random materials such as an electrolytic solution and electrolytes at the electrode interface, and (5) the elucidation of thermodynamic or physical instability phenomena inside the storage batteries. Measurement techniques for in situ observations of the reaction inside storage batteries via X-ray diffraction (XRD), confocal X-ray diffraction, X-ray absorption spectroscopy (XAS), and hard X-ray photoelectron spectroscopy (HAXPES) have been mainly employed for this purpose.

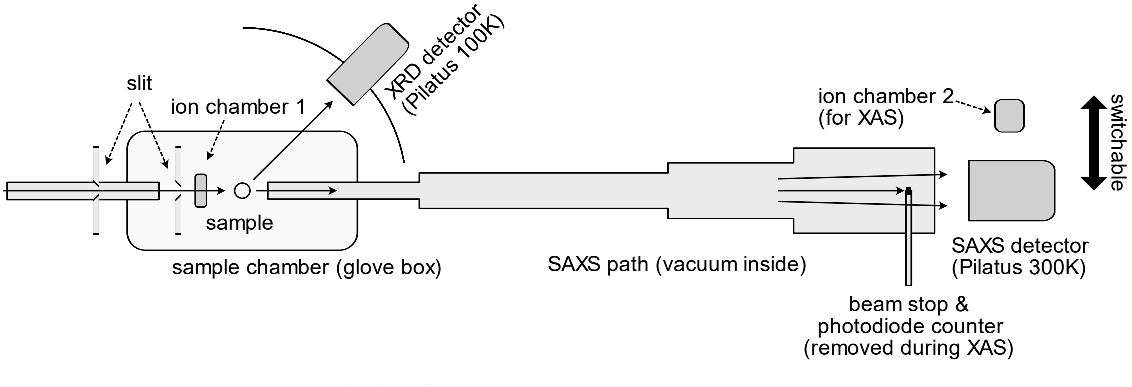

Fig. 1. Schematic drawing of the XRD/XAS/SAXS system at BL28XU.

2. Development of Small-Angle X-ray scattering (SAXS) measurement system

During the charge–discharge reaction of a battery, multiple changes such as chemical reactions, valence changes, and structural changes occur concertedly and simultaneously. Among such changes, structural change at the nanometer scale, e.g., particle formation and growth of conversion-type electrode materials, could not be probed by the methods the beamline had been offering. Therefore, we built a SAXS measurement system with which XRD and XAS measurements can to be operated quasi-simultaneously. The camera length of the SAXS setup is fixed at 3 meters owing to optical and spatial constraints. The schematic drawing of the system is shown in Fig. 1. With this system, a sequence of XRD, XAS, and SAXS measurements can be performed within a few minutes. We expect that this system will contribute to gaining a better understanding of the reaction mechanism of innovative batteries.

The upstream components of the SAXS system between the focusing mirrors and the sample stage were initially shared with the XRD setup; the beam path at the shutter and attenuators was exposed to air and the other path was in vacuum ducts with both ends covered with Kapton films. With this setup at the photon energy $E = 9$ keV, the parasitic scattering hindered expected signals where $q < 0.05$ nm^{-1}. In order to reduce the parasitic scattering, we made the following improvements: (1) introduction of a scatterless slit adequate for the photon energy typically used in SAXS measurements, (2) introduction of a new in-vacuum unit of a shutter and attenuators, which led the upstream beam to just before the sample totally in vacuum without the need for Kapton films or passing through air, and (3) adjustment of the slit position. Figure 2 shows a scattering pattern when no sample was inserted, i.e., the system background signal, at $E = 9$ keV. Parasitic scattering was greatly decreased and the lower end of the effective q-range was extended to 0.02 nm^{-1} or less.

This new upstream system for SAXS is built on a horizontally slidable base stage and can be replaced with the XRD setup in a short time.

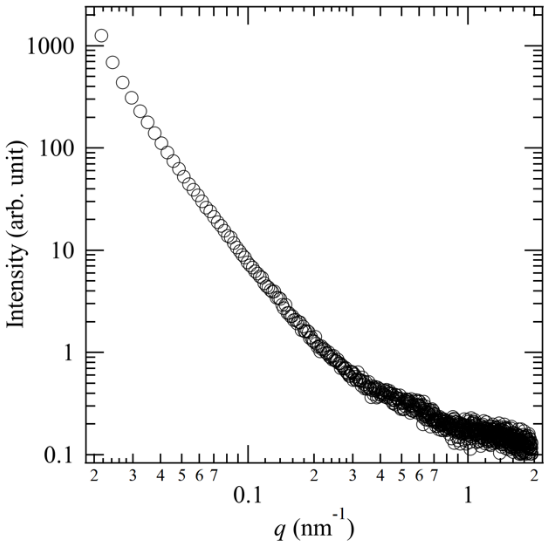

Fig. 2. Scattering pattern after the reduction of parasitic scattering. $E = 9$ keV.

Acknowledgments

This work is based on the results of a project entitled, “Research and Development Initiative for Scientific Innovation of New Generation Batteries 3 (RISING3)”, JPNP21006, commissioned by the New Energy and Industrial Technology Development Organization (NEDO). The investigation into the reduction of parasitic scattering was carried out in collaboration with Prof. Yohei Nakanishi, Prof. Motoki Shibata, Prof. Tsukasa Miyazaki, and Prof. Mikihito Takenaka (Kyoto University).

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