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 the majority of the beamtime of BL28XU for the research and development of these battery systems.

The main subjects of the RISING3 project 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.

2. Development of Ultra-small-angle X-ray scattering (USAXS) 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. To understand the whole picture of the concerted phenomena, combined use of several measurement methods is desirable. For this purpose, a combinational measurement of XRD and XAS had been often employed in the beamline. However, by this method the structural change at the nanometer scale, e.g., particle formation and growth of conversion-type electrode materials could not be probed. Therefore, the author recently built a small-angle X-ray scattering (SAXS) measurement system with which XRD and XAS measurements can be operated quasi-simultaneously[1]. With this system, a sequence of XRD, XAS, and SAXS measurements can be performed within a few minutes.

The system began to be employed for structure analyses of various materials soon after its development. The system contributed to elucidation of the structure model of a lithium-ion conversion battery with a FeF_3 positive electrode[2]. While the system has been actively used by users, it has turned out that there is also a demand for smaller q range that the system does not cover. However, the camera length of the SAXS path is fixed at 3 meters and it is difficult to extend it, owing to spatial constraints of the experimental hutch 1 (EH1), where the system is installed.

To extend the camera length, a USAXS measurement system was newly built. The system is schematically depicted in Fig. 1[3]. The main difference from the SAXS system is that the vacuum duct located between the sample and the detector is extended to the experimental hutch 2 (EH2). This leads the sample-detector length of 9.1 meters. While this kind of setup was probably not considered when the hatches were built, the duct embedded in the wall between EH1 and EH2 is wide enough and thus, we could build the setup without processing of radiation shields. With this setup, a minimum q = 0.0069 nm^{-1} was attained at photon energy E = 9 keV.

The beamline equips a channel-cut compact monochromator, which allows quick change of the energy of the incident X-rays. Taking advantages of this characteristics, one of the beamline user groups have challenged time-resolved anomalous USAXS measurement[3]. Anomalous USAXS measurement is a contrast variation technique that uses the scattered intensity of USAXS with incident X-ray energy near the absorption edges of one component in the multi-component system. Among a limited number of USAXS beamlines in the world, the

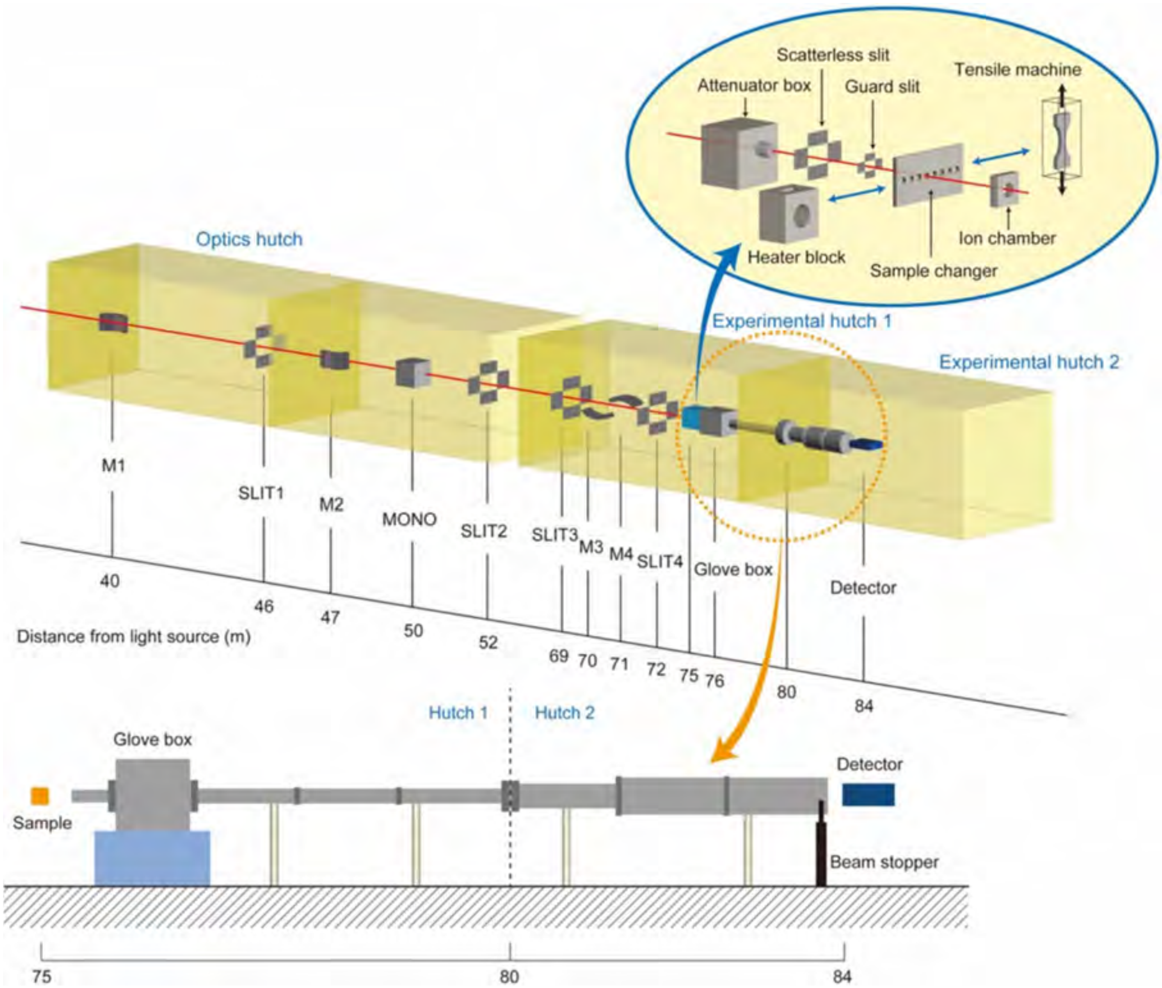

Fig. 1 Schematic illustration of the USAXS setup at the BL28XU beamline. [3]

capability of excellent time-resolution in energy-modulated measurements has made the system especially unique. The capability of the system to exploit element-specific studies in complex materials has attracted the interest of researchers.[4]

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 author thanks Prof. Yohei Nakanishi, Prof. Motoki Shibata, Prof. Tsukasa Miyazaki, and Prof. Mikihito Takenaka (Kyoto University) for the contribution in design and construction of the USAXS setup. The author acknowledges support from the Quantum Beam Analyses Alliance (QBAA).

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