BL08W
High Energy Inelastic Scattering

Introduction

BL08W is the beamline that can deliver the highest energy X-rays, the range of which is from 100 to 300 keV, emitted by the only wiggler source at SPring-8 [1,2]. This beamline is used in several research fields, including Compton scattering, high-energy X-ray diffraction [3], and high-energy X-ray fluorescence analysis [4]. Three methods of Compton scattering can be performed in this beamline. The first method is magnetic Compton scattering (MCP) for studying magnetic states [5]. The second method is high-resolution Compton scattering (HCP) with a high momentum resolution of 0.1 a.u. for studying electronic states and fermiology [6]. The third method is Compton scattering imaging (CSI) using a CdTe two-dimensional detector (HEXITEC) having energy dispersive capability in each pixel [7]. Also, structural studies of disordered materials by pair distribution function analysis using high-energy X-ray diffraction and studies on cultural properties by high-energy X-ray fluorescence analysis (XRF) can be performed. In addition to the above, simultaneous measurement of X-ray diffraction and Compton scattering imaging is now possible. This annual report describes this method.

Simultaneous measurement of XRD and CSI

Although BL08W is capable of XRD as well as CSI, simultaneous measurement of them has not been performed so far. We have developed a new system for simultaneous measurement of CSI and XRD. This method makes it possible to simultaneously obtain information on the electronic state and crystal structure of battery materials in a metal container by simultaneously capturing the internal state and temporal changes during power generation using CSI and XRD. The geometry of this method allows us to restrict the measurement position for both CSI and XRD. This makes it possible, for instance in battery materials, to exclude Compton scattered and diffracted X-rays emitted from the battery casing.

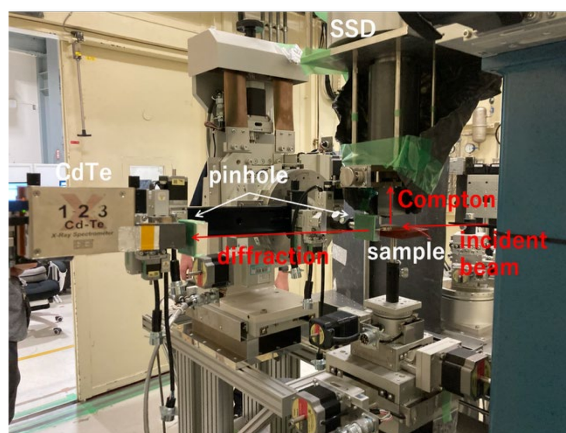

Fig. 1. The condition of simultaneous measurement of XRD and CSI.

Figure 1 shows the experimental setup of the simultaneous measurement system. A 9-element Ge solid-state detector (SSD) is installed in the vertical direction to perform Compton scattering measurements at a scattering angle of around 90°. The field of view is confined to 1 mm² by a collimator. XRD measurements are conducted by mounting two pinholes and a CdTe detector on a rail, and rotating the rail in the vertical direction. The two pinholes, each with a size of 100 μm, limit the field of view to a range comparable to, or slightly

larger than, that obtained in CSI measurements with an SSD. The positions of the pinholes and the detector can be adjusted along the rail. For example, when the first pinhole is placed 1 mm from the sample and the second pinhole is positioned 300 mm downstream from the first, the field of view at a scattering angle of 5° is approximately 1 mm². Note that at scattering angles lower than this, the field of view increases, while at higher angles it decreases.

The overall measurement time is determined by the XRD measurement. For the XRD measurements, data were collected over a scattering angle range of 1°–30° with a step interval of 0.01° and a counting time of 10 s per point, resulting in a total measurement time of approximately 8 hours.

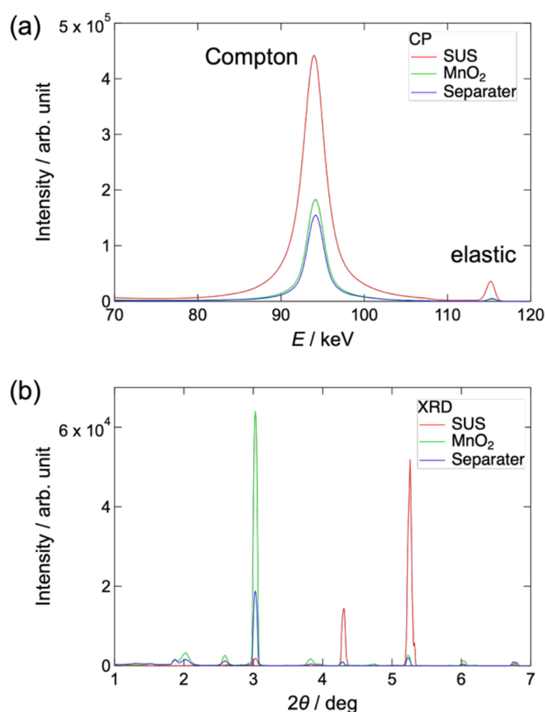

Fig. 2. Simultaneous CP (a) and XRD (b) data of coin cell (CR2032).

As an example, Fig. 2 shows simultaneous CP and XRD data for a coin cell measured with a 115.6 keV incident beam. Compton peaks and diffraction patterns were successfully obtained simultaneously, and they differ depending on the location of observation in the coin cell.

In situ measurements of battery materials and related samples are accompanied by continuous variations in Li content as well as degradation effects. By simultaneously probing changes in the electronic state (including Li content) using Compton scattering and monitoring structural changes using XRD, highly reliable data can be obtained.

This experimental method has been used since the 2024B term. Future work includes improving the angular resolution and compensating for shifts in the diffraction angles at high angles and reducing the measurement time in XRD.

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