Construction of multiscale structure measuring system on iMATERIA

Ibaraki Univ., Haruyuki Takahashi

1. Introduction

Solid oxide fuel cells (SOFC) are being applied for the power source not only at commercial use but consumer use. It is important to evaluate the degradation of SOFC in the severe operating circumstances. SOFC cells consist of crystalline solid electrolytes, such as YSZ and Ni-cermet electrodes having microstructure. The crystal structures of electrolytes and electrodes give information on precipitates, the strain of crystal lattice, the lattice defects and so on. On the other hand, the microstructure of electrodes is obtained from the small angle scattering measurements. The multiscale structure ranging from 10 to 0.01 nm is useful for the crystalline materials with microstructure, such as fuel cells, lithium-ion batteries, composites, and catalysts.

The iMATERIA (BL20) has the small angle and backward detectors suitable for the multiscale structure measurements. The small angle scattering and the diffraction experiments are however, performed exclusively at present. The aim of the present investigation is to construct the multiscale structure measuring system using the novel high-neutron-absorbing glasses for the simultaneous acquisition of both small angle scattering and high-resolution diffraction data.

2. Experiment

Stable gadrinium-containing lithium borosilicate glasses were synthesized successfully. The first slit using the glass was set at the upper steam side of the sample changer. The first slit leads the neutron beam into the sample cell holder and suppresses the diffraction from the sample changer elements. The second slit using the glass was set at the upper steam side of the sample cell. The second slit focuses the neutron beam suitable to the small angle scattering and suppresses the diffraction from the sample cell. The first and the second slits also forms the path to the backscattering diffraction. The Si powder supplied from NIST and the nano-powdered silica glass are used as standard samples. The standard samples were measured to verify the reproducibility of the multiscale structure data in comparison with the normal configuration in the small angle scattering and the diffraction measurements.

3. Results

The small angle part of multiscale profile for empty cell is shown in Fig. 1. The empty cell is composed of two aluminium sheets of 0.1mm thick. The powdered samples or the smaller solid samples than sample holder window diameter is fixed between aluminium sheets. The small angle scattering for empty cell includes the contribution from aluminium sheets. So, observed small angle scattering is the superposition of the small angle scattering of sample and empty cell. The high-resolution backscattering diffraction profile for empty cell is shown in Fig.2. All of diffraction peaks were assigned to aluminium. Although the sample cell is made of

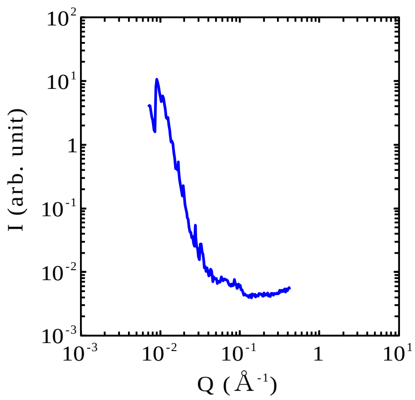

Fig. 1 Small angle scattering from empty cell.

aluminium, the open cell without aluminium sheets gives no diffraction peaks. The diffraction from aluminium comes from aluminium sheets. Moreover, cadmium plate is used for the neutron absorber at the sample changer. The diffraction from cadmium is not detected in Fig.2. It was verified that the diffraction from cadmium plate and aluminium sample cell was effectively suppressed by the first and the second glass slits, respectively.

The multiscale scattering profile of Si is shown in Fig. 3. Neutron scattering from 0.007 to 20Å^{-1} was observed using the small angle, the low angle, and the backward detectors. The small angle scattering comes from the grains of Si powder. The small angle scattering intensity from the sample is much higher than that from empty cell shown in Fig. 1. The peak at 2Å^{-1} is (111) diffraction of Si from low angle detector. The oscillating look of diffraction profile above 3Å^{-1} is responsible for a log-log plot. Normal linear plot of diffraction is shown in Fig. 4. High-resolution diffraction profile of Si is observed by backward detector. The powder diffraction profile of Si is available for the Rietveld structure refinement. Calculated crystal structure factor and diffraction profile coincides well with observed one.

Figure 5 shows the small scattering part of multiscale structure for the nano-powdered silica glass. The scattering profile reproduce with that from the normal coordination of small angle scattering measurement of iMATERIA.

4. Conclusion

The multiscale structure measuring system from 0.007 to 20 Å^{-1} using high-neutron-absorbing glass slits has been constructed on iMATERIA. The validity of observed small angle scattering and high-resolution diffraction parts were verified by Si powder and nano-powdered silica glass. The multiscale structure measuring system is expected to apply to the structure analysis of electrodes and electrolytes of fuel cells, composites, catalysts, and nano precipitated alloys.

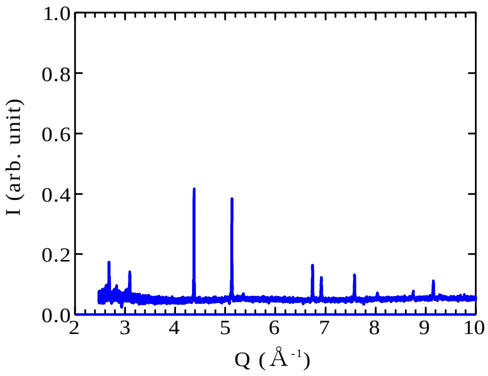

Fig. 2 Diffraction profile for empty cell.

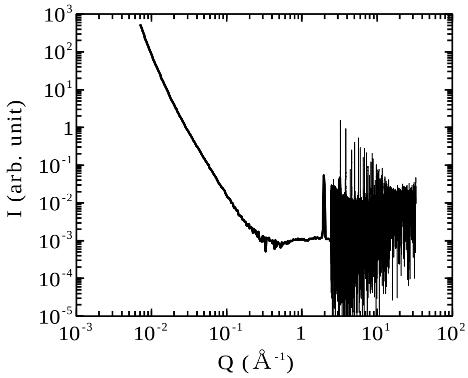

Fig. 3 Multiscale structure profile for Si.

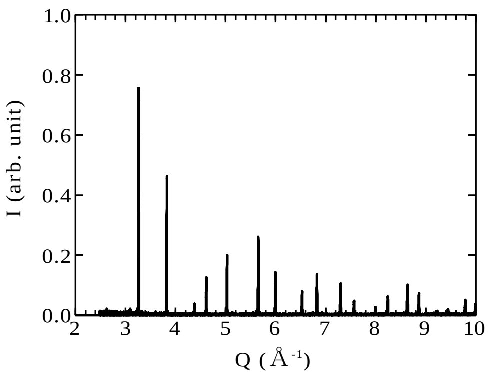

Fig. 4 Diffraction profile for Si.

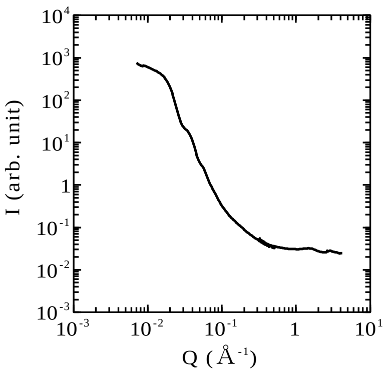

Fig. 5 Small angle scattering for nano-powdered silica glass.