MERIT internship (domestic) report

Department of Materials Engineering
Mizuguchi lab, D2
MERIT 6th
Katsuaki Nakazawa

Implementation period
July 1, 2019-July 31, 2019

Acceptance
National Institute for Materials Science (NIMS) Advanced Materials Analysis Center, Production Environment Measurement Technology Development Group
(Group leader: Dr. Kazutaka Mitsuishi)

Theme
Investigating a new analysis method using 4D-Canvas for fine structures

Contents
NIMS, the recipient, introduced 4D-Canvas, the latest electron detector for scanning transmission electron microscopy (STEM), this year. A feature of this 4D-Canvas is high-speed signal readout. Conventional electron detectors can only obtain the accumulated amount of electrons at each scanning point (x, y). However, 4D-Canvas can acquire distribution of scattered electrons, that is, a diffraction pattern (kx, ky) by the high-speed signal readout. This type of STEM that acquires 4D data is called 4D-STEM. The acquisition of this 4D data I (x, y, kx, ky) has a possibility to obtain a new fine structure.

As a result of this 4-dimensional data and the ability to acquire more information, the degree of freedom in analysis has increased. On the other hand, sufficient research has not been conducted in the past. The analysis method peculiar to 4D-STEM has not been fully developed yet because the 4D-data dimension is difficult to interpret intuitively, the amount of data is enormous, the equipment is very expensive and it has not spread to other laboratories.

Therefore, in this research project, I worked on the development of a new microstructural analysis method utilizing the characteristics of 4D-STEM

.

This intern mainly developed or implemented three methods.

1. Installation of Ptychography method.

The Ptychography method is a high-resolution phase recovery method. I wrote the script for

ptychography in python.

2. Determination of thickness and composition at low damage using electron scattering

In conventional STEM, the thickness and composition of samples have been measured mainly by spectroscopic methods such as STEM-EELS and STEM-EDX. However, these spectroscopic methods require a large amount of electron beam irradiation, making it difficult to observe materials that are sensitive to electron beams. We focused on the scattering angle depending on the atomic species. The difference in scattering angle was detected by 4D-STEM and the composition distribution was measured. In addition, since the diffraction pattern changes depending on the thickness, the thickness was measured by using this change. As a result, it was confirmed that the composition and thickness could be measured with an electron beam dose of 1/10 or less compared to STEM-EELS.

3. Observation of the glass-crystal phase transition process by in situ observation.

In addition to 4D-STEM, an in-situ observation heating holder was used to analyze the crystallization process by observing changes in diffraction patterns. In addition, by using the method developed in 2. together, the relationship between the crystallization position and the composition was analyzed.

・ Acknowledgments

I would like to thank Dr. Mitsuishi, who taught me how to operate STEM and 4D-STEM and the theory of Ptychography, and Ms. Shiomi, who prepared for the internship. I would also like to thank Professor Teruyasu Mizoguchi, Professor Hiroshi Okamoto and MERIT program for giving me this opportunity for this internship.