Morphology analyses of micro-crystals using electron back-scattering pattern technique and stereographic SEM observation

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It is well known that morphology of minerals often depends on the environment where the minerals grow. Shortly speaking, the morphology of crystals is the result of different growth rates along various crystal directions. Hence, information of the mineral morphology is important to understand the environment where the minerals formed, as well as crystal chemistry of the minerals. Especially in the biomineralization processes, new insights can be deduced from the morphology analyses because organic molecules often interact with specific surfaces of biominerals and change their shapes drastically. The morphology of magnetite in the Mars meteorite, which has a uni-axially elongated shape just like that in magnetotactic bacteria, is a good example.

Historically morphologies of minerals have been determined using a reflecting goniometer and single X-ray diffraction. However, this technique is not applicable to small crystals less than several tenth millimeters. Transmission electron microscopy (TEM) is a unique tool to determine the morphology of such small crystals, using magnified images and corresponding electron diffraction. The TEM images, however, are basically shadowgraphs and it is often difficult to recognize three-dimensional crystal morphologies, as compared with recent high-resolution scanning electron microscopy (SEM).

Electron back-scattering diffraction (EBSD) technique can obtain crystallographic information (crystalline phase, crystal orientation, etc.) from the specimen in an SEM, by analyzing Kikuchi patterns. We are now developing a technique to analyze morphology of micro-crystals by the combination of EBSD and stereographic SEM observation.

The system is composed of a Hitachi S-4500 SEM with cold filed-emission electron source and a ThermoNoran Phase-ID EBSD detector. Obtained Kikuchi-patterns are analyzed by self-made software. The SEM has a specimen stage that can tilt specimen up to 45 degrees along a horizontal axis. Two SEM photographs of a crystal with different tilt angles are recorded and the angles from the tilt axis for a crystal edge are measured from each photograph. Then the polar coordinates, where the polar direction corresponds to that toward the EBSD detector, of the edge are calculated from the two measured angles by a successive approximation method. In the next, an EBSD pattern is obtained from the crystal and the relationship between the crystal-axes and the polar coordinate is determined. From these results, the crystallographic direction ([u,v,w]) of the edge is readily calculated.

In this presentation, the application of the method to the morphology analyses of the polymorphs of $CaCO_3$ (calcite, aragonite and vaterite) is reported, and the accuracy and limitation of the method are discussed.