Morphological characteristics of ordered kaolinite: Investigation using electron back-scattered diffraction

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ABSTRACT

Morphology of kaolinite crystals with high-crystallinity (Keokuk, Iowa) has been investigated by electron back-scattered diffraction (EBSD) and high-resolution scanning electron microscopy (SEM). EBSD patterns from the specimen generally correspond to those expected from ordered kaolinite. Elongated hexagonal crystals always show the a-axis parallel to the elongated direction. The side-facets of these crystals are in parallel to the c-axis, which is inclined by about 15° from the normal to the basal plane. The Miller indices of the side-facets are exactly ±(110), ±(1T0), and ±(010). A facet indexed as ±(130) is developed in some crystals. These morphological characteristics must be reflected in the ordered stacking sequences (the position of the octahedral vacancy site and the direction of the interlayer shift) of the Keokuk kaolinite. Inversely, the crystallinity of individual kaolinite grains may be evaluated from their morphology in a SEM. The feasibility to discriminate the enantiomers in kaolinite using EBSD is also described.

INTRODUCTION

Morphological features of minerals are closely related to their crystal structures, compositions, impurities, and their growth conditions (e.g., Bunn 1961). In general, crystal growth precedes developing specific facets to minimize the surface energy of the crystal (Toscheck 1973). Recently, atomistic simulations have been successful to some extent in predicting the surface structure and morphology of minerals (e.g., Watson et al. 1997; Baetzold and Yang 2003). Conversely, information concerning the surface energies or the surface structures of the specific facets may be obtained from the observed morphology (Sunagawa et al. 1995).

Most phyllosilicates have morphology with predominance of the basal plane. Other facets determine the apparent shape of a phyllosilicate, for example, lath, rhombus, hexagonal or irregular forms. Phyllosilicates commonly have variable stacking sequences of the unit layers, which results in a variety of polytypes and/or stacking faults. It is not well understood how these stacking sequences are reflected in the morphology of the minerals. Güven et al. (1980), for example, investigated the relationship between the polytypes of illite and their morphology by using transmission electron microscopy (TEM) and selected area diffraction (SAD). They reported that lath-shaped illite has a 1M stacking sequence and is elongated along its a-axis parallel to the direction of the lateral shift between adjacent 2:1 layers. From this result lath or ribbon-like shapes of phyllosilicates may imply an ordered and simple (e.g., one-layer periodicity) stacking sequence (Tsipursky and Drits 1984). However, more experimental results are necessary to confirm this speculation.

For the analyses of crystal morphology, it is necessary to determine the relationship between crystal form and crystallographic orientation. For small crystals such as those of clay minerals, the combination of TEM imaging and corresponding SAD has been used for such studies. It is, however, difficult to establish three-dimensional morphology by using TEM, because TEM images are only the projections of specimens with the transmitted electrons. Recently, TEM-CT (computed tomography) for analyzing three-dimensional morphology is being developed (Midgley and Weyland 2003), but still is not common. Generally, the incident beam should be parallel to the basal plane to determine the stacking sequence in phyllosilicates by SAD. On the other hand, the beam direction should be close to the normal of the basal plane to understand their morphology by TEM imaging. As the maximum angle for specimen tilt is limited, we typically cannot obtain these two orientations for the same crystal in a TEM. Consequently, TEM and SAD may not be the best technique for the morphological analyses of phyllosilicates.

On the other hand, scanning electron microscopy (SEM) is effective for observing stereoscopically the morphology of the small crystals. Development of the field-emission gun considerably improved the resolution of SEM owing to a brighter electron source with a smaller energy spread. Using electron back-scattered diffraction (EBSD), crystalline materials, and their orientation can be determined in a SEM. Consequently, it has become possible to determine the relationship between the crystal form and orientation of individual grains by combining

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