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Electron microscopic study of the dehydration of diaspore

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ABSTRACT

The thermal transformation of natural, single-crystal diaspore (D) to corundum (C) and water has been analyzed in detail by means of transmission electron microscopy (TEM). Pieces of single-crystal diaspore were partially dehydrated at temperatures ranging from 400 to 600 °C and were prepared in cross-section for TEM studies. The transformation of diaspore was studied at the faces $(100)_D$, $(010)_D$, and $(001)_D$, with special attention to the microstructural details at the reaction interface.

Irrespective of the transformation direction, the dehydration product is highly porous. Nanometer-sized pores form two-dimensional arrays arranged parallel to the basal plane of corundum forming lamellae that are separated by thin regions of dense corundum. The periodicity of the lamellar structure is 3.7 nm, which causes distinct satellite reflections in diffraction patterns in the case of dehydration at moderate temperatures (<450 °C). At higher temperatures, the degree of regularity, as well as the satellite reflection intensities, decrease.

During transformation, the diaspore crystal cleaves parallel to $(010)_D$, and micro-cracks are developed parallel to $(100)_D$ and to $(001)_D$ in the corundum due to the misfit of the lattices. The entire crack system as well as the lamellar pore system play an important role in the dehydration process because they serve as effective transport paths for the water vapor. The rapid dehydration at the $(010)_D$ faces is attributed to fast-propagating cracks along $[010]_D$. The slow transformation processes along $[100]_D$ and $[001]_D$ are discussed in terms of difficulties in crack propagation and in generating suitable crack systems, respectively.