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In situ study of the goethite-hematite phase transformation by real time synchrotron powder diffraction

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ABSTRACT

The temperature induced goethite-hematite phase transformation that occurs at about 250 °C was studied using in situ synchrotron X-ray powder diffraction with a capillary Debye-Scherrer geometry and a translating image plate system (TIPS). To our knowledge, this is the first time the goethite-hematite transformation has been investigated in real time. The sample was a pure, synthetic, stoichiometric goethite with 1 μm long needle-shaped crystals. The microstructural characterization showed that the sample was well crystallized. The Rietveld refinement of 30 powder patterns extracted from the image in the range 25–800 °C demonstrates that an intermediate phase with non-stoichiometric composition (*"protohematite"*) forms after the decomposition of goethite. The cell parameter *b* of goethite dramatically decreased during the phase transformation while *a* and *c* instead continued to increase. Protohematite is iron-deficient and retains residual hydroxyls for charge balance. With temperature protohematite progressively transforms into hematite. Empty layers (pores) are consequently formed about the hematite clusters. The distribution of iron vacancies was modeled in the powder patterns with stacking faults that were simulated using anisotropic broadening coefficients of the pseudo-Voigt profile function. Its disappearance with temperature was effectively followed with a decrease of the density of stacking faults.