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Structure of synthetic 2-line ferrihydrite by electron nanodiffraction

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ABSTRACT

Comparison of experimental single-crystal electron diffraction patterns of synthetic two-line ferrihydrite (2LFh) with simulated single-crystal electron-diffraction patterns indicates that a synthetic 2LFh sample contains highly disordered material and nanocrystals with structures based on hexagonal (ABAB) and cubic (ABC) stacking of close-packed layers of O^{2-} and OH^{-} ions. An apparently continuous variation in ordering exists between the highly disordered material and each of the crystalline structures, suggesting that both nanocrystalline structures represent local extremes of three-dimensional ordering. Experimental diffraction patterns were obtained using electron nanodiffraction, a technique in which the finely focused beam from a field-emission gun in an electron microscope can be used to produce diffraction patterns from areas <1 nm across. Nanodiffraction patterns from the highly disordered material have diffuse streaks rather than distinct reflections, and are consistent with a two-dimensional structure that consists of close-packed anionic layers with essentially complete stacking disorder and nearly random distribution of Fe atoms. The structure with cubic stacking is similar to maghemite and has ~25% of the Fe in tetrahedral sites. The structure with hexagonal stacking consists of double chains of face-sharing Fe octahedra; each octahedron shares one face, two edges, and three corners with adjacent octahedra. Previous results from transmission electron microscopy, powder X-ray and electron diffraction, and synchrotron-based techniques reflect the overall high degree of structural disorder rather than the characteristics of the maghemite-like and double-chain structures.