Off-axis spin orientation in goethite nanoparticles

Neutron diffraction is a powerful technique for determining the magnetic structure of antiferromagnetic materials. However, for some of these, determining the detailed magnetic structure remains a challenge. In goethite ($\alpha$-FeOOH) the antiferromagnetic unit cell coincides with the chemical unit cell and, consequently, nuclear and magnetic diffraction peaks occur at the same positions. Analysis of diffraction data from goethite is further complicated by finite-size peak broadening, resulting from goethite commonly occurring in nanocrystalline form. For these reasons, determining the magnetic structure of goethite has been challenging, and few detailed studies have been published. Even today, not all aspects of the magnetic structure are well established. Here, we investigate the magnetic structure of three samples of goethite nanoparticles with polarized neutron powder diffraction (xyz-polarization analysis). Two samples consist of acicular goethite particles that are approximately 40 nm long and with different thicknesses, and one sample consists of pseudospherical particles with a diameter of approximately 5 nm. The larger particles consist of several crystallites whereas the 5-nm particles are mostly single crystalline. The polarization analysis enables us to separate magnetic scattering from nuclear and spin-incoherent scattering, resulting in data that can readily be analyzed. For the two samples with the larger particle size, we find nuclear correlation lengths in the [100] direction that are approximately 3 nm longer than the magnetic correlation lengths, indicating a magnetically disordered layer perpendicular to the antiferromagnetic modulation direction. We find no evidence of a magnetically disordered surface layer in the 5-nm particles. We find the magnetic structure to be antiferromagnetic but, in contrast to most previous studies, we find the spin orientation in all three samples to make an angle of 28-30° with respect to the crystallographic b axis.

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