A quantitative phase map of nanometeric Iron(III) oxide

SEUNGYEOL LEE AND HUIFANG XU[*]

NASA Astrobiology Institute, Department of Geoscience, University of Wisconsin – Madison, Madison, Wisconsin 53706 (*correspondence: hfxu@geology.wisc.edu)

There is great interest in nanosized iron oxides, because of their technological application and prospective utilization due to its magnetic property. Among iron oxides, ε-Fe_2O_3 is considered as a remarkable phase due to a giant coercive field at room temperature, coupled magneto-electric features, a significant ferromagnetic resonance that is a distinctive characteristic in any other metal oxides [1, 2]. We present the first quantitative phase map for ε-Fe_2O_3 via γ-ε-α pathway, based on X-ray powder diffraction (XRD) and high-resolution transmission electron microscopy (HRTEM). Our recent study shows the ε-Fe_2O_3 nano-minerals occur in oxidized basalt.

The nanosized Fe_2O_3 polymorphs are synthesized by a thermal decomposition method between 800 ºC to 1100 ºC [3]. The method increases the thermal stability of ε-Fe_2O_3 through SiO_2 matrix [3]. The polymorphous transformations of Fe_2O_3 are identified by XRD patterns between 850ºC and 1000ºC. The individual nanoparticles are examined using HRTEM to study their structure, size, and crystal interfaces. The cubic crystal structure of γ-Fe_2O_3, with the average crystal size of ~5 nm is thermally unstable at elevated temperature. The γ-Fe_2O_3 to ε-Fe_2O_3 phase transition takes place once the γ-Fe_2O_3 nanoparticles reach a certain critical size between ~10 nm at 1000 ºC and ~13 nm at 850 ºC. The orthorhombic crystal structure of ε-Fe_2O_3 are typically ~50 nm and ranges from ~10 nm to ~ 200 nm. The shape of ε-Fe_2O_3 is elongated along *a*-axis with combinations of sphenoid, pinacoid and pedion forms. The ε-Fe_2O_3 nanoparticles have (110) or (-110) twinning with composition plans of {130} and (100) due to pseudo-hexagonal symmetry of the crystal structure. The critical size of ε-Fe_2O_3 to α-Fe_2O_3 phase conversion is approximately between ~150 nm at 1000 ºC and ~100 nm at 850 ºC in amorpous silica matrix. The rhombohedrally centered hexagonal structure of α-Fe_2O_3 is most stable phase of all three polymorphs of α-Fe_2O_3. HRTEM images show crystal interfaces of ε-Fe_2O_3 and α-Fe_2O_3 along *c*-axis. It is proposed that the hexagonal packing of ε-Fe_2O_3 (001) surface could serve as the substrate / interface for α-Fe_2O_3.

[1] Gich *et al*. (2005) J. Appl. Phys. 98, 044703. [2] Tucek *et al*. (2010) Chem. Mater. 22, 6483-6505. [3] Kelm & Mader (2005) Anorg. Allg. Chem. 631, 2383-2389.