Oxidation state investigation of iron oxide minerals by ELNES

U. Golla-Schindler[1], R. Hinrichs[1,2], and A. Putnis[1]

[1] Institut für Mineralogie, University of Muenster, Germany
[2] Instituto de Geociências, Universidade Federal do Rio Grande do Sul, Porto Alegre, Brazil

One excellent technique to determine the oxidation states of different mineral phases is to investigate the ELNES (Electron Loss Near Edge Structure). Especially for iron oxide minerals the near edge structure of the O-K edge [1] and the Fe L-edge can be used to determine the oxidation state, the iron oxide phase, and additionally the chemical composition. This work deals with iron bearing minerals in two systems.
The first system was composed of colloidal magnetic iron-based nanoparticles. These nanoparticles are used for medical purposes to enhance the contrast between normal and diseased tissues or to indicate organ functions or blood flow. Their structure is supposed to consist of an oxidized rim and iron core [2,4]. CTEM investigations enable the imaging and measurement of the rim and core size. Fig 1 (a). HRTEM confirms the crystalline structure of the particles. This investigation uses the ELNES of the O K-edge shown in Fig. 1 (b), to differentiate between the possible iron-oxide phases of the rim (FeO, α-Fe_2O_3 (hematite), γ-Fe_2O_3 (maghematite), Fe_3O_4 (magnetite), α-FeO(OH) (goethite)). Quantitative analysis of the EELS spectrum yields a chemical composition consistent with maghematite.
The second batch of analysis examined diminute iron oxide minerals in alkali feldspars of granitic rocks (Fig2 (a)). It has long been supposed, that the clouded pink-red feldspars in granites owe their colour to hematite inclusions [3], but optical petrography and microprobe analysis can't resolve the diminute structure in many cases. Using TEM and EELS these precipitates can be analysed. The ELNES investigation of the O K-edge confirms that the iron oxide phase of the small precipitates consists of hematite, as can be seen in Fig2 (b).
The investigations were performed using two different TEM's. A conventional JEOL 3010 operating at 297 kV equipped with a LaB_6 cathode, postcolumn Gatan Imaging Filter and a 1 K slow-scan CCD camera and a LIBRA 200FE operating at 200 kV, equipped with a field emission gun, a 4 K slow-scan CCD Camera and a 90° in-column Omega energy filter.

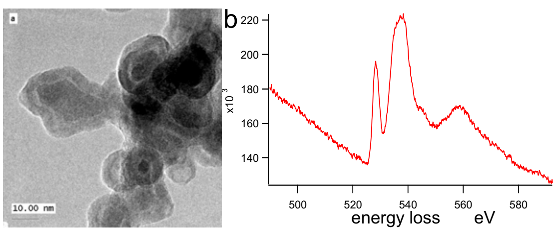

Fig.1.(a) Image of the iron oxide core shell Nanoparticles (b)ELNES of the O K-edge

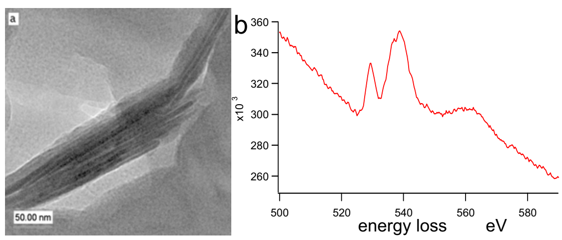

Fig.2. (a) hematite particle in feldspar and (b) ELNES of the O K-edge

References
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