NANOSTRUCTURED HIGHLY CRYSTALLINE, POROUS RHOMBOHEDRAL IRON OXIDES OF SUBMILLIMETER SIZE

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Abstract

We present here a case of obtaining nanostructured iron oxides by thermal treatment of submillimeter rhombohedral $FeCO_3$ single crystals. Conversion of $FeCO_3$ to Fe_3O_4 was realized inside a quartz tube by heated to 450 C, 2 h under Ar flow (1 ml/s). Conversion of Fe_3O_4 to -Fe_2O_3 was realized by mantaining the as-resulting magnetite for 7 h at 270 C in air. The increase in density (from 3.9 g/cm3 for siderite to 5.24 g/cm3 for magnetite and 4.9 g/cm3 for maghemite) caused quasi-ordered internal pores-grains pattern, with mesocrystalline appearance. The X-ray Line-Profile Fitting-based microstructure analysis gave 64 ± 6 nm and 84 ± 8 nm for the average inner grains size in magnetite and maghemite, in good agreement with the SEM observations. While the crystals exhibit smooth facets, internal porosity leading to remarkably large specific surface area (88.55 m2/g for magnetite, and as 40.14 m2/g for maghemite as evaluated by means of the BET method) was observed; the grain arrangement suggests a mesocrystalline structure. The sharp peaks in the XRD spectrum indicate highly crystallinity. Both Fe oxyde crystals exhibit magnetic hysteresis with saturation magnetization (92.1 emu/g for magnetite and 85.5 emu/g for maghemite). These crystals and their characteristics could use in applications where the highly porosity and highly crystallinity are necessary.

Keywords: Siderite, iron oxide, single crystalline, mesocrystalline, topotactic

ACKNOWLEDGEMENTS

This work was supported by the projects POSDRU/159/1.5/S/137070.

Author did not supply full text of the paper/poster.