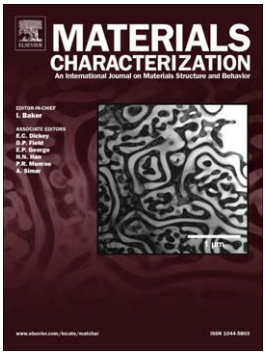

Quantitative phase analysis and microstructure characterization of magnetite nanocrystals obtained by microwave assisted non-hydrolytic sol–gel synthesis

Corrado Sciancalepore, Federica Bondioli, Tiziano Manfredini, Alessandro Gualtieri

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Quantitative phase analysis and microstructure characterization of magnetite nanocrystals obtained by microwave assisted non-hydrolytic sol-gel synthesis

Corrado Sciancalepore[a], Federica Bondioli[b,c], Tiziano Manfredini[a,c], Alessandro Gualtieri[d]

[a] Department of Engineering "Enzo Ferrari", University of Modena and Reggio Emilia, Via Vignolese 905, 41100, Modena, Italy

corrado.sciancalepore@unimore.it, tel: +00390592056224, fax: 00390592056243

[b] Department of Industrial Engineering, University of Parma, Parco Area delle Scienze, 181/A, 43124 Parma, Italy

[c] INSTM Consortium, Via G. Giusti 9, 51121 Firenze, Italy

[d] Department of Chemical and Geological Science, University of Modena and Reggio Emilia, Via S. Eufemia 19, 41121, Modena Italy

Abstract

An innovative preparation procedure, based on microwave assisted non-hydrolytic sol-gel synthesis, to obtain spherical magnetite nanoparticles was reported together with a detailed quantitative phase analysis and microstructure characterization of the synthetic products. The nanoparticles growth was analyzed as a function of the synthesis time and was described in terms of crystallization degree employing the Rietveld method on the magnetic nanostructured system for the determination of the amorphous content using hematite as internal standard. Product crystallinity increases as the microwave thermal treatment is increased and reaches very high percentages for synthesis times longer than 1 h. Microstructural evolution of nanocrystals was followed by the integral breadths methods to obtain information on the crystallite size-strain distribution. The results of diffraction line profile analysis were compared with nanoparticles grain distribution estimated by dimensional analysis of the transmission electron microscopy (TEM) images. A variation both in the average grain size and in the distribution of the coherently diffraction domains is evidenced, allowing to suppose a relationship between the two quantities. The traditional integral breadths methods have proven to be valid for a rapid assessment of the diffraction line broadening effects in the above-mentioned nanostructured systems and the basic assumption for the correct use of these methods are discussed as well.