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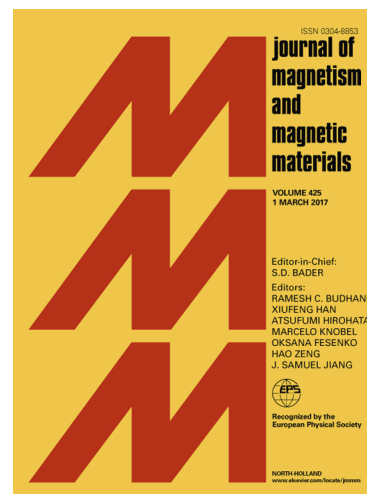
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# Identification of ferric oxide polymorphs in nanoparticles prepared by sol-gel method and maximization of $\epsilon$ -Fe<sub>2</sub>O<sub>3</sub> content

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**Keywords:** magnetically ordered nanoparticles;  $\epsilon$ -Fe<sub>2</sub>O<sub>3</sub>; hyperfine interactions; Mössbauer spectroscopy; sol-gel synthesis; iron oxide nanocomposites

## Abstract

The effects of annealing temperature (1000-1125 °C) and Fe<sub>2</sub>O<sub>3</sub> mass fraction (0.3-0.5) in the initial mixture on the composition of ferric oxide polymorphs within the Fe<sub>2</sub>O<sub>3</sub>/SiO<sub>2</sub> nanocomposite prepared by sol-gel method were investigated. The structural and magnetic properties of prepared samples were characterized in detail by combination of X-ray powder diffraction (XRPD), high-resolution transmission electron microscopy, magnetic measurements and a local probe technique - transmission Mössbauer spectroscopy (TMS). The optimal conditions for preparation of  $\epsilon$ -Fe<sub>2</sub>O<sub>3</sub> rich sample were established and ~91 % of the  $\epsilon$ -phase was reached with the characteristic particle diameter of 25 nm. It was observed that with increasing mass fraction of Fe<sub>2</sub>O<sub>3</sub> in the starting material, the optimal annealing temperature decreases, while the width of the particle size distribution tends to increase. The relative concentrations of the four present polymorphs derived from XRD and TMS are in very good mutual agreement.

## 1 Introduction

Iron oxides in all their various forms are one of the most used metal oxides with various applications both in scientific and industrial fields [1]. The four well-known crystalline polymorphs of ferric oxide Fe<sub>2</sub>O<sub>3</sub> have significantly different structural and magnetic properties. While the crystalline  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> and  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub> occur in nature,  $\beta$ -Fe<sub>2</sub>O<sub>3</sub> and  $\epsilon$ -Fe<sub>2</sub>O<sub>3</sub> are almost exclusively synthesized in the laboratory. Recently, the formation of a new Fe<sup>3+</sup> oxide polymorph, named  $\zeta$ -Fe<sub>2</sub>O<sub>3</sub>, was reported [2]. Its monoclinic structure with *I2/a* symmetry is prepared under high pressure treatment, however, is stable at ambient conditions. Nanoparticles of ferric oxides have excellent magnetic, catalytic, biochemical and other properties that make them suitable for specific technical, environmental and biomedical

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