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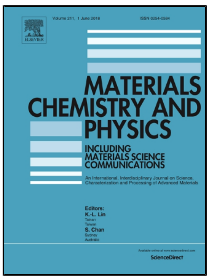

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Washing effect on the structural and magnetic properties of $NiFe_2O_4$ nanoparticles synthesized by chemical sol-gel method.

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Abstract. $NiFe_2O_4$ nanocrystalline samples were synthesized by the sol-gel method. The X-ray diffraction patterns of the as- synthesized samples (S- $NiFe_2O_4$) showed the formation of main spinel ferrite structure, with an average crystalline size of ~50 nm. After a washing process with magnetic separation two sets of $NiFe_2O_4$ particles was observed, one with ~36 nm and other with very small size (~2-3 nm). X-ray photoelectron spectroscopy (XPS) indicated the presence of Fe^{3+} and Ni^{2+} ions on the sample surface. Mössbauer spectra were recorded at 77 K and room temperature and were least square fitted for two sextets which have been assigned to the iron ions occupying the two symmetry sites of the ferrite structure. Magnetic measurements are consistent with the magnetic moment of Ni^{2+} ions suggesting the formation of an ideal inverse spinel structure in the sample S-$NiFe_2O_4$. Moreover, after the washing process with magnetic separation (sample P- $NiFe_2O_4$), when the formation of extremely small particles was determined, the Mössbauer and magnetic measurements indicated the reorganization of Ni^{2+}/Fe^{3+} ions in the tetragonal and octahedral sites of the spinel structure and some features related to thermal relaxations of the small particles are determined.

Keywords Sol-gel polymerization method; nickel ferrite nanoparticle; Mössbauer spectroscopy, magnetization, XPS-spectroscopy.

1 Introduction

In the last years, magnetic semiconductor nanostructured materials based on ternary transition-metal oxides have been extensively studied, due to the change in physical and chemical properties compared to their bulk samples, thus opening up opportunities for industrial and biomedical applications. Nickel ferrite ($NiFe_2O_4$) nanoparticles belong to this class of materials, presenting superior magnetic, thermal and transport properties [1-4], and has been already used in a many of applications, including gas sensing, catalysis, environmental cleanup, magnetic cell separator, magnetic drug carrier, tumor treatment, among others [5-9]. The magnetic properties are strongly affected at the nanoscale dimensions, because when the particle size decreases down to the nano-size, each particle can behave as a single magnetic domain. Besides, the properties which depends on the surface area are also modified because the surface becomes very large in comparison to the bulk material [10]. The electron spins on the surface of a magnetic material are canted (or disordered) because of reduced spin–spin exchange near the surface, reduced coordination number and broken bonds [11]. An extensive amount of research has focused on learning how changes in grain size, cation distribution, and surface properties affect the magnetic properties of ferrite magnetic nanoparticles. Ferrites crystallize in a spinel-type cubic structure, described by the formula AB_2O_4, where (A) and (B) represent the tetrahedral and octahedral sites, respectively, divided into two different ideal structure types, namely, normal and inverse spinels. In normal spinels, A ions of ferrites AB_2O_4 are solely located on