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Evidence for microwave-induced recrystallization in NiZn ferrites

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Abstract

A series of ferrites was prepared using the microwave method, starting with hematite as precursor in the $Ni_{1-x}Zn_xFe_2O_4$ ($x = 0$–1) system. After synthesis, the NiZn ferrite samples were irradiated with a microwave field for 90 s. X-ray diffraction (XRD) measurements were performed to yield the lattice constant as function of the amount x of Zn substitution, before and after irradiation. The lattice parameter was found to increase much slower with increasing x for the microwave irradiated specimens. Mössbauer spectroscopy was performed at room temperature and spectra were analyzed using the binomial distribution. For $x = 0.4$, 0.5 and 0.6 the resonance lines are much sharper for the irradiated samples and indicate that a microwave-induced recrystallization of the NiZn ferrites occurs in the composition range specified.

Keywords: Magnetic materials; Sintering; Mössbauer spectroscopy; Microstructure

1. Introduction

Ferrites are ferrimagnetic materials known to crystallize in a spinel structure, in which both divalent and trivalent cations are distributed among tetrahedral (A) and octahedral (B) sites. Magnetic divalent cations (such as Ni^{2+}) often prefer the octahedral sites and produce an inverse spinel structure. Diamagnetic divalent cations (such as Zn^{2+}) have preference for the tetrahedral positions and the resulting structure is a normal spinel.

Owing to their various technological applications, NiZn ferrites have attracted recently considerable scientific interest. The ferrites, in powder or thin film forms, were prepared by the high temperature solid state reaction method [1], coprecipitation [2], sol–gel method [3], pulsed laser deposition [4], high energy ball milling [5] and hydrothermal technique [6]. Recently, we proposed the microwave sintering procedure for the preparation of the NiZn ferrites [7]. However, the postproduction irradiation effects induced by a microwave field on these structures have not been examined.

In the process of microwave heating, the materials absorb microwave energy themselves and then transform it into heat within the sample volume. Thus, sintering can be completed in shorter times. Other advantages of microwave processing are higher efficiency, enhanced reaction and sintering rate as well as shorter cycle times and cost savings [8].

In this paper we propose to compare the structural and magnetic properties of NiZn ferrites prepared by microwave sintering with those corresponding to the samples subjected to postproduction irradiation with a microwave H-field. We characterize the structural and magnetic properties of the ferrite specimens using XRD and Mössbauer spectroscopy.

2. Experimental

$Ni_{1-x}Zn_xFe_2O_4$ ($x = 0$–1) were prepared using high purity NiO, ZnO, α-Fe_2O_3 in stoichiometric proportions. After mixing and grinding, the powders were calcined at 1100 °C for 4 h. All compositions were sintered at 1275 °C for 30 min using a multimode microwave furnace (2.45 GHz, Amana Radarange, Model RC/20 SE) operating at maximum input power of 2.0 kW (the output power of magnetron is roughly 50–60% of the input power). The samples were placed on a thin SiC plate that also acted as a pre-heater. In our experiments, the emissivity of the single wavelength IR pyrometer was set at $e = 0.75$, and this value was chosen by calibrating against the optical pyrometer set at the other side of the tube [8]. After obtaining the NiZn ferrite samples, they were subjected to a postproduction H-field treatment using a single mode TE_{103} cavity for 90 s [9]. The X-ray diffraction spectra were recorded with a Rigaku D-2013 diffractometer with Cu Kα radiation ($\lambda = 1.540598$ Å). Lattice constants were determined by Rietveld refinement. The Mössbauer spectra were recorded at room temperature using a source of ^{57}Co in Rh matrix and an MS-1200 constant

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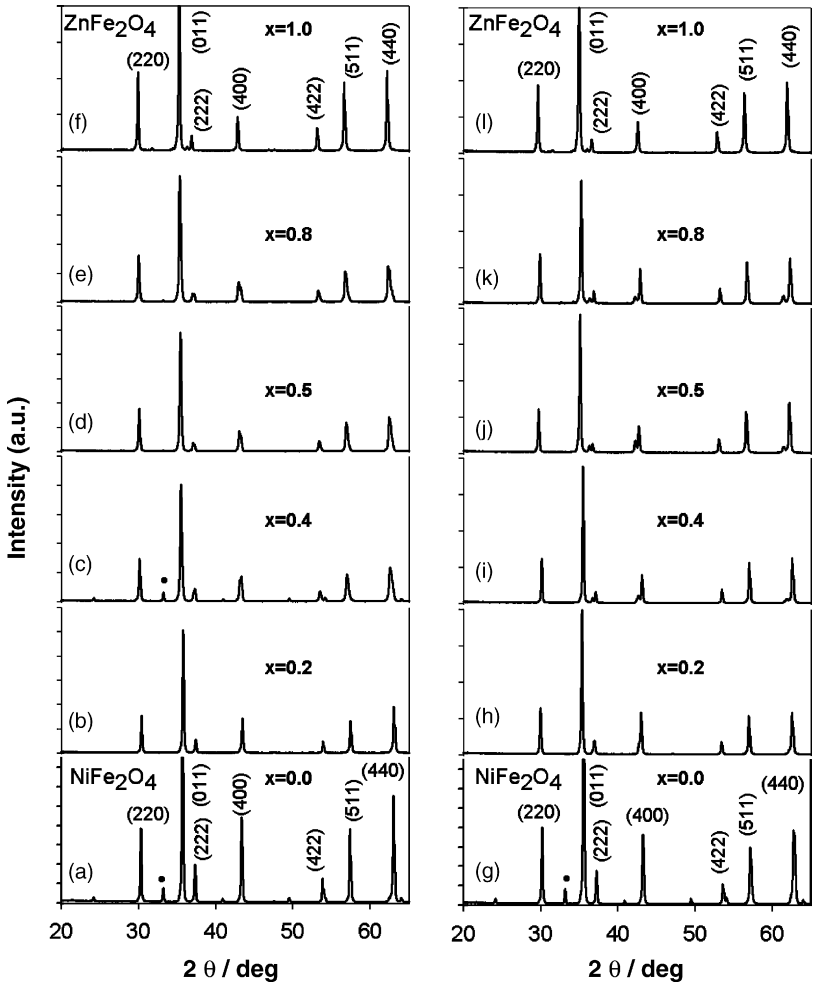

Fig. 1. XRD patterns of the nickel–zinc ferrites sample before (a–f) and after microwave irradiation (g–l).

acceleration spectrometer. All spectra were analyzed by nonlinear least-squares fitting using the NORMOS program within the frame of the binomial distribution model.

3. Results and discussion

Fig. 1(a–l) displays the XRD patterns of the $Ni_{1-x}Zn_xFe_2O_4$ ($x = 0$–1) ferrites, before and after irradiation with the additional microwave field, respectively. Fig. 2 plots the lattice constant as function of the amount x of Zn substitution, for both as-sintered and irradiated samples. It can be observed that in both cases, the lattice parameter increases linearly with increasing x, due to the greater ionic radius of zinc ions with respect to the ionic radius of iron ions. Moreover, it can be observed that the slope of the linear increase is smaller for the case of the samples irradiated with the microwave field. These results can be correlated with the onset of recrystallization in the irradiated specimens.

Fig. 3(a–l) shows the room temperature transmission Mössbauer spectra of the $Ni_{1-x}Zn_xFe_2O_4$ ($x = 0$–1) system before and after microwave field irradiation, respectively. The spectra were analyzed by least-squares fitting using the binomial distribution model [10]. The refined values of the hyperfine parameters are given in Tables 1 and 2 for specimens before and

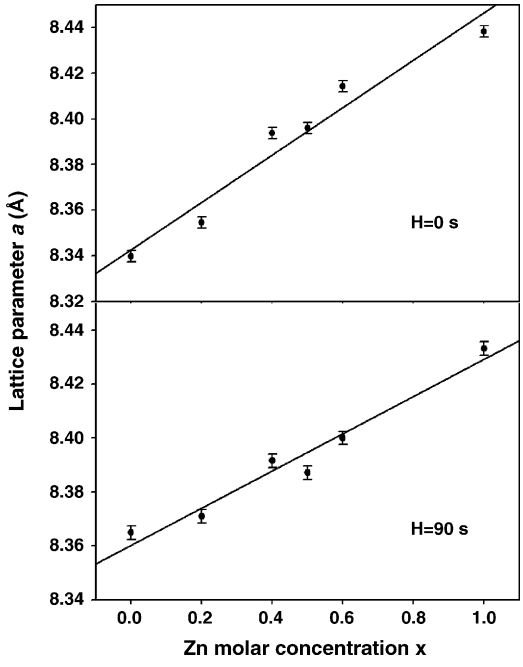

Fig. 2. Lattice parameter a as a function of Zn molar concentration x for ferrite samples before and after microwave irradiation.