Synthesis of Ni ferrite powders by coprecipitation and hydrothermal methods

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The coprecipitation and hydrothermal methods were used to obtain Ni ferrite nanopowders with $Ni_{0.8}Fe_{2.2}O_4$ stoichiometry. Morphological and magnetic properties of the synthesized nickel ferrites nanopowders have been studied. The structural properties of the $Ni_{0.8}Fe_{2.2}O_4$ nanopowders were studied by X-ray diffraction (XRD). Surface morphology was examined by scanning electron microscopy (SEM). The results show that both the synthesis method and conditions used to obtain the Ni ferrites have significant influence on their structure and properties. The low frequency (50 Hz) magnetic behavior of the Ni ferrites also exhibits important differences evidenced by magnetic hysteresis curves.

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1. Introduction

Nickel ferrites are important materials due to their magnetic properties, weak electric conductivity, leading to lower eddy current losses and having high electrochemical stability. They crystallize in a spinel structure and exhibit tunable magnetic behavior, that makes them usable in transformer core, antenna rod, recording head and loading coil applications [1,2,3].

Ferrites are also used currently in high-frequency devices, because of their high permeability in the radio frequency region, having also high electrical resistance, mechanical hardness and chemical stability [4]. Ferrite powders are used also in microwave devices, insulators and phase shifters [5,6]. Microwave technology is moving constantly to higher frequencies and higher bandwidths, into the mm wave range [7]. The increased frequency range used in communication requires special ferrites [8,9,10] in order to make them applicabile for high frequency microwave devices.

The magnetic behavior of ferrites has recently become of increasing interest, as strong electromagnetic absorption led to shielding applications in microwave devices [11,12,13].

In the present work we prepared nickel ferrite $Ni_{0.8}Fe_{2.2}O_4$ nanopowders by the coprecipitation and hydrothermal methods and analysed their structural and magnetic properties.

2. Experimental

Nickel ferrites powders with the stoichiometry of $Ni_{0.8}Fe_{2.2}O_4$ were prepared by coprecipitation (sample A in Fig.1a)) and hydrothermal (sample B in Fig.1b)) using iron nitrate and nickel sulfate.

The coprecipitation technique was performed by mixing together iron nitrate and nickel sulfate with bidistilled water, after adding sodium hydroxide and heating the solution to 90°C. The solution was heated for 5 hours using magnetic agitation at 400 rpm, then cleaned with bidistilled water and alchool and filtered for 5 hours to obtain neutral pH. After drying, the powder was calcined at 200°C for 2 hours in a Nabertherm Oven.

In the hydrothermal method, we used iron nitrate and nickel sulfate with bidistilled water and sodium hydroxide and heated the solution in a autoclave at 200°C for 5 hours in an Heraeus 6060 UT Stove. The final solution was cleaned and filtered with double distilled water for 5 hours to obtain neutral pH.

3. Morphology characterization

3.1 SEM investigation

Structural micrographs of nickel ferrites powders were obtained by using Scanning Electron Microscopy (SEM) with an FEI Company microscope, type Inspect S.

Typical SEM micrographs of $Ni_{0.8}Fe_{2.2}O_4$ powder obtained by the two synthesis methods are shown in Fig. 1a), b). The micrographs show the structure grown on the substrate with smallest particles sizes bellow 100 nm.

For the sample A, shown in Fig.1a), the surface exhibits plate shapes as compared with the sample B, shown in Fig.1b), were particles have many circular rock type shapes. Thus we observed differences in the aggregation of the material using the two techniques.

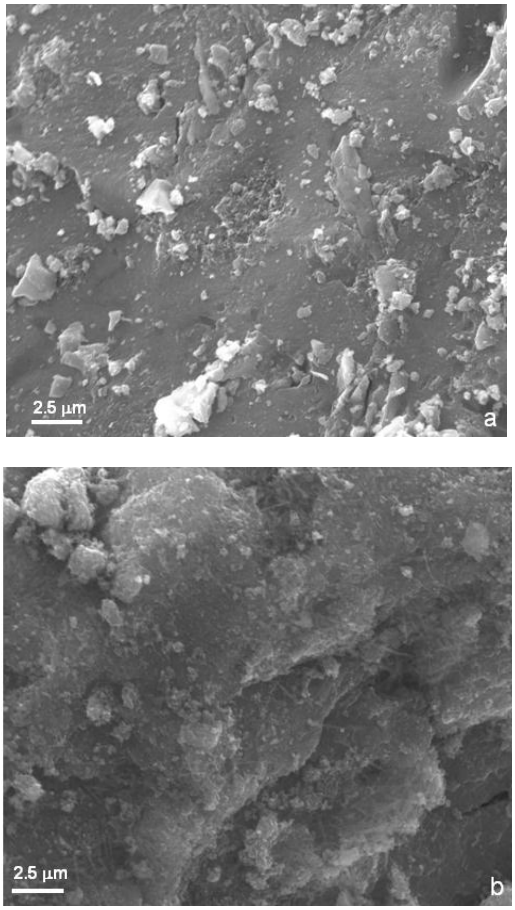

Fig.1 SEM micrographs of the $Ni_{0.8}Fe_{2.2}O_4$ powder for sample A - oprecipitation (a) and sample B- hydrothermal (b).

3.2 XRD investigation

The nickel ferrite powders structures were characterized using X-ray diffraction (XRD) with a Philips difractometer, type X'Pert PRO MPD in the 2θ range of 20–90°.

Fig.2 shows typical X-ray diffraction patterns of $Ni_{0.8}Fe_{2.2}O_4$ powder obtained by the two methods showing two predominant diffraction peaks at 35° and 64° of the planes (311) and (440) for each sample.

The crystal structure of $Ni_{0.8}Fe_{2.2}O_4$ is cubic spinel with unit cell size a = 3A˚. The $Ni_{0.8}Fe_{2.2}O_4$ nanopowder obtained by the hydrothermal method is oriented along the (311), (440) and (511) planes, see Fig.2b), while for the coprecipitation method the nanopowder is oriented along the (311) and (440) planes, see Fig.1a). Other orientations corresponding to (220), (400) and (731) planes are also present with relative low intensities. The calculated avarage size of particles using Scherrer formula [14] gave approximately 30 nm for sample B, see Fig.2b), and about 15 nm for sample A, see Fig.2a). There are differences of the morphology according to the synthesizing methods.

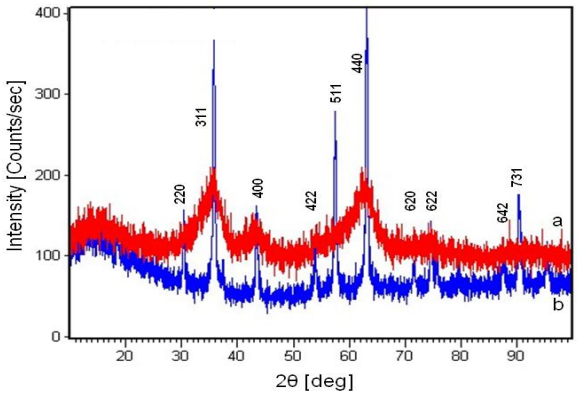

Fig.2 DRX graphs of $Ni_{0.8}Fe_{2.2}O_4$ powders for sample A - coprecipitation (a) and sample B - hydrothermal (b)

4. Magnetic hysteresis measurements

The magnetic investigations on the powders in the as-prepared state were carried out at room temperature under AC (50 Hz) applied magnetic fields of amplitudes up to 160 kA/m, thus integrated fluxmeter-based hysteresisgraphs [15] were obtained.

Typical M-H graphs of the $Ni_{0.8}Fe_{2.2}O_4$ powders obtained by the two synthesis methods are given in Fig.3a) and b). While the sample A, see Fig.3a) exhibits weak magnetism the sample B, see Fig.3b) is a typical soft ferrite with 4kA/m coercivity, 240 A/m remanence and 900 A/m saturation magnetization.

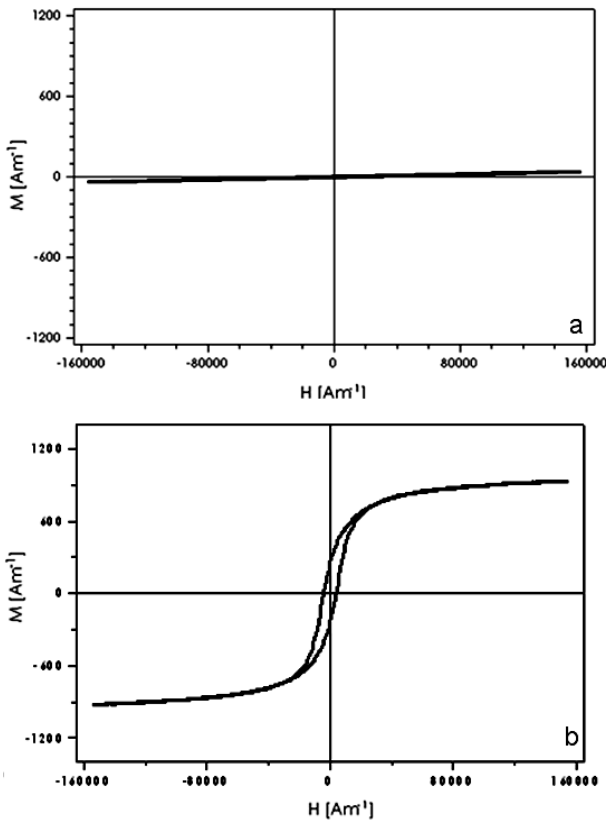

Fig.3 Hysteresis graphs of $Ni_{0.8}Fe_{2.2}O_4$ powders for sample A - coprecipitation (a) and B - hydrothermal (b)

5. Discussion

$Ni_{0.8}Fe_{2.2}O_4$ powders synthesized by the coprecipitation and hydrothermal techniques show ferrimagnetic and ferromagnetic behaviors respectively [16]. Nickel ferrite obtained by coprecipitation had almost zero magnetization as compared with the samples obtained by the hydrothermal technique seen from the clear hysteresis curve and higher magnetization. Thus, we conclude that the synthesis method has important influence on the magnetic behavior of the nickel ferrite nanopowders.

The investigation of the morphology and structure of $Ni_{0.8}Fe_{2.2}O_4$ nanopowders shows also structural differences for the two synthesis techniques. The samples obtained by the hydrothermal technique has a structure composed of coarse grains. As compared, the coprecipitation method gave compact plate like forms of various sizes. However, the smallest particles sizes are similar for both methods giving an aproximatively value of 100 nm.

We believe thus that the differences in the structural properties of the materials obtained by the two methods are reflected also in their different magnetic properties.

6. Conclusions

The nickel ferrite nanopowders obtained by the hydrothermal technique shows clear magnetic hysteresis, while those synthesized by the coprecipitation technique exhibit only weak magnetic properties.

The ferrites investigated by electronic microscopy showed a different aggregation structure for the two synthesis techniques although the x-ray diffraction curves for both methods show main cristalinity peaks wich are typical for nickel ferrites.

Thus we conclude that the hydrothermal method was a better synthesis technique then coprecipitation for obtaining ferrites with ferromagnetic behavior.

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