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Synthesis of Fe–Ni nano-particles by low-temperature hydrogen reduction of mechanically alloyed Ni-ferrite

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ABSTRACT

Fe–Ni nano-particles were synthesized by mechanical alloying and subsequent low-temperature hydrogen reduction of oxide mixtures. First, single-phase nickel ferrite was mechanically synthesized for 30 h with steel balls to powder weight ratio of 20 while the milling was operated at 300 rpm. The as-milled powder was then placed in contact with hydrogen at 600 °C for 1 h. Reduction reactions resulted in production of Fe–Ni nano-particles. Phase identification, morphological and microstructural studies, chemical analysis and magnetic property determination were carried out by X-ray diffraction (XRD), scanning electron microscopy (SEM), transition electron microscopy (TEM), energy dispersive spectrometry (EDS) and vibration sample magnetometry (VSM) on the samples. Results are described in the paper.

1. Introduction

Fe–Ni magnetic nano-particles have important applications in catalysts, magnetic fluids and high-density recording media due to their low thermal expansion and their remarkable magnetic properties [1]. In such applications, composition of the particles is considered the key characteristic affecting their magnetic properties. Many compositions of the Fe–Ni alloy have previously been investigated [2–4].

Mechanically activated processes have received considerable attention in the fields of solid-state chemistry, physics and materials science in recent years [5–7]. Mechanical activation like high-energy ball milling of crystalline solids provides a route to solids far from equilibrium such as nano-structured materials, amorphous alloys and quasi-crystalline phases [6,7]. The technological advantage of this process is that the powder can be produced in large quantities and the processing parameters can easily be controlled at an industrial level [7].

Reduction of nickel ferrite via conventional synthesis has been reported by Bahgat et al. [8]. Reduction of different ferrites such as ferrites of cobalt, zinc, strontium, barium and magnesium has also previously been investigated [8–12]. Results of most recent studies on hydrogen reduction of nickel ferrite synthesized by mechanical alloying are illustrated as a novel method for production of magnetically soft, Fe–Ni nano-particles in this paper. Both as-milled and hydrogen reduced powders were magnetically soft. Magnetization saturation highly increased due to the strain relaxation and phase transformation of ferrite to iron based alloy.

2. Experimental procedure

A stoichiometric mixture of analytical grade hematite (Fe_2O_3) (MERCK, Gmbh) and black nickel oxide (NiO) (Aldrich, USA) was mechanically milled in a planetary ball-mill. The milling was carried out in a hardened steel vial containing steel balls having 10 and 15 mm diameters under atmospheric air. The ball-to-powder mass-ratio was 20:1 and the milling speed was 300 rpm. After 30 h of milling, the powder produced was reduced with hydrogen for 60 min at different temperatures. The phases formed in the samples were identified by X-ray diffraction (XRD) (Philips PW 3710, Netherlands) having Cu Kα radiation. Microstructure and particle size of the reduced powders were determined by scanning electron microscopy (SEM) (VEGA II XMU, Tescan, Czech) and transmission electron microscopy (TEM) (CM200, Philips). Phase composition of the reduced powder was determined by electron dispersive spectrometry (EDS) equipment attached to the SEM. Magnetic properties of the reduced nano-particles were evaluated by vibration sample magnetometer (VSM) (assembled in Iran) run at the room temperature.

3. Results and discussion

After 30 h of milling, a single-phase nickel ferrite spinel having the X-ray diffraction pattern indicated in Fig. 1 was obtained.

From the data available in the literature [13], the standard Gibbs free energy change of Reaction (1) was evaluated to be −18.3 kJ/mol. This quantity indicated that the milling kinetically assisted the thermodynamically spontaneous reactions to form $NiFe_2O_4$ from the starting mixture, while the surfaces of the solid particles of the mix-

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