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Effect of hydrogen reduction on microstructure and magnetic properties of mechanochemically synthesized Fe–16.5Ni–16.5Co nano-powder

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ABSTRACT

Most recent findings on structural and magnetic properties of Fe–Ni–Co nano-powders produced by mechanical alloying and subsequent low-temperature hydrogen reduction are presented in this paper. At 300 rpm, with ball to powder weight ratio of 20, single phase nickel–cobalt ferrite is mechanically synthesized for 50 h. The as-milled powder is then subjected to 1 h hydrogen reduction at 700 °C. Hydrogen reduction results in the formation of Fe–16.5%Ni–16.5%Co nano-powders. The phases of the powders are identified by X-ray diffraction (XRD) utilizing Cu Kα radiation. Scanning electron microscopy (SEM) and transmission electron microscopy (TEM) are used to study the morphology and the average size of the nano-powder particles. Chemical analysis of the phases present in the reduced sample is determined by electron dispersive spectrometry (EDS). The magnetic properties of the powders are measured by a vibration sample magnetometer (VSM). Results indicate a noticeable change in the magnetic properties of the samples due to $Ni_{0.5}Co_{0.5}Fe_2O_4$ compositional change into Fe–16.5Ni–16.5Co nano-powder.

1. Introduction

Fe–Ni–Co alloys demonstrate good soft magnetic properties. Magnetostrictive properties foster their use in manufacturing of magneto-resistance sensors and a magneto-resistance heads [1]. Fe rich Ni–Co–Fe ternary alloys have demonstrated low thermal expansion. Their commercial applications include microwave guides, spacecraft optics, laser housings and printed wired boards [2,3].

Mechanochemical processing is a novel method for production of nano-sized particles. Mechanochemical synthesis is particularly suitable for large-scale economic production of these particles. Its processing parameters can easily be controlled even at industrial scale [4,5].

Reduction of conventionally synthesized nickel–cobalt ferrite has been reported by Bahgat et al. [6]. Reduction of other ferrites such as zinc, strontium, barium and magnesium ferrites has been investigated by other investigators [7–9]. Recent studies on mechanical alloying/hydrogen reduction for production of nickel–cobalt ferrite nano-powders are reported here.

2. Experimental procedure

Starting materials were analytical grade hematite (Fe_2O_3) (MERCK, Gmbh), nickel oxide (NiO) (Aldrich, USA) and cobalt oxide (Co_3O_4) (MERCK, Gmbh). A mixture of these ingredients resulting in Fe:Ni:Co molar ratio of 4:1:1 was mechanically milled at 300 rpm by a planetary ball mill having hardened steel vials containing 15 and 20 mm diameter balls of 20:1 ball to powder weight ratio for 50 h. The milled sample was placed into a tube furnace being purged with argon while gradually heated up to 700 °C at a rate of 10 °C/min. A hydrogen generator linked to the furnace supplied high-purity hydrogen (99.9999%) required for reduction of the milled sample. The reaction time was arbitrarily fixed at 1 h after reaching 700 °C; while the tests were performed at different temperatures to find the temperature for completion of the reduction. Hydrogen flow was adjusted at 50 cm^3/min at this stage.

X-ray diffraction (XRD) (Philips PW 3710, Netherlands) was used to identify the phases present in the samples. Microstructural characteristics such as morphology, particle size and crystal structure of the samples were analyzed by scanning electron microscope (SEM) (VEGA II XMU, Tescan, Czech) and transmission electron microscope (TEM) (CM200, Philips). Electron dispersive spectrometry (EDS) equipment attached to the SEM was used to determine the composition of the reduced phases. The magnetic properties of both as-milled and reduced samples were measured by vibrating sample magnetometer (VSM) (I.R. Iran) at room temperature.

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