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Synthesis and Characterization of FeCo Nanoparticles by Hydrothermal Method

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Introduction

Magnetic nanomaterials are of major interest due to the wide range of potential technological applications in which these materials can be used¹, such as high-density data storage, catalysis and medical diagnosis^{2,3}. Transition metallic materials, especially Co, Fe and Ni, and their alloys, exhibit particular properties that are rather different from those of their corresponding bulk counterparts^{4,5}. FeCo is considered as a very attractive system due to its magnetic, catalytic, and mechanical properties compared to the elemental Fe and Co metals⁶. Previously, many methods have been used to elaborate FeCo NPs, such as mechanical alloying, electrodeposition and thermal decomposition. More recently, chemical routes, hydrothermal method notably, became a common way for the synthesis of bimetallic alloys, because they offer the ability for a controllable composition and morphology during the synthesis process. Furthermore, the hydrothermal route, i.e., the co-reduction of metal salts by an appropriate reducing agent, is distinguished by its simplicity, low cost, and also flexibility in the choice of synthesis parameters⁷. However, due to the highly broad difference in the reduction potentials of metals, the synthesis of binary alloys by co-reduction of metal salts faces major difficulties. Thus, the convenient choice of synthesis parameters⁸ is still a challenging purpose and requires further research.

Experimental Study

1.1 Synthesis of FeCo NPs

In a typical synthesis process of FeCo NPs, a solution is first prepared by dissolving predetermined quantities of precursors: (FeSO₄·7H₂O and chloride CoCl₂·6H₂O) in ethanol/water mixing solution. Then, sodium hydroxide is added drop wise to the metal salts solution under vigorous stirring. Afterwards, N₂H₄·H₂O (hydrate hydrazine 85%) is added to the above solution. The mixed solution was heated at 100 °C for 30 min and then cooled down to room temperature. The black fluffy product at bottom of the solution was collected using a magnetic bar, and cleaned with deionized water and absolute ethanol. The process was repeated several times. The final product was dried in air at 50 °C for 4 h. A series of five powder samples was prepared with different predetermined masses of precursors, and will be named as S-1, S-2, S-3, S-4 and S-5, respectively.

1.2 Sample Characterization

The room temperature x-ray diffraction (XRD) patterns of the FeCo NPs powders were recorded in a Philips X-PertPro diffractometer equipped with a Cu K α radiation ($\lambda=1.54060$ Å). The scanning range was from 30 to 100° with a step of 0.02° in 2 θ . The morphology of the FeCo powders was studied through several images obtained with a PHILIPS ESEM XL 30 FEG scanning electron microscope (SEM)



equipped with Energy Dispersive X-ray analyser (EDX).

Results and Discussion

The alloys with the compositions Fe₁₀Co₉₀, Fe₁₅Co₈₅, Fe Co , Fe Co and Fe Co were synthesized via hydrothermal route. The XRD patterns of the binary alloys show the presence of many peaks that can be indexed as the Bragg reflections belonging to two different phases: body faced cubic (BCC) and face-centered cubic (FCC) crystal structures. It can be observed that additional impurity phases such as iron oxide and cobalt oxide are not present, indicating the purity of the synthesized powders. The phase composition, lattice parameter, a(nm), and the mean crystallite size, <D(nm)>, were obtained using the Scherrer method by the X'Pert HighScore software. The SEM micrographs of the five FeCo NPs samples evidence that the morphology of the synthesized FeCo nanostructures changes with the change of the Fe/Co content.

Conclusion

We have successfully synthesized pure FeCo NPs by means of a low cost and surfactant-free hydrothermal method at low temperatures (105 °C) and short annealing periods (30 min) using sodium hydroxide (NaOH). The concentration of NaOH had a significant role on the co- reduction of metal salts and favors the formation of alloy phase and its morphology. Moreover, the line-broadening analysis of the XRD patterns tells us that the average values of NP size for all samples were found to be below 30 nm, which indicates the nanometric state of elaborated powders.

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