



Hydrothermal-induced oriented growth of Fe–Co alloy and Sm^{3+} -substituted magnetite nanowire composites

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ABSTRACT

Fe–Co alloy and Sm^{3+} -substituted magnetite nanowire composites ($\text{Co}_x\text{Fe}_{1-x}/\text{Co}_y\text{Fe}_{1-y}\text{Sm}_z\text{Fe}_{2-z}\text{O}_4$) have been synthesized via a hydrothermal method without using surfactants or templates. The effects of substitution on structure and morphology were investigated by powder X-ray diffraction, X-ray photoelectron spectroscopy, Mössbauer spectroscopy, and transmission electron microscopy (TEM). The TEM image shows that the average diameter of the magnetite nanowires is about 40 nm and the length is several micrometers. The $z = 0.1$ composite shows relatively high saturation magnetization (92.3 emu/g) detected by a vibrating sample magnetometer. The possible growth mechanism of the nanowires is discussed on the basis of the crystal structure of the materials. From the perspective of thermodynamics, we explain the postulated mechanism of the hydrothermal reaction.

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1. Introduction

In the past decade, considerable attention has been drawn to one-dimensional (1D) inorganic nanostructured composite materials, including nanotubes [1], nanorods [2], and nanowires [3], because of both their interesting physical properties and wide range of potential applications in nanodevices [4–15]. A variety of inorganic nanowires have been synthesized in the past 5–10 years [16]. Among these nanowires, magnetite nanowires are of great interest because of their potential applications in magnetic-recording media and other areas. Although 1D nanostructures of magnetite could be prepared via different approaches, the controllable growth of 1D morphology based on a surfactant-free synthetic route is still a challenge. It is well known that the hydrothermal route to synthesize magnetite nanowires has been studied extensively for its simple process and large-scale production [17]. The morphology of magnetite nanowires can be controlled during the hydrothermal process in the presence of surfactants or templates.

In addition, conventional Fe–Co alloy has been widely used in industry. Great efforts have been made to investigate the magnetic properties of the Fe–Co alloy because of their novel physical and chemical properties [18].

The composite Fe/Fe₃O₄ belongs to an important class of magnetic materials, which is very well known owing to its remarkable magnetic properties particularly favorable for its electromagnetic as well as conducting properties [19–22]. Considerable attention has been paid to the Fe/Fe₃O₄ composite, due to its vast applications that range from making electrical devices to performing catalysis. An important modification in the structure as well as magnetic properties can be achieved by introducing a relatively small amount of foreign ions into the Fe/Fe₃O₄ composite. Cobalt is another important element that has been found to improve the coercivity of the material and increase the chemical stability [23]. It is well known that rare-earth ions play an important role in deterring the magnetocrystalline anisotropy in 4f–3d intermetallic compounds [24,25]. Till to date, some researchers have studied their influence on ferromagnetic oxides [26–28]. From Rezlescu's studies, the rare-earth ions with relatively a large radius and a stable valence 3+ state were found to be the best substituents for improving the magnetic as well as electrical properties of the soft magnetic ferrite [29]. Keeping all this in mind we considered Co to be the most suitable candidate for substitution. Therefore, we synthesized the Fe–Co alloy and cobalt-substituted magnetite nanowire composites doped with Sm^{3+} and expected the composites have the desirable coercive field and saturation magnetization. Thus, the effect of rare-earth ions substitution seems extremely valuable in this aspect.

To our knowledge, there have been few reports about the synthesis of the Fe–Co alloy and cobalt-substituted magnetite nanowire composites. In this manuscript, we synthesized the

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composites under hydrothermal condition without using surfactants or templates.

2. Experimental

The chemical reagents used were ferrous chloride tetrahydrate ($\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$), cobalt chloride hexahydrate ($\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$), samarium chloride hexahydrate ($\text{SmCl}_3 \cdot 6\text{H}_2\text{O}$) and potassium hydroxide (KOH). All chemicals used in this experiment were of analytical grade and were used without further purification. Distilled water degassed with N_2 gas for half an hour was used for preparing aqueous solution of $\text{Fe}^{2+}:\text{Co}^{2+}:\text{Sm}^{3+}$ ions (2:1:z). z represents the molar concentration at which Sm^{3+} substituted Fe^{3+} in magnetite. The aqueous solution was transferred into a Teflon-lined stainless autoclave, and the potassium hydroxide was dissolved into distilled water and then added into the autoclave slowly. During the experiment, N_2 was continuously passed through the solution to prevent the oxidation of Fe^{2+} in the system. The autoclave was put into an oven, kept at 150°C for 3 h, and then cooled to room temperature naturally. After completion of the reaction, the black solid products were collected by magnetic separation and washed several times with water and ethanol. The final products were dried in a vacuum oven at 40°C for 10 h.

3. Characterization

X-ray diffraction (XRD) pattern was performed on a Tokyo X-ray diffractometer with $\text{Cu K}\alpha$ radiation ($\lambda = 0.15405\text{ nm}$). X-ray photoelectron spectroscopy (XPS) was collected on an ESCALAB M K_{11} X-ray photoelectron spectrometer with $\text{Mg K}\alpha$ X-ray as the excitation source. Mössbauer spectrum was recorded at 295 K by using a computerized Oxford MS-500 Mössbauer spectrometer of the electromechanical type in constant acceleration mode. The transmission electron microscope (TEM) image was taken with a Hitachi H-800 TEM, using an accelerating voltage of 200 kV. Magnetic measurements were carried out at room temperature using a vibrating sample magnetometer (Digital Measurement System JDM-13) with a maximum magnetic field of 10,000 Oe.

4. Results and discussion

4.1. Structure and morphology of Fe–Co alloy and cobalt-substituted magnetite nanowire composites doped with Sm^{3+}

As is shown in Fig. 1, the presented XRD pattern features seven Bragg diffraction peaks in the 2θ range of $25\text{--}65^\circ$, which can be easily indexed as (2 2 0), (3 1 1), (2 2 2), (4 0 0), (4 2 2), (5 1 1), and (4 4 0) planes. All of the peaks can be indexed to the face-centered cubic phase of Fe_3O_4 with a lattice constant of $a = 8.394\text{ \AA}$, which is very close to the reported data (JCPDS 85-1436, $a = 8.393\text{ \AA}$). In addition, it is observed from the XRD pattern that the (1 1 0) plane represents body-centered cubic (BCC) structure of the Fe–Co alloy. The SmFeO_3 phase appears when the substitution value (z) is 0.2. And the peak intensity of SmFeO_3 increases with the increasing concentration of the Sm^{3+} ions. This means that 0.1 is the optimal value for the substitution of Fe^{3+} ions by Sm^{3+} ions. When z is 0.3 or 0.4, the peaks of SmFeO_3 appear besides the peaks of spinel, BCC and FCC structures. No extra reflections are detected from the XRD pattern. To further prove the composition of the composite, the product was examined by XPS. The spectra are shown in Fig. 2a and b, corresponding to the binding energy of $\text{Fe}2\text{p}$ and O1s. The figures show that the peaks located at 710 and 724 eV correspond to $\text{Fe}2\text{p}_{3/2}$ and $\text{Fe}2\text{p}_{1/2}$, and the peaks of 530 eV could

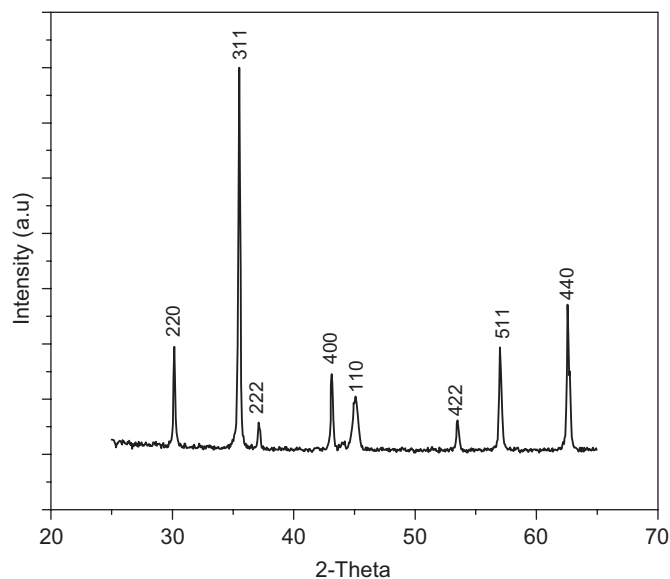


Fig. 1. XRD pattern of the Fe–Co alloy and cobalt-substituted magnetite nanowire composite doped with Sm^{3+} synthesized at 150°C for 3 h.

be attributed to O1s. The data are very close to the reported values of Fe_3O_4 in other literatures [30,31]. The peaks of the C1s, O1s, Co2p, Fe2p, and Sm3d are shown in Fig. 2(c).

As can be seen from Fig. 3a, the diameter of the nanowires is about 40 nm and the length is about $2\text{ }\mu\text{m}$. The size of Fe–Co alloy with spherical morphology in Fig. 3a is less than 100 nm, while the XRD pattern shows that these particles exhibit BCC structure. Selected area electron diffraction (SAED) pattern taken from the spherical particles areas in Fig. 3a turns out to be a series of diffraction rings. Every diffraction ring corresponds to a crystal plane, and it confirms that the obtained spherical particles are Fe–Co alloy. Fig. 3b shows that the Fe–Co alloy has a BCC structure because there exhibit the diffraction ring pattern of (1 0 0), (1 1 1), (1 1 0), (2 0 0) and (2 1 1) planes in the SAED pattern.

It is well known that magnetic materials possess easy magnetic axes. Magnetite, for example, has easy magnetic axes along (1 0 0) crystal direction. Therefore, external conditions such as magnetic field, surfactants, and hydrothermal process might induce oriented growth, for example, along the easy magnetic axis (1 0 0) crystal direction, which could induce the formation of 1D nanostructure of magnetite. Thus, the direction of growth was further determined to be the (1 1 0) crystal direction, it is just one of the easy magnetic axis of Fe_3O_4 . One-dimensional structure is indeed a product of oriented growth of materials, which is influenced by its growth character and complex experimental conditions. Therefore, it is of great importance to select the different experimental parameters carefully and these parameters should be well controlled. It is obvious that the hydrothermal conditions directly affect the growth process of magnetic nanowires. In addition, previous work have shown that magnetite exhibits preferential growth along the (1 1 0) crystal direction under certain conditions [32,33]. The influence of chemical potential on the shape evolution of nanocrystals has been elucidated by Peng et al. [34,35]. In the case of 1D nanostructure growth, it would be beneficial to have a higher chemical potential, which is mainly determined by the concentration of alkaline aqueous solution because of high concentration of OH^- ion and higher chemical potential in the solution favor growth of nanowires over other possible iron oxide crystal forms.

In conclusion, it is observed that hydrothermal process can significantly influence the growth behavior of magnetite

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