

Article

Simple Synthesis of NdFeO₃ Nanoparticles By the Co-Precipitation Method Based on a Study of Thermal Behaviors of Fe (III) and Nd (III) Hydroxides

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Abstract: In this study, a nanostructured NdFeO₃ material was synthesized via a simple process of the hydrolysis of Nd (III) and Fe (III) cations in hot water with 5% NaOH as a precipitating agent. According to the results of the thermal behaviors of each hydroxide, either containing Fe (III) or Nd (III), the perovskite type of neodymium orthoferrite NdFeO₃ was simply synthesized by annealing a mixture of Fe (III) and Nd (III) hydroxides at 750 °C. The nanostructured NdFeO₃ was obtained in spherical granules with diameters of around 30 nm. The magnetic properties of the material were a coercive force (H_c) of 136.76 Oe, a remanent magnetization (M_r) of 0.68 emu·g⁻¹, and a saturation magnetization (M_s) of 0.79 emu·g⁻¹.

Keywords: co-precipitation method; NdFeO₃; perovskite; crystal structure; magnetic property

1. Introduction

In recent decades, studies on the synthesis and characteristics of inorganic nanoparticles, and nano-structured materials—including chemical components, structures, and morphologies—have drawn much interest due to several advantages of nanoscale particles in comparison with micron-sized ones [1,2]. Specifically, as the particle size decreases from the microscale to the nanoscale, materials based on the orthoferrites of rare-earth elements (RFeO₃, where R is a rare-earth element) exhibit a clear perovskite structure and a high sensitivity in terms of electrical and magnetic properties [3–7]. Moreover, the nano-sized crystalline perovskite materials can be applied in several advanced technologies such as solid oxide fuel cells, catalysts, chemical sensors, and electrode materials. [8–11]. Among these applications, the NdFeO₃ nanoscale materials, known as orthorhombically distorted perovskites [12,13], have significant potential applications in As (V) adsorption [8] and the fabrication of electrical and magneto-optic devices [12–15].

Nanosized NdFeO₃ materials have been synthesized by various methods, including high-temperature ceramic fabrication [12,13,16,17], sol–gel [8,14], combustion [18], ultrasound, and



time, pH, and stoichiometric ratio between the components, have to be well controlled [16,21]. In [14], NdFeO₃ particles with an average size in the range of 200–1100 nm were obtained via a sol–gel process using starch, and they were annealed at 1600 K. Here, the coercive force H_c and magnetization M_s were 900 kA.m⁻¹ and 1.5 A·m²·kg⁻¹, respectively. The products of nano-NdFeO₃ associated with these values of H_c and M_s are commonly used in the fabrication of devices working in a strong magnetic field. Note that to achieve saturation magnetization in a strong applied magnetic field, all NdFeO₃ particles must be in the superparamagnetic state that can be obtained by decreasing the size of NdFeO₃ particles from the microscale to the nanoscale.

Pure RFeO₃ orthoferrites (where R is La or Y), as well as RFeO₃ orthoferrites doped by Ca, Ba, Zn, and Co cations, can be produced by the following two-step approach without any surfactant: (1) the hydrolysis of cations in boiling water and (2) precipitation using appropriate agents [22–27]. This two-step process to synthesize the nanoscale NdFeO₃ has not been reported in the literature so far. Moreover, the benefit of the study on the thermal behaviors of individual or mixed Nd (III) and Fe (III) hydroxides in the synthesis of NdFeO₃ nanoscale materials has not been considered. Therefore, this study aims to (1) fabricate NdFeO₃ nanoscale materials via a simple precipitation of Fe (III) and Nd (III) cations in boiling water by using appropriate inorganic agents and (2) indicate the benefit of the study on the thermal behaviors of NdFeO₃ nanoscale materials of NdFeO₃ nanoscale materials and (2) indicate the benefit of the study on the thermal behaviors of NdFeO₃ nanoscale materials agents and (2) indicate the benefit of the study on the thermal behaviors of individual or mixed Nd (III) and Nd (III) cations in boiling water by using appropriate inorganic agents and (2) indicate the benefit of the study on the thermal behaviors of individual or mixed Nd (III) and Fe (III) hydroxides in the synthesis of NdFeO₃ nanoscale materials.

2. Experimental

2.1. Synthesis of Fe₂O₃, Nd₂O₃ and NdFeO₃ Nanoscale Particles

The procedure to synthesize the Fe₂O₃ or Nd₂O₃ nanoscale particles was proposed as follows: A certain amount of Fe(NO₃)₃ (or Nd(NO₃)₃) is slowly added into hot water (t > 90 °C) placed on top of a magnetic stirrer at the rate of one drop per second. The mixture is further stirred for 10 min and then let to cool down to ~30 °C. In the next step, the NaOH solution (5%) is slowly added to the mixture under continuous stirring until the color of the solution turns light pink due to the phenolphthalein indicator. The precipitate is filtered and washed with distilled water until a neutral pH is obtained. This precipitate is then naturally dried at room temperature for three days, milled into powder, and annealed at an appropriate temperature that is decided after analyzing thermo-gravimetric analysis–differential scanning calorimetry (TGA–DSC) data. The NdFeO₃ nanoscale particles are synthesized by using the appropriate ratio of mixed Fe(NO₃)₃ and Nd(NO₃)₃ solutions, following the steps presented in the introduction.

2.2. Characteristics

The thermal behaviors of the precipitates were investigated with a TGA–DSC analyzer (Labsys Evo, TG-DSC 1600 °C, SETARAM Instrumentation, Caluire, France). The samples were placed in a platinum cylindrical crucible and heated from 30 to 1000 °C at 10 K.min⁻¹ in dried air.

The morphology and size of NdFeO₃ nanoparticles were determined by transmission electron microscopy (TEM, Jeol JEM-1400, Jeol Ltd., Tokyo, Japan)) and field emission scanning electron microscopy (FESEM, S-4800, Hitachi, Japan). Elemental composition, which was taken from an average of five different positions in each sample, was determined by energy-dispersive X-ray spectroscopy (EDX, Horiba H-7593, Horiba, Northampton, UK). The magnetic characteristics of the samples (coercive force H_c , remanent magnetization M_r , and saturation magnetization M_s) were measured at room temperature with a vibrating sample magnetometer (VSM) MICROSENE EV11 (Japan) with a maximum applied field of 16000 Oe.

The structure and phase composition of the materials were investigated via X-ray powder diffraction (XRD, D8-ADVANCE, Bruker, Bremen, Germany) with CuK α radiation (λ = 1.5406 Å). Here, the range of 2 θ was 20–80° with a step size of 0.02°. The crystallite sizes of NdFeO₃ were calculated as the full width at half maximum (FWHM) of the most intense diffraction peaks, based on instrumental broadening correction, according to the Debye–Scherrer equation [20]:

$$D = \frac{0.89\lambda}{\beta\cos\theta} \tag{1}$$

where *D* is the average diameter of the crystallite, 0.89 is the Debye–Scherrer constant, λ is the CuK α wavelength of the incident X-radiation (0.15406 nm), β is FWHM, and θ is the diffraction angle of the maximum reflection.

The lattice parameters and cell volume were calculated by Equations (2) and (3), respectively [20]:

$$\frac{1}{d^2} = \frac{h^2}{a^2} + \frac{k^2}{b^2} + \frac{l^2}{c^2}$$
(2)

$$V = a \times b \times c \tag{3}$$

where d (Å) is the distance between crystalline planes with Miller indices (h, k, and l); a, b, and c are the lattice parameters; and V is the cell volume.

3. Results and Discussion

3.1. Thermal Behaviors of the Precipitates Containing Fe (III) or/and Nd (III)

The TGA/DSC results of the precipitate containing Fe (III) are shown in Figure 1. The TGA curve shows that the onset of initial weight loss (~20.9%) started near 60 °C, reached a maximum at ~122 °C, and had a cut-off at ~200 °C. The DSC curve presents a large endothermic peak at ~122 °C, attributed to the decomposition of Fe(OH)₃ to FeOOH, according to Reaction (4) as follows:

$$Fe(OH)_3 \longrightarrow FeOOH + H_2O$$
 (4)

$$\Delta m\% = \frac{18}{55.85 + 17 \times 3} \times 100\% = 16.8\%$$
(5)

The difference between the TGA results and the theoretical calculation (~4.1%) may have been caused by the exposure of the sample to the atmosphere. The weight loss between 200 and 330 °C (~9.0%) may have stemmed from the decomposition of FeOOH to γ -Fe₂O₃, according to Reaction (6) as follows:

$$2FeOOH \longrightarrow \gamma - Fe_2O_3 + H_2O \tag{6}$$

$$\Delta m\% = \frac{18}{2 \times (55.85 + 33)} \times 100\% = 10.1\% \tag{7}$$

The difference between the TGA results and the theoretical calculation at this stage was ~1.1%. The DSC curve exhibits a large exothermic peak at ~295 °C, which can be attributed to the phase transition of γ -Fe₂O₃ $\rightarrow \alpha$ -Fe₂O₃ [28]. Above 330 °C, no further weight loss occurred, indicating that there was no change in the phase of the product.

The TGA/DSC results of the precipitate containing Nd (III) are shown in Figure 2.

The TGA curve shows the total weight loss (~27%) from around 60 to ~750 °C. Assuming that the formula of the precipitate containing Nd (III) was Nd(OH)₃, it can consequently be stated according to Reaction (8), with a theoretical weight-loss of 13.8%:

$$2Nd(OH)_3 \longrightarrow Nd_2O_3 + 3H_2O \tag{8}$$

The difference between the TGA result and the theoretical calculation (~13.2%) can be explained as follows: the precipitate consisted not only of Nd(OH)₃ hydroxide but also Nd₂O(CO₃)₂·1.4H₂O or Nd₂(CO₃)₃·8H₂O due to the adsorption of CO₂ by the sample exposed to the atmosphere. This is a natural phenomenon of rare earth lanthanide elements [29–31]. The DSC curve presents endothermic peaks that can be attributed to the decomposition of compounds of Nd, such as Nd(OH)₃ and Nd₂O(CO₃)₂·1.4H₂O or Nd₂(CO₃)₃·8H₂O. Above 750 °C, no further weight loss occurred, indicating that there was no change in the phase of the product. Based on the thermal behaviors of the precipitates containing Fe or Nd, perovskite-type neodymium orthoferrite NdFeO₃ can be simply synthesized by annealing the mixed Fe (III) and Nd (III) hydroxide at 750 °C. This feature can be observed from the TGA/DSC results of the precipitate containing Nd (III) and Fe (III), as seen in Figure 3.



Figure 1. Thermo-gravimetric analysis/differential scanning calorimetry (TGA/DSC) results of the precipitate containing Fe (III).



Figure 2. TGA/DSC results of the precipitate containing Nd (III).



Figure 3. TGA/DSC results of the precipitate containing Nd (III) and Fe (III) (the mol ratio of Fe^{3+} to Nd³⁺ is 1/1).

The TGA curve (Figure 3) shows that the total weight loss of the mixture of Nd (III) and Fe (III) annealed at ~750 °C was 30.75%. Therefore, there was a basic carbonate of Nd (III) in the component of the precipitate, in addition to Fe(OH)₃ and Nd(OH)₃. Indeed, if a precipitate consists of Fe(OH)₃ and Nd(OH)₃, its total weight loss must be around 17.80% (Reaction (9)).

$$Nd(OH)_{3} + Fe(OH)_{3} \xrightarrow{750^{\circ}C} NdFeO_{3} + 3H_{2}O$$
(9)

$$\Delta m\% = \frac{18 \times 3}{106.85 + 195.24} \times 100\% = 17,8\%$$
(10)

All thermal decomposition processes of $Fe(OH)_3$ and $Nd(OH)_3$, as well as the basic carbonate of Nd (III), have endothermic effects, as presented in the TGA–DSC curves at the peaks of 268.7, 326.7, and 681.3 °C (Figure 3). The DSC curve displays an exothermic peak at ~750 °C that can be attributed to the formation of the NdFeO₃ phase, according to Reaction (11), in agreement with the TGA–DSC results shown in Figure 2.

$$Nd_2O_3 + Fe_2O_3 \xrightarrow{750^{\circ}C} 2NdFeO_3$$
 (11)

As a result, the NdFeO₃ perovskite can be synthesized by annealing the mixed Fe (III) and Nd (III) hydroxide at 750 $^{\circ}$ C for 1 h.

3.2. XRD Results

Figure 4 shows the XRD results of NdFeO₃ orthoferrite formed after annealing at 750 °C for 1 h. The pattern shows that the sample peaks had the typical pattern of the NdFeO₃ orthorhombic phase, in agreement with the powder diffraction file (PDF) No. 00-074-1473. The crystallite size of NdFeO₃ orthoferrite, calculated according to Equation (1), was $D_{XRD} = 28.41$ nm. This result indicates that the crystalline NdFeO₃ particles possessed an average crystallite size that was smaller than that of the particles that were fabricated via ultrasound or co-precipitation techniques using octanoic acid as an organic surfactant [19,20], whereas the lattice parameters of the perovskite-type neodymium orthoferrite NdFeO₃ calculated by Equations (2) and (3) were not significantly different from those associated with the samples reported in [19,20] (Table 1).

Table 1. Lattice parameters and magnetic properties of crystalline NdFeO₃ particles annealed at 750 °C.

D (nm)	a (Å)	b (Å)	c (Å)	V (Å ³)	$M_{\rm s}$ (emu/g)	<i>Н</i> _с (Ое)	$M_{ m r}$ (emu/g)
28.41	5.4990	5.5910	7.7592	238.5559	0.797	136.76	0.68



Figure 4. XRD patterns of NdFeO₃ formed at 750 °C.

3.3. SEM, TEM, and EDX Results

SEM and TEM images show that the crystalline NdFeO₃ nanoparticles were uniform in terms of spherical granules with diameters of ~30 nm (Figure 5). The chemical composites at five different positions in the sample were determined via EDX and EDX-mapping spectra. The EDX and EDX-mapping results show the peaks of Nd, Fe, and O without any unexpected contamination (Figure 6). The percentages of the masses of Nd, Fe, and O in the sample (calculated as an average value at five various positions) were slightly different from those obtained theoretically ($|m_{exp}/m_{calc}| = 95-97\%$).



Figure 5. (a) SEM and (b) TEM images of crystalline NdFeO₃ nanoparticles annealed at 750 °C for 1 h.



Figure 6. Energy-dispersive X-ray spectroscopy (EDX) (left) and EDX-mapping spectrums (right) of NdFeO₃ nanoparticles annealed at 750 °C for 1 h.

3.4. VSM Results

We proceeded to analyze the following magnetic properties (measured at ~30 °C) of NdFeO₃ nanoparticles: the saturation magnetization M_s of 0.797 emu·g⁻¹, the coercive force H_c of 136.76 Oe, and the remanent magnetization M_r of 0.68 emu·g⁻¹, which was quite small. Note that the magnetization did not yet approach the saturation value at a very high magnetic field around 16,000 Oe, as seen in Figure 7, which describes the monochromatic increase of the magnetization as the field grew. Meanwhile, ceramic NdFeO₃ nanoparticles typically achieve a saturation magnetization of $M_s < 0.7$ emu/g or $M_s > 1.0$ emu/g, as presented in [32] and [14], respectively, at the external magnetic field of 600 Oe. Another interesting feature here is that the saturation magnetization of the NdFeO₃ nanoparticles was significantly higher than those of other orthoferrites of rare-earth elements RFeO₃ (R = La, Y) [23–27]. Therefore, the NdFeO₃ nanoparticles have potential applications to fabricate devices that can be applied in a high magnetic field.



Figure 7. Field dependence of the magnetization of the NdFeO₃ nanoparticles annealed at 750 °C for 1 h.

4. Conclusions

As shown in this study, crystalline NdFeO₃ nanoparticles can be simply synthesized via by hydrolyzing Nd (III) and Fe (III) cations in hot water with an alkaline agent and annealing at 750 °C for 1 h. The typical pattern of the iron neodymium oxide single-phase (NdFeO₃, orthorhombic) was observed in typical peaks of the XRD result. The SEM and TEM results showed the crystalline NdFeO₃ in sphere nanoparticles with average diameters of around 30 nm. The magnetic properties of the material were comprehensively investigated, with a coercive force (H_c) of 136.76 Oe, a remanent magnetization (M_r) of 0.68 emu·g⁻¹, and a saturation magnetization (M_s) of 0.797 emu·g⁻¹. The results provide a sound foundation for the application of this magnetic material based on its high saturation magnetization.

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