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Facile Synthesis of Inverse Spinel $NiFe_2O_4$ Nanocrystals and their Superparamagnetic Properties

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Spinel $NiFe_2O_4$ nanocrystals have been obtained by means of a novel composite-hydroxide-salt-mediated approach, which is based on a reaction between metallic salt and metallic oxide in the solution of composite-hydroxide-salt eutectic at ~225 °C and normal atmosphere without any organic dispersant or capping agent. The obtained products are characterized by an X-ray diffraction (XRD), a transmission electron microscopy (TEM) and an alternating gradient magnetometer (AGM). The formation process of $NiFe_2O_4$ nanosheet is proposed to begin with a ''dissolution-recrystallization'' which is followed by an ''Ostwald ripening'' mechanism. The $NiFe_2O_4$ nano-octahedrons can be obtained through adjusting the reaction water content in the hydroxide melts at constant temperature. At 300 K, magnetic hysteresis loops at an applied field of 15 kOe show zero coercivity, indicating the superparamagnetic behavior of the as-prepared $NiFe_2O_4$ nanocrystals.

Keywords: *$NiFe_2O_4$ nanocrystals, morphology, mechanism, magnetic properties*

1. Introduction

Nanocrystalline spinel ferrites with the general formula MFe_2O_4 (M = divalent metal ions, e.g. Ni, Co, Cu, Zn, Mg, Mn, Cd, etc.) are attractive for their interesting magnetic, magnetoresistive, and magneto-optical properties. As a kind of important spinel ferrite, $NiFe_2O_4$ has attracted much interest because of its fascinating magnetic and electromagnetic properties[1]. $NiFe_2O_4$ powders have been studied to use in many fields, such as ferrofluids[2,3], catalysts[4], gas sensors[5-8], biomedicine[9] and so on[10-12]. $NiFe_2O_4$ is a cubic ferromagnetic oxide with a typical inverse spinel structure where Ni^{2+} ions occupy octahedric B-sites and Fe^{3+} ions equally distributed between tetrahedric A-sites and octahedric B-sites[13]. Nanoscale $NiFe_2O_4$ ferrites have been successfully synthesized by various methods including sol-gel[11,14-16], solid-state reaction[6,17-18], co-precipitation[7,12,19,20], mechanochemical[21], rheological phase reaction method[22], pulsed wire discharge[23], arc plasma assisted gas phase synthesis method[24], combustion[25], surfactant-assisted refluxing method[26], micromulsion[27], electrospinning[28], thermolysis of mixed metal-oleate complexes[29], sonochemical[30] and hydrothermal[31-35].

In this paper, we report a novel one-step, simple method to directly synthesize uniform, mass-production nanostructured $NiFe_2O_4$ by the composite-hydroxide-salt-mediated (CHSM) method. The method is based on the reaction between one metallic oxide and the other metallic oxide in a solution of molten mixed potassium nitrate and potassium hydroxide eutectic at ~225 °C and normal atmosphere. Furthermore, because of no addition of organic dispersant or capping agent in the reaction system, the final product can be easily purified. The main advantage of this method is the easy recycle of by-products, due to application of the salt nitrate and hydroxide of the same metal. The morphology of $NiFe_2O_4$ can be controlled by adjusting the content of H_2O and reaction duration. The reaction mechanism has also been discussed. In addition, the dependence of magnetic properties on morphologies and grain size of final products has been clearly observed.

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2. Experimental

All chemicals used, such as $NiCl_2·6H_2O$, Fe_2O_3, were analytical grade reagents and without any further purification. Synthesis of $NiFe_2O_4$ nanomaterials was carried out by the composite-hydroxide-salt-mediated (CHSM) method without using any capping agent. The synthesis steps are as follows: (1) a total of 20 g of KOH and KNO_3 was mixed at a ratio of 63.8:36.2 and placed in a 25ml Teflon vessel. (2) A mixture of 0.5 mmol $NiCl_2·6H_2O$ and 0.5 mmol Fe_2O_3 each was used as the raw material for reaction, 0-2 mL H_2O were also put into the vessel, and was placed on the top of the hydroxide and salt nitrate in the vessel. (3) Then, the Teflon vessel was put into a furnace preheated to 235 °C. (4) After the hydroxide and salt nitrate were totally molten (about 30 minutes later), the molten reactants were mixed uniformly by shaking the covered vessel. (5) After the setting reaction time (12, 24, 48 hours), the vessel was taken out and cooled to room temperature naturally. The product was collected by centrifugation and through washing with deionized water and ethanol. (6) The products ($NiFe_2O_4$) synthesized at 235 °C with 0 mL H_2O for 12 hours, 24 hours, 48 hours and with 2 mL H_2O for 48 hours were designated as sample S1, S2, S3 and S4, respectively.

Phase analysis of the products was performed by X-ray diffraction measurement (XRD, D8-Advance, Germany) with the use of Cu *K*α radiation (λ = 1.5418 Å) in the 2θ range from 20° to 70° . The morphology and the size of the synthesized samples were characterized by a transmission electron microscopy (TEM, JEM-100CXII, Japan) with the emission voltage of 120 kV. The magnetic hysteresis loops was obtained at room temperature on a alternating gradient magnetometer (AGM, Micromag Model 2900) with a maximum external field $H_m \approx 1194$ kA.m^{-1} (15 kOe).

3. Results and Discussion

The crystal structure and phase purity of the synthesized products was characterized by XRD. Figure 1 shows the XRD patterns of samples S1-S4. It can be seen in Figure 1a that S1 was poorly crystallized. However, the crystallization quality of $NiFe_2O_4$ was improved with the increase of reaction time (Figure 1b, c). The labeled diffraction peaks at (311), (222), (400) and (440) reveals the information of typical inverse spinel structure according to the standard value for bulk $NiFe_2O_4$ phase (JCPDS file No.22-1086). In the XRD pattern of sample S4 that 2 mL water was used during reaction (Figure 1d), the characteristic diffraction peak (220) of spinel $NiFe_2O_4$ begins to emerge. The results of XRD indicate that the crystallinity of samples is dependent of reaction time and water content.

Figure 2 gives the TEM images of samples S1-S4. In Figure 2a, a large number of amorphous products (the dark in the figure) as well as a small number of $NiFe_2O_4$ nanocrystals formed in S1, which agrees with the weak peaks in XRD spectrum (Figure 1a). However, only a few amorphous product is found in Figure 2b, and it almost disappears in Figure 2c. Irregular morphology of the $NiFe_2O_4$ nanosheets with peculiar shape was formed in S2 and S3. The possible growth mechanism has been presented as "oriented aggregation" of primary nanoparticles, which involve self-assembly of adjacent particles in a common crystallographic orientation and joined at a planar interface. This involves the spontaneous decrease of overall energy in the whole system[12,36]. However, when 2 mL is used, $NiFe_2O_4$ nano-octahedrons instead of nanosheets were obtained in S4 (Figure 2d). Our experiments have revealed that the morphology can be controlled through adjusting the reaction water content in the hydroxide melts with constant temperature.

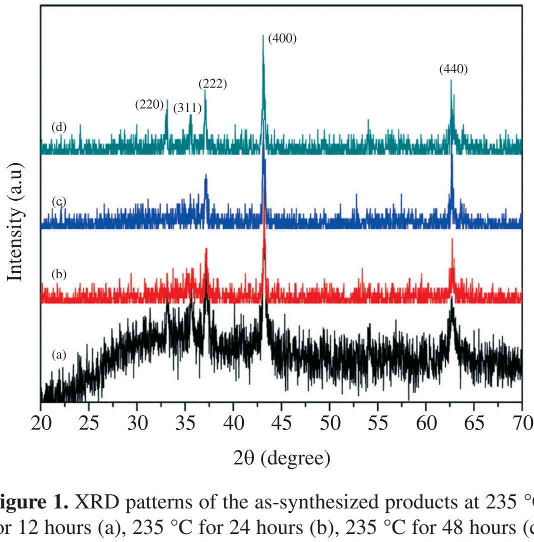

Figure 1. XRD patterns of the as-synthesized products at 235 °C for 12 hours (a), 235 °C for 24 hours (b), 235 °C for 48 hours (c) and 235 °C for 48 hours with 2 mLH_2O (d).

Based on the time-dependent morphology evolution described above, the formation process of $NiFe_2O_4$ nano-octahedrons could be proposed to be the ''dissolution-recrystallization'' firstly and then be the ''Ostwald ripening'' mechanism. At the initial stage, a large amount of nano-crystallites nucleate and grow into nanosheets to minimize the overall energy of the system. A small amount of water in the hydroxide melts could alter the viscidity and acidity in the melts, which may be the key factor to affect phase crystallization[37]. Owing to water addition, quick nucleation and growth rate may directly result in the formation of $NiFe_2O_4$ nano-octahedrons. As is known, the KOH/KNO_3 composite melts possess large viscidity but H_2O reduces the viscidity in the melts and increases the diffuse ability of the melt atoms. As reported by Wang[38], the ratio of growth rate along the <100> and <111> directions (R) determines the geometrical shape of a crystal. The octahedron consisting of eight highly stable {111} planes resulted from a much higher growth rate along the <100> direction than the <111> direction due to the lowest energy of the {111} surfaces. Generally, low reaction rate is favorable to fully exhibit the crystalline habit. The theoretical growth habit of the MFe_2O_4 oxometallates crystal is the octahedron because the {111} surfaces have the lowest energy[39]. Here, the inverse spinel $NiFe_2O_4$ nanocrystalline has an octahedron shape.

From the above experimental results, a possible reaction mechanism for the synthesis of $NiFe_2O_4$ in hydroxide and salt solution is suggested as follows. Although the melting points (T_m) of both pure potassium hydroxide and potassium nitrate are over 300 °C, T_m = 404 °C for KOH and T_m = 337 °C for KNO_3, the eutectic point at KOH/KNO_3 = is only about 225 °C, the eutectic point at KOH/KNO_3 = 63.8:36.2 is only about 225 °C. During the reaction process, hydroxides work not only as the solvent but also as the reactant for reducing the reaction temperature. In the molten hydroxide, Fe_2O_3 reacts with KOH to form a hydroxide-soluble $K_2Fe_2O_4$.

$$Fe_2O_3 + 2KOH = K_2Fe_2O_4 + H_2O \quad (1)$$

At the same time, $NiCl_2$ reacts with hydroxide to form $Ni(OH)_2$, which is dissolved in the hydroxide solutions:

$$NiCl_2 + 2KOH = Ni(OH)_2 + 2KCl \quad (2)$$

The $K_2Fe_2O_4$ produced in (1) reacts with the $Ni(OH)_2$ produced in process (2) and to form an indissoluble solid $NiFe_2O_4$:

$$Ni(OH)_2 + K_2Fe_2O_4 = NiFe_2O_4 + 2KOH \quad (3)$$

Because the viscosity of hydroxide is large, the formation of $NiFe_2O_4$ is slow and it is not easy for the nanostructures to agglomerate, which maybe the key for receiving dispersive single-crystalline nanostructures during

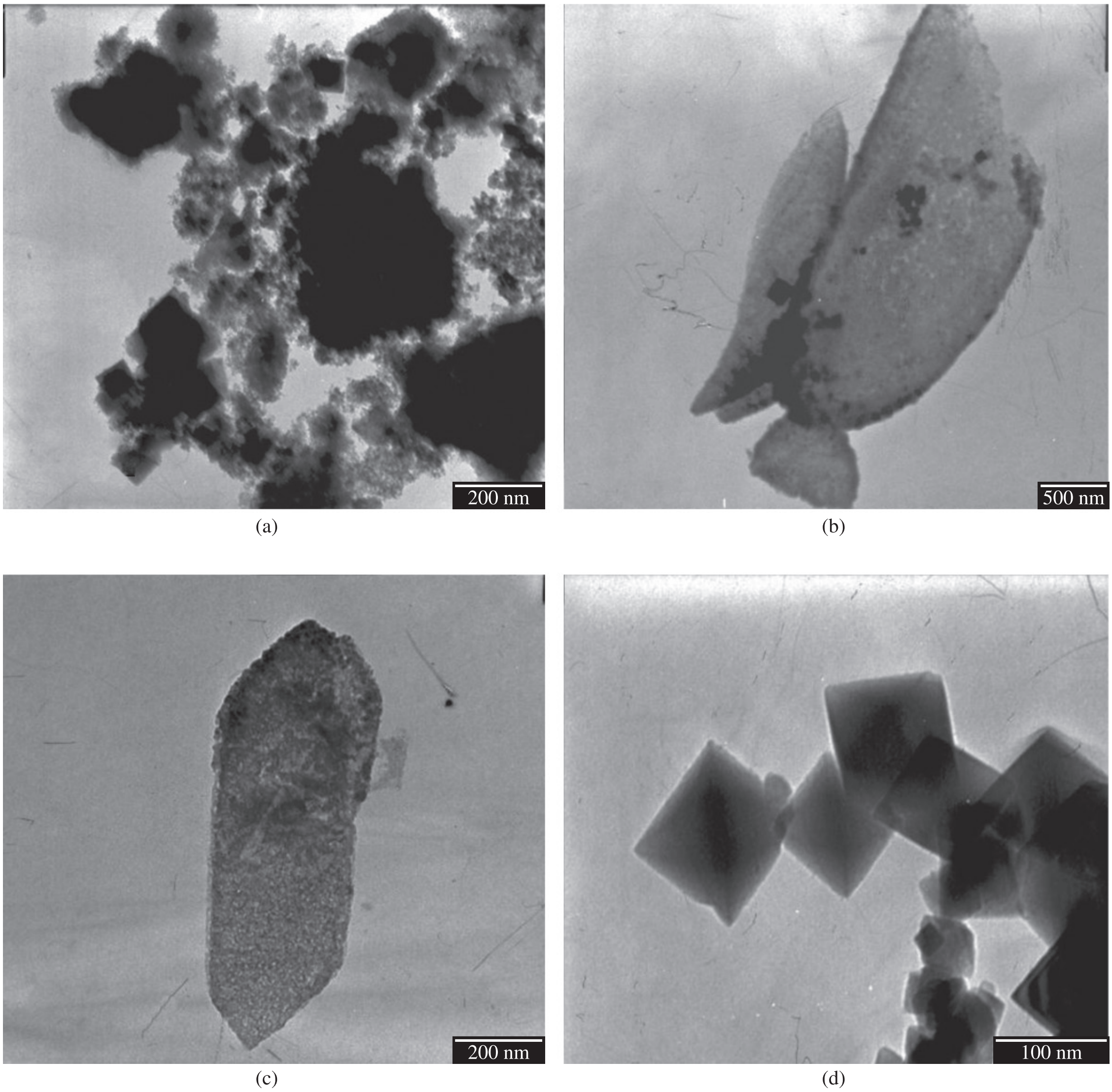

Figure 2. TEM images of the $NiFe_2O_4$ nanocrystals synthesized at 235 °C for 12 hours (a), 235 °C for 24 hours (b), 235 °C for 48 hours (c) and 235 °C for 48 hours with 2 mL H_2O (d).

the reaction without using an organic surface-capping material. The hydroxides mediate the reaction, but they are not part of the final nanostructures.

The magnetic properties of this nanostructured ferrite system were studied with the help of AGM. Figure 3 shows the hysteresis loops of sample S1, S3, S4. Zero coercivity features the three $NiFe_2O_4$ nanocrystals, which indicates the presence of superparamagnetic behavior. The saturation magnetization (M_s) are about 22.9, 41.4 and 48.5 emu.g^{-1} for S1, S3 and S4, respectively, as shown in Figure 3. The reported value of the saturation magnetization, calculated using Neel's sublattice theory for cubic inverse spinel $NiFe_2O_4$ is 50 emu.g^{-1}[40] and reported value of the saturation magnetization experimentally observed for bulk $NiFe_2O_4$ is 56 emu.g^{-1}[41]. Chkoundali et al.[42] pointed out that the large values of saturation magnetizations usually correlated with the highest crystallinity of the nanoparticles. Therefore, compared with bulk $NiFe_2O_4$ ferrite, due to the different

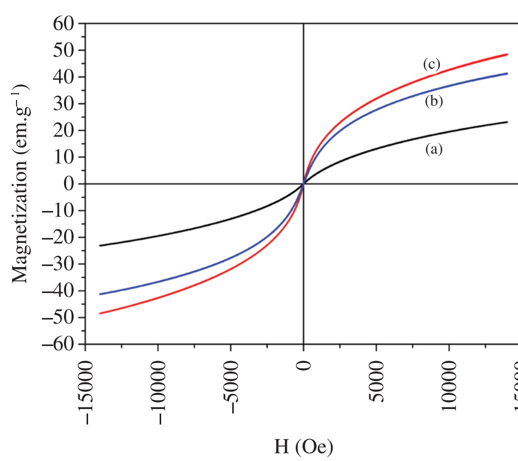

Figure 3. The magnetization curves of the nickel ferrite nanocrystals synthesized at 235 °C for 12 hours (a), 235 °C for 48 hours (b) and 235 °C for 48 hours with 2 mL H_2O (c).

crystallinity as shown in Figures 1 and 2, the smaller M_s and the increasing trend from S1 to S4 in our case is rational.

4. Conclusions

In summary, $NiFe_2O_4$ nanocrystals have been synthesized via the CHSM approach, which is simple, low-cost, high-yield and easy-recycle for by-products. The crystallization and morphology of $NiFe_2O_4$ nanocrystals can be controlled through adjusting the reaction water content in the hydroxide melts with constant temperature. The magnetic hysteresis loops with zero coercivity at room temperature suggest the superparamagnetic behavior of $NiFe_2O_4$ nanocrystals.

Acknowledgements

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