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Short communication

Magnetic properties of nanosize $NiFe_2O_4$ particles synthesized by sol–gel auto combustion method

J. Azadmanjiri [a,*], S.A. Seyyed Ebrahimi [b], H.K. Salehani [a]

[a] *Islamic Azad University-Damavand Branch, P.O. Box 39715-194 Damavand, Iran*
[b] *Center of Excellence in Magnetic Materials, Faculty of Engineering,*
Department of Metallurgy and Materials, University of Tehran, Tehran, Iran

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Abstract

A nitrate–citrate gel was prepared from metal nitrates and citric acid by sol–gel auto combustion process, in order to synthesize $NiFe_2O_4$ ferrite. X-ray diffraction showed the formation of $NiFe_2O_4$ and the inclusion of α-Fe_2O_3. However, with increasing the calcination temperature, this inclusion phase (α-Fe_2O_3) decreased. TEM was also used to characterize the microstructure of the nanosize $NiFe_2O_4$. Magnetization hysteresis of the nickel ferrite powders was performed at room temperature. It is shown that this powder which is composed of crystalline $NiFe_2O_4$ monophase, exhibited $M_s = 54$ (emu/g) at magnetic field of 2 (kOe).

Keywords: A. Sol–gel processes; C. Magnetic properties; Nickel ferrite; Auto combustion

1. Introduction

The nickel ferrite powder has been successfully synthesized by solid-state reaction [1], high-temperature self-propagating [2], microemulsion [3,4], solvothermal and hydrothermal synthesis [5–10] and other methods [11,12]. In this work nanosize $NiFe_2O_4$ particles were synthesized by the sol–gel auto combustion method. The combustion process is an oxidation–reduction reaction in which the NO_3^- ion is oxidant and the carboxyl group is reductant. Due to the fact that the properties and application aspects of the ultrafine powders depend strongly on the particle size and morphology, the preparation of ultrafine particles well defined in size and morphology has attracted more interest in recent years [13–15]. Here, for the first time preparation of uniform nickel ferrite particles is reported. With the metal nitrates, citric acid and ammonia precursor by sol–gel auto combustion method and magnetization properties of crystalline $NiFe_2O_4$ monophase are also carried out.

2. Experimental

The chemical materials used in experiments were $Fe(NO_3)_3{\cdot}9H_2O$, $Ni(NO_3)_2{\cdot}6H_2O$, $C_6H_8O_7{\cdot}H_2O$ and NH_4OH to prepare $NiFe_2O_4$ ferrite material. Appropriate amounts of metal nitrates and citric acid were first dissolved in a minimum amount of de-ionized water. The molar ratio of nitrates to citric acid was 1:1. A small amount of ammonia was added to the solution to adjust the pH value at about 7. Then, the mixed solution was poured into a dish and heated and stirred to transform into a xerogel. When ignited points were observed, the dried gel burnt in a self-propagating combustion manner until all the gel was burnt out completely to form loose powder. The powder was then calcined at different temperatures. The phase identification, magnetization hysteresis and microstructure of the calcined powder were performed by XRD, VSM and TEM, respectively.

3. Results and discussion

Fig. 1 shows the XRD patterns of dried gel and as-burnt powder. The dried gel powder is amorphous in nature. The as-burnt powder is a small quantity of nickel ferrite as well as α-Fe_2O_3 phase. This indicates that the $NiFe_2O_4$ ferrites are directly formed after auto combustion of gels. The direct

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* Corresponding author. Tel.: +98 2122543654; fax: +98 2122543654.
E-mail address: Jalal_azad2000@yahoo.com (J. Azadmanjiri).

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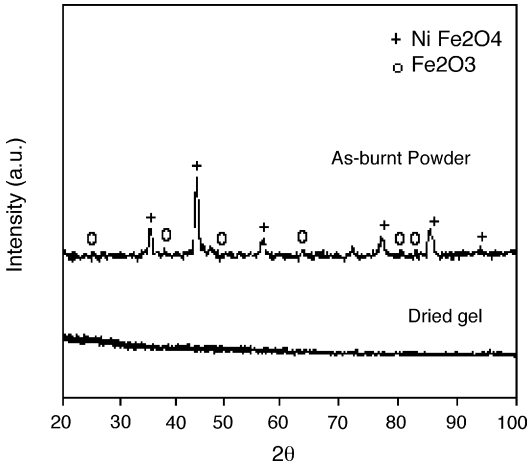

Fig. 1. XRD patterns for the dried gel and as-burnt powder.

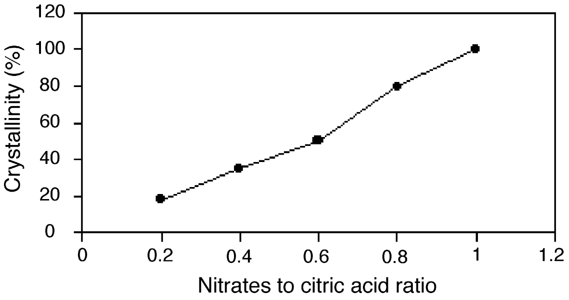

Fig. 2. Crystallization behavior of nitrates to citric acid ratio.

transformation of crystalline ferrite from amorphous gel during combustion is surely due to the higher degree of compositional homogeneity and the greater heat generated from the exothermic reaction of nitrates and citric acid. As the carbon chains in citrates are decomposed during combustion, adjacent atoms that are homogeneously distributed throughout the matrix can easily come into contact and form crystal lattice at a considerable rate. It was also observed that crystallization of the $NiFe_2O_4$ accelerated with increasing ratio of nitrates to citric acid up to 1:1 (Fig. 2), at this ratio gel burns completely, no carboxyl groups and NO_3^- ions remain. In fact, for an oxidation–reduction reaction, the reaction is most violent and complete only when the ration of oxidants to reductants is 1:1.

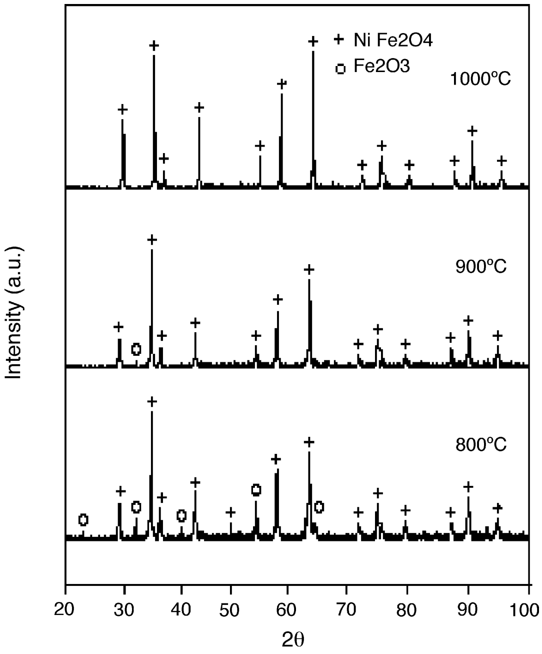

Fig. 3. XRD patterns corresponding to calcined powders at the range of 800–1000 °C for 1 h with Fe/Ni ration of 2.

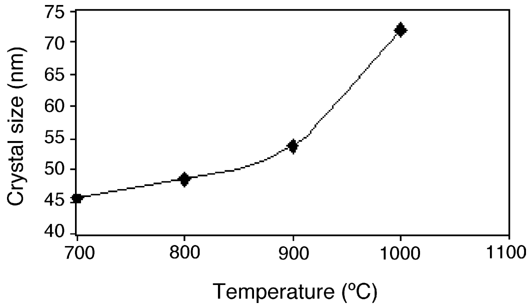

Fig. 4. The effect of temperature on the crystallite size.

X-ray diffraction studies also have been carried out on the calcined powder at the range of 800–1000 °C (Fig. 3). The phases observed in 800 and 900 °C were α-Fe_2O_3 and $NiFe_2O_4$ with increasing the calcination temperature, only at 1000 °C single phase $NiFe_2O_4$ could be obtained. Hence, increasing the calcination temperature was beneficial in order to eliminate that deteriorate the magnetic properties and to form single phase $NiFe_2O_4$. Fig. 4 shows the effect of temperature on the crystallite size. The crystal size was determined from the line width of the reflection (3 1 1) in XRD patterns using the Scherrer's formula. It was found that crystal size of the product was greatly accelerated with increasing of temperature and reaches to 72 nm at 1000 °C. The TEM photograph of the synthesized powder, shown in Fig. 5, indicates that the powder is uniform in particle size. The particle size is about 70–80 nm, as measured using XRD method. This reveals that the synthesized powder is well dispersed and no aggregates are

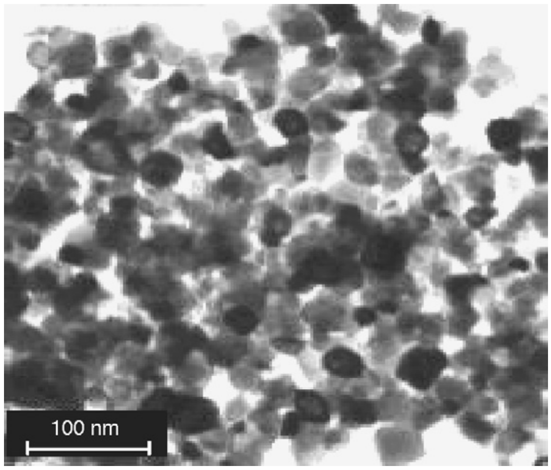

Fig. 5. TEM photograph of the synthesized powder.

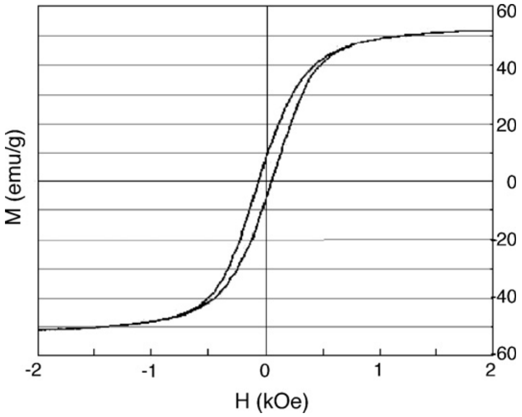

Fig. 6. The hysteresis loop of the $NiFe_2O_4$ powders calcined at the 1000 °C for 1 h.

formed during combustion. Fig. 6 shows the magnetic characterization of the nickel ferrite powders was performed at room temperature and hysteresis loop of the typical product have been showed in this figure. Compared with the nickel ferrite powders synthesized by other method [16–18], the as-prepared product possessed higher saturation magnetization moment, $M_s = 54$ (emu/g), at magnetic field of 2 (kOe), which might be due to their high degree of crystallization and uniform morphologies [13].

4. Conclusions

A series of nitrate–citrate gels were prepared from metal nitrates and citric acid by a sol–gel process in order to synthesize $NiFe_2O_4$ ferrite. The study of the effect of the calcination temperature on the phase constitution indicated that Fe/Ni = 2 and calcination at 1000 °C resulted in formation of single phase $NiFe_2O_4$. The as-prepared nickel ferrite powders possessed higher saturation magnetization moment due to their high degree of crystallization and uniform morphologies.

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