World Academy of Science, Engineering and Technology
International Journal of Materials and Metallurgical Engineering
Vol:8, No:12, 2014

Production of Sr-Ferrite Sub-Micron Powder by Conventional and Sol-Gel Auto-Combustion Methods

M. Ghobeiti-Hasab

Abstract**—**Magnetic powder of Sr-ferrite was prepared by conventional and sol-gel auto-combustion methods. In conventional method, strontium carbonate and ferric oxide powders were mixed together and then mixture was calcined. In sol-gel auto-combustion method, a solution containing strontium nitrate, ferric nitrate and citric acid was heated until the combustion took place automatically; then, as-burnt powder was calcined. Thermal behavior, phase identification, morphology and magnetic properties of powders obtained by these two methods were compared by DTA, XRD, SEM and VSM techniques. According to the results of DTA analysis, formation temperature of Sr-ferrite obtained by conventional and sol-gel auto-combustion methods were 1300°C and 1000°C, respectively. XRD results confirmed the formation of pure Sr-ferrite at the mentioned temperatures. Plate and hexagonal-shape particles of Sr-ferrite were observed using SEM. The Sr-ferrite powder obtained by sol-gel auto-combustion method had saturation magnetization of 66.03 emu/g and coercivity of 5731 Oe in comparison with values of 58.20 emu/g and 4378 Oe obtained by conventional method.

Keywords**—**Sr-ferrite, Sol-gel, Magnetic properties, Calcination.

I. Introduction

PERMANENT magnetic material of Sr-ferrite needs to have a high coercivity; which it can be achieved by obtaining a powder finer than critical single-domain size (1 µm). Coercivity is the intensity of the magnetic field needed to reduce the magnetization of material to zero after it has reached saturation. The magnetic state that best exhibits this quality is the stable single-domain state. The magnetic state of a crystal is strongly dependent on both size and shape. Although very small grains are single domain, they are no longer magnetically stable owing to thermal agitation and display super-paramagnetic behavior. Larger grains above the single-domain size (multi-domains) leads to the decrease of coercivity [1]. Fig. 1 shows this subject schematically.

Industrially, Sr-ferrite is produced by conventional ceramic method, which involves mixing of the constituents ($SrCO_3$ and Fe_2O_3), calcination and prolonged ball milling of calcined material to yield sub-micron size powders. The powders prepared by this method require high temperature of calcination (> 1200°C) [2].

The various chemical routes such as hydrothermal [3], salt-melt [4], co-precipitation [5] and sol-gel auto-combustion [6] techniques are applied to synthesize this material with lower calcination temperatures and smaller particles.

Dr. Mehdi Ghobeiti Hasab is a faculty member of the Materials and Metallurgical Engineering Department, Dezful Branch, Islamic Azad University, Dezful, Iran (e-mail: ghobeiti@ut.ac.ir, ghobeiti@yahoo.com).

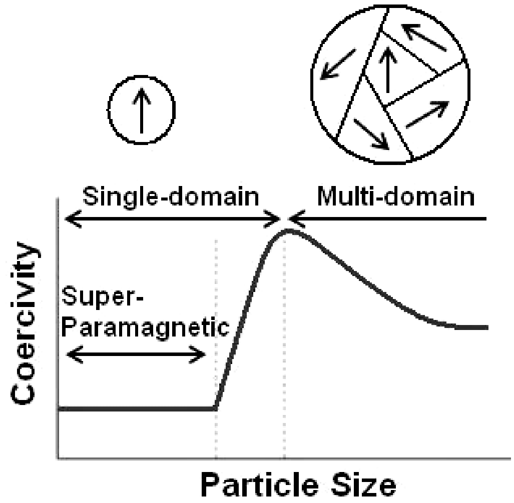

Fig. 1 Variation of coercivity with particle size

Sol-gel auto-combustion method is a technique for obtaining Sr-ferrite powder using an exothermic reaction between dissolved metal nitrates ($Fe(NO_3)_3$ and $Sr(NO_3)_2$) and an organic fuel (citric acid). Instead of using a high temperature furnace to supply the energy needed for reaction of the constituents, the reaction itself supplies the energy; therefore, the temperature needed for the formation of final phase can be decreased [7]-[11].

In this research, Sr-ferrite was produced by conventional and sol-gel auto-combustion methods. Then, formation temperature, morphology and magnetic properties of Sr-ferrite obtained by these methods were compared.

II. Experimental

In order to obtaining Sr-ferrite by conventional method, sub-micron powders of strontium carbonate and iron oxide with Sr/Fe molar ratio of 1/10 were mixed together in a mixer for 1 hour and then calcined at 1300°C for 1 hour. For obtaining the Sr-ferrite powder by sol-gel auto-combustion method, ferric nitrate and strontium nitrate in Sr/Fe molar ratio of 1/10 were dissolved in distilled water and then citric acid in citric acid/metallic nitrates molar ratio of 1/1 was added. pH of solution was adjusted in 7 by addition of ammonia. This solution was slowly evaporated until a gel was formed. The gel was dried and then heated on a plate dish until the combustion took place automatically. As-burnt powder was calcined at 1000°C in a resistance furnace with air atmosphere for 1 hour to form single-phase Sr-ferrite. The flow chart of sol-gel auto-combustion process is shown in Fig. 2. All the reagents used in this research were analytical grade and purchased from Merck.

The thermal behavior of powders was evaluated by differential thermal analysis (DTA). X-ray diffraction (XRD, Cu-Kα radiation) was used for phase identification of the calcined powders. The morphology of Sr-ferrite powder was observed by scanning electron microscopy (SEM). Vibrating sample magnetometry (VSM) was applied to characterize the saturation magnetization and coercivity of the powders.

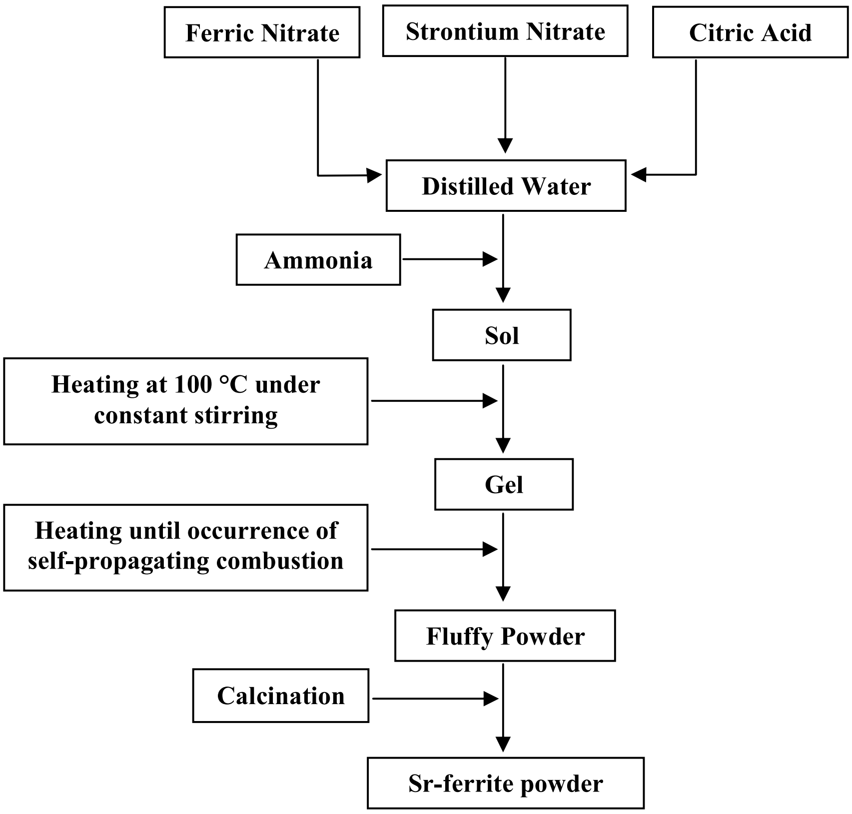

Fig. 2 Flow chart for the synthesis of Sr-ferrite powder by sol-gel auto-combustion method

III. Results and Discussion

The DTA curves of powders before calcination in air atmosphere are shown in Fig. 3. The exothermic peak marked in figure belongs to the crystallization of Sr-ferrite phase. For the powder obtained by conventional method, the formation temperature of Sr-ferrite is about 1300°C, while for the powder obtained by sol-gel auto-combustion method is about 1000°C. Decrease in the formation temperature of Sr-ferrite prepared by sol-gel auto-combustion method can be due to the more homogeneous composition and structure of powder. Also, probably, the finer particles obtained by sol-gel auto-combustion method are more reactive and need to the lower temperatures for the formation of the final phase.

The XRD patterns of the powders obtained by conventional and sol-gel auto-combustion methods in the range of 2θ:20-70° (the range of main diffraction peaks of Sr-ferrite phase) have been presented in Fig. 4. As shown, the Sr-ferrite phase is formed successfully in these two samples and no impurity phases are detected.

Fig. 5 shows the SEM micrograph of these two samples. According to this figure, plate like and hexagonal morphology of the particles obtained by sol-gel auto-combustion could be observed more clearly. The degree of particles agglomeration in the conventional method is much higher than the sol-gel auto-combustion method.

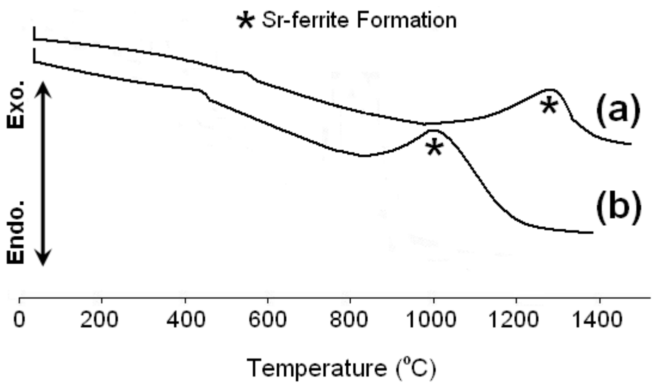

Fig. 3 DTA curve of powder obtained by (a) conventional method (b) sol-gel auto-combustion method, before calcination process

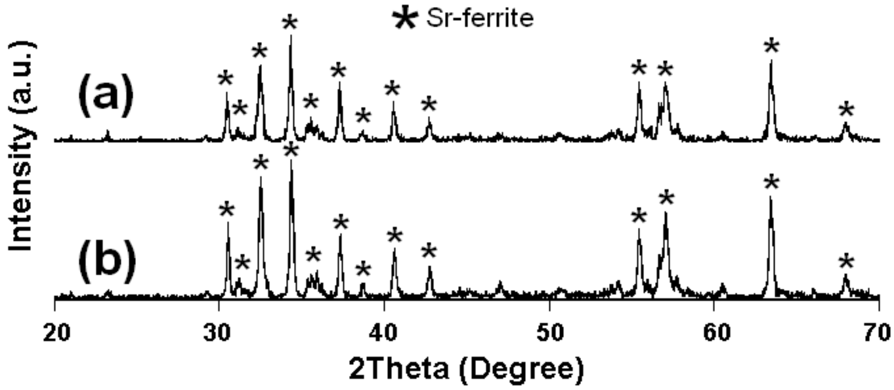

Fig. 4 XRD pattern of powder obtained by (a) conventional method (b) sol-gel auto-combustion method, after calcination process

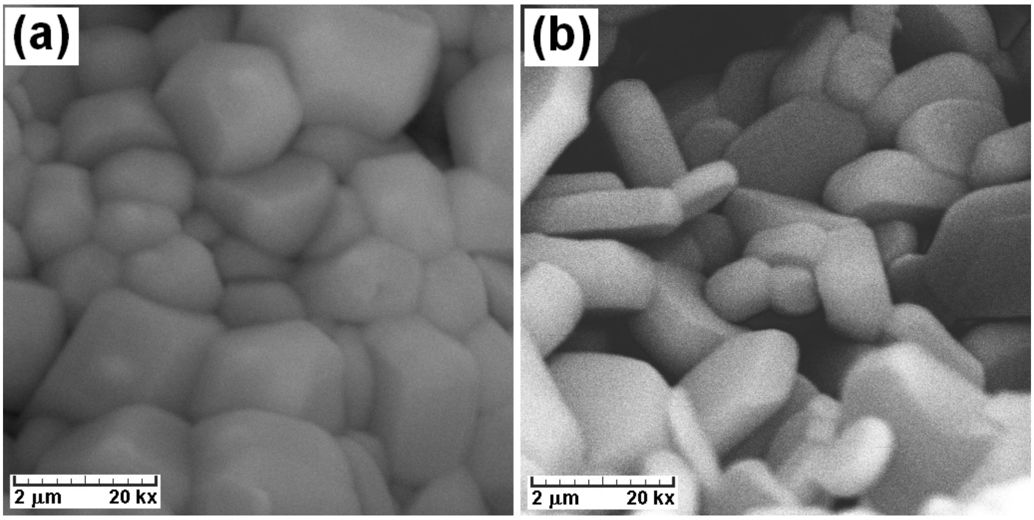

Fig. 5 Comparison of the morphology of Sr-ferrite powder obtained by (a) conventional method (b) sol-gel auto-combustion method

TABLE I
COMPARISON OF SATURATION MAGNETIZATION AND COERCIVITY OF STRONTIUM FERRITE POWDER OBTAINED BY CONVENTIONAL AND SOL-GEL AUTO-COMBUSTION METHODS

Method of production	Saturation magnetization	Coercivity
Conventional	58.20 emu/g	4378 Oe
Sol-gel auto-combustion	66.03 emu/g	5731 Oe

Table I presents the saturation magnetization and coercivity of Sr-ferrite powders obtained by conventional and sol-gel auto-combustion methods. The higher value of saturation magnetization and coercivity in the sample obtained by sol-gel auto-combustion method is clearly observed. According to the SEM photographs presented in Fig. 5, it is deduced that the morphology of the particles has the strong influence on the magnetic properties. A permanent magnet with higher values of saturation magnetization and coercivity can store a higher magnetic energy density.

IV. CONCLUSION

Sr-ferrite Powders were prepared by conventional and sol-gel auto-combustion methods. In conventional method after calcination at temperature of 1300°C, a single phase of Sr-ferrite was formed. Formation temperature of Sr-ferrite powder obtained by sol-gel auto-combustion method decreased to 1000°C. This was probably due to more homogeneous composition and structure of this sample. Saturation magnetization and coercivity of Sr-ferrite powder obtained by sol-gel auto-combustion method were of 66.03 emu/g and of 5731 Oe, respectively, in comparison with 58.20 emu/g and 4378 Oe for sample obtained by conventional method. In the other word, magnetic properties of Sr-ferrite obtained by sol-gel auto-combustion method are better than conventional method.

REFERENCES

[1] A.R. Muxworthy and W. Williams, "Critical superparamagnetic/single-domain grain sizes in interacting magnetite particles implications for magnetosome crystals", *Journal of the Royal Society Interface*, vol. 6, pp. 1207–1212, 2009.

[2] G. Benito, M.P. Morales, J. Requena, V. Raposo, M. Vazquez and J.S. Moya, "Barium hexaferrite monodispersed nanoparticles prepared by the ceramic method", *Journal of Magnetism and Magnetic Materials*, vol. 234, pp. 65–72, 2001.

[3] E. Sada, H. Kumazawa and H.M. Cho, "Synthesis of barium ferrite ultrafine particles by a hydrothermal method", *Industrial & Engineering Chemistry Research*, vol. 30, pp. 1319–1323, 1991.

[4] T.S. Chin, S.L. Hsu and M.C. Deng, "Barium ferrite particulates prepared by a salt-melt method", *Journal of Magnetism and Magnetic Materials*, vol. 120, pp. 64–68, 1993.

[5] S.R. Janasi, M. Emura, F.J.G. Landgraf and D. Rodrigues, "The effects of synthesis variables on the magnetic properties of co-precipitated barium ferrite powders", *Journal of Magnetism and Magnetic Materials*, vol. 238, pp. 168–172, 2002.

[6] M. Ghobeiti Hasab and Z. Shariati, "Magnetic properties of Sr-ferrite nano-powder synthesized by sol-gel auto-combustion method", *International Journal of Chemical, Nuclear, Metallurgical and Materials Engineering,* vol. 8, pp. 965–968, 2014.

[7] M. Ghobeiti Hasab, S.A. Seyyed Ebrahimi and A. Badiei, "The effect of surfactant hydrocarbon tail length on the crystallite size of Sr-hexaferrite powders synthesized by a sol–gel auto-combustion method", *Journal of Magnetism and Magnetic Materials*, vol. 310, pp. 2477–2479, 2007.

[8] M. Ghobeiti Hasab, S.A. Seyyed Ebrahimi and A. Badiei, "Comparison of the effects of cationic, anionic and nonionic surfactants on the properties of Sr-hexaferrite nanopowder synthesized by a sol–gel auto-combustion method", *Journal of Magnetism and Magnetic Materials*, vol. 316, pp. 13–15, 2007.

[9] M. Ghobeiti Hasab, S.A. Seyyed Ebrahimi and A. Badiei, "An investigation on physical properties of strontium hexaferrite nanopowder synthesized by a sol–gel auto-combustion process with addition of cationic surfactant, *Journal of the European Ceramic Society*, vol. 27, pp. 3637–3640, 2007.

[10] M. Ghobeiti Hasab, S.A. Seyyed Ebrahimi and A. Badiei, "Effect of different fuels on the strontium hexaferrite nanopowder synthesized by a surfactant-assisted sol–gel auto-combustion method, *Journal of Non-Crystalline Solids*, vol. 353, pp. 814–816, 2007.

[11] M. Ghobeiti Hasab, S.A. Seyyed Ebrahimi and R. Dehghan, "Surfactant-assisted sol-gel auto-combustion synthesis of Sr-hexaferrite nano-crystalline powder using different fuels and basic agents", *International Journal of Modern Physics*, vol. 5, pp.744–751, 2012.