

See discussions, stats, and author profiles for this publication at: <https://www.researchgate.net/publication/276307321>

Synthesis of Strontium Hexaferrite Using D-Fructose as a Fuel

Article in *Transactions - Indian Ceramic Society* · January 2015

DOI: 10.1080/0371750X.2010.11090837

CITATIONS

3

READS

136

4 authors:



Kanagesan Samikannu

University of Malaya

77 PUBLICATIONS 524 CITATIONS

[SEE PROFILE](#)



Sinnappan Jesurani

Mother Teresa Women's University

42 PUBLICATIONS 275 CITATIONS

[SEE PROFILE](#)



Ramaiyan Velmurugan

Annamalai University

18 PUBLICATIONS 176 CITATIONS

[SEE PROFILE](#)



Kalaivani Thirunavukarasu

SRM Institute of Science and Technology

33 PUBLICATIONS 252 CITATIONS

[SEE PROFILE](#)

Some of the authors of this publication are also working on these related projects:



first one conventional sintering & 2nd microwave sintering calcium copper titanate ,high dielectric material [View project](#)



Oral deliver of ifosfamide [View project](#)

Synthesis of Strontium Hexaferrite Using D-Fructose as a Fuel

S. Kanagesan*, S. Jesurani**, R. Velmurugan and T. Kalaivani

Center for Material Science and Nano Devices
Department of Physics, SRM University
Kattankulathur – 603 203 (Tamil Nadu), India

[MS received April 1, 2010; revised copy received October 13, 2010]

Strontium hexaferrite ($\text{SrFe}_{12}\text{O}_{19}$) powder was synthesized by sol-gel based combustion technique. In this technique, strontium and iron nitrates were used as the source nitrates and D-fructose as the fuel. The phase formation, thermal properties, particle morphology and surface morphology of the sintered pellets were analyzed by X-ray diffraction (XRD), thermogravimetry / differential thermal analysis (TG-DTA) and high-resolution scanning electron microscope (HR-SEM). Crystalline particles obtained by the calcinations of the precursor powder at 900°C , showed the crystalline diffraction pattern with the corresponding major angles, 30.41° , 31.02° , 32.37° , 34.24° , 35.81° , 37.21° , 40.47° and 42.60° respectively. HR-SEM studies have revealed that the structure of calcined particles is a mixed hexagonal and nearly plate like hexagonal, and the surface of the sintered pellets results in hexagonal structure with good magnetic properties.

[Keywords : Sol gel combustion, Hexaferrite, D-fructose, Magnetization]

Introduction

The M-type strontium hexaferrite (SrF) is a hard magnetic material with magnetoplumbite structure. It possesses a fairly large magneto crystalline anisotropy, high Curie temperature and large saturation magnetization. It is used for high density magnetic recording and magneto-optical recording. It is also extensively used as a permanent magnet for high frequency core materials and in the application of microwave devices in millimeter waveband due to its low cost, corrosion resistance, high coercivity and chemical stability.¹⁻³

From the chemistry point of view, it has complex magnetoplumbite type structure and the super exchange interaction of the Fe ions in this complex structure is an interesting subject. The Fe ions are coordinated octahedrally (FeO_6), trigonal bipyramidally (FeO_5) and tetrahedrally (FeO_4) with oxygen ions.⁴ The electronic structure of this ferrite is strongly influenced by the exchange interactions between the irons. The most stable form of the ferrite is a ferrimagnet with the Fe^{3+} ions at the 4f sites having their spin polarization anti-parallel to the rest of the Fe^{3+} ions, in agreement with Gorter's prediction.⁵

The magnetic properties depend very much on the synthesis route, the processing parameters, the reaction temperature and the microstructural grain growth.⁶ Various methods are used to prepare strontium ferrite including conventional solid state mixing,^{7,8} co-precipitation,^{9,10} salt melting,¹¹ glass crystallization,¹² sol-gel¹³ and low temperature combustion synthesis.¹⁴

The chemical route (wet chemical method) is a potential advantage to prepare multicomponent oxide over the

conventional solid state reaction method. It includes better homogeneity, lower processing temperature and better compositional control.¹⁵

The authors have synthesized the barium and strontium hexaferrites using D-fructose as a fuel. The synthesis of barium hexaferrites has been reported elsewhere.¹⁶ In this work, strontium hexaferrite was prepared using D-fructose as the fuel to produce SrF precursor at 130°C . While the prepared precursor was characterized by TG-DTA, the calcined powder and the sintered pellets were characterized by XRD, HR-SEM and vibrating sample magnetometer (VSM). The phase formation, surface morphology and magnetic properties were studied and the results are reported.

Experimental

Powder Synthesis

The powder was prepared by sol-gel based combustion technique with D-fructose as a fuel. Analytical grade $\text{Sr}(\text{NO}_3)_2$, $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ and D-fructose were used as starting materials. Nitrates and fuel ratio was 1:1. The stoichiometric amount of metal nitrates and fuel were taken, dissolved in distilled water and stirred by magnetic stirrer for 2 h to get a solution (sol). The sol was heated at 80°C with continuous stirring till it changed into a sticky liquid (gel) and the product was preheated at 130°C in a hot air oven for two days to get the precursor. The precursor was calcined at three different temperatures. The calcined powder was pelletized into a disc using uniaxial pressure of 4 ton load in a hydraulic press after mixing with small amount of PVA solution. Pellets were sintered in a muffle furnace at three different temperatures of 1000° , 1050° and 1150°C for 3 h in air. The average sintered density was measured by dimensional method prior to sintering by using vernier and electronic balance.

*Corresponding author; e-mail : kanagu1980@gmail.com

**Department of Physics, Jeyaraj Annapackiam College for Women, Periyakulam – 625 601 (Tamil Nadu), India

Measurements

Thermogravimetric analysis of the mixture composed of strontium nitrate, iron nitrate and D-fructose (precursor) was carried out between 28° and 1200°C on NETZSCH STA 409 C/CD in the static air atmosphere at the heating rate of 10°C.min⁻¹. The DTA analysis of the same mixture was also carried out on the same instrument at same condition. X-ray diffraction pattern was taken for crystalline powder using (PANalytical X'pert pro) CuK_α radiation ($\lambda = 0.15406$ nm) at 45 kV and 40 mA in a wide range of 2θ ($10^\circ < 2\theta < 80^\circ$). The crystalline calcined powder was dispersed in acetone and coated on the silicon wafer. The dispersed particles' morphology and surface morphology of the sintered pellet were analyzed using FEI Quanta FEG 200 high resolution scanning electron microscope (HR-SEM). Hysteresis loops were measured at room temperature with the vibrating sample magnetometer (VSM) (Lakeshore 7304 model).

Results and Discussion

The thermogram of the precursor of strontium hexaferrite derived by mixing strontium nitrate, ferric nitrate and D-fructose in air atmosphere with a heating rate of 10°C.min⁻¹ is shown in Fig. 1. The initial weight loss from room temperature to 185°C is due to the removal of water.¹⁷ Two distinct endothermic peaks (200°-400°C) and one exothermic peak around 430°C, correspond to dry fructose that burns drastically after absorbing some energy, and then gives out great quantity of heat. In order to verify this, separate TG was taken for D-fructose and it is shown in Fig. 2. It shows that there was a major weight loss between 200° and 400°C, thus supporting our analysis. The endotherms with the maxima at 712° and 753°C are due to the decomposition of nitrates and the starting formation of hexaferrite. The continuous exothermic process happens above 800°C, which correspond to the formation and transition of crystalline phases. Therefore, D-fructose produces self-heat to promote the reaction and

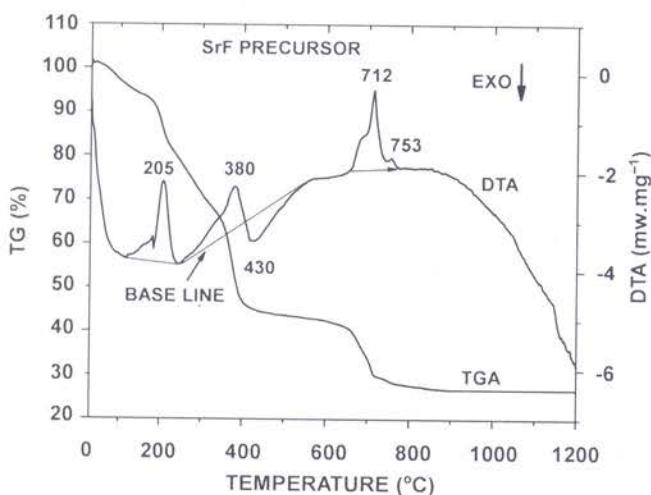


Fig. 1 – TG-DTA curves for the precursor

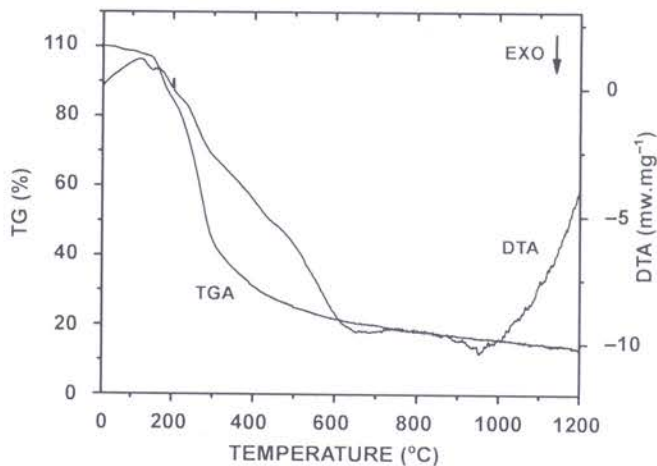


Fig. 2 – TG-DTA curves for D-fructose

to reduce the crystallization temperature of the hexaferrite. The exothermic and endothermic regions in the DTA pattern are consistent with the change regions in the TG pattern. There is no considerable weight loss above 890°C, confirming the formation of the stable strontium ferrite. This analysis, therefore, illustrates the optimum calcination temperature for strontium hexaferrite to be around 900°C.

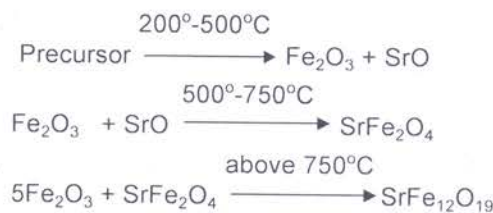


Figure 3 shows the XRD patterns of the powders calcined at 500°, 750° and 900°C for 3 h in air. For the precursor calcined at 500°C, the powders can be described as Fe₂O₃ and SrO, and then the phase of SrFe₂O₄ and hexagonal SrFe₁₂O₁₉ can be detected for the samples

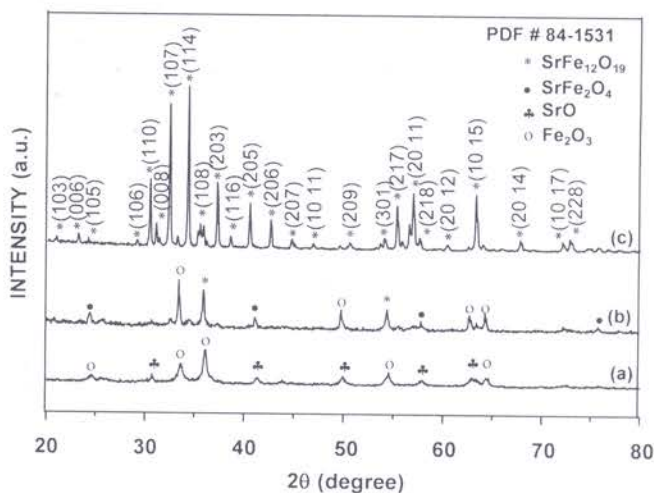


Fig. 3 – XRD patterns of the powders calcined at: (a) 500°C, (b) 750°C and (c) 900°C

calcined at 750°C. The clear diffraction peak of SrFe₁₂O₁₉ could be obtained at 900°C, which coincides with the JCPDS file number: 84-1531. Intermediate Fe₂O₃ plays an important role in the formation of the hexaferrites.

Figure 4a shows the morphology of strontium hexaferrite powder calcined at 900°C for 3 h. All the particles are not well defined in shape and clear boundaries. Morphology of the calcined powder is well crystallized, and the particles are plate like hexagonal / hexagonal in structure. The particles are not uniformly hexagonal but are mixed in nature.² The phase development of sintered disc samples are shown in Figs. 4b, 4c and 4d. The pellet sintered at 1000°C (4b) shows high porosity. Porosity is reduced with increase in grain growth.¹⁸ These grains are contacted to each other in relatively less macro-pores formation. The pellet sintered at 1050°C (4c) exhibits a partially sintered microstructure due to the extensive degree of particle coarsening and sintering at such a high temperature. At 1150°C (4d) it is observed that the microstructure density is 94% of the

theoretical density. It is observed that the particle shape is an ideal hexagonal but the grain size varies. Since the heat treatment has a significant effect on the morphology and the phase constitution of the products, it strongly influences the magnetic properties of the powders.

The magnetic properties of strontium hexaferrite are listed in Table I and the hysteresis curve is shown in Fig. 5. When the precursor is calcined at 900°C for 3 h, Sr-hexaferrite attains hexagonal / plate like structure with a maximum saturation magnetization (*M_s*) of 46.93 emu.g⁻¹ and an intrinsic coercive force (*H_c*) of 6693.69 Oe. When the powder is uniaxially pressed at 4 ton and sintered at 1150°C, it results in hexagonal shape and uniform grains; it also results in optimum magnetic properties with a saturation magnetization of 54.88 emu.g⁻¹ and a maximum intrinsic coercive force of 4734.56 Oe. These clearly indicate that the saturation magnetization increases with increase in the grain size. Similarly the coercivity decreases with the increase in the grain size. Garcia-Cerda *et al.*¹⁹ have reported that the saturation magnetization of

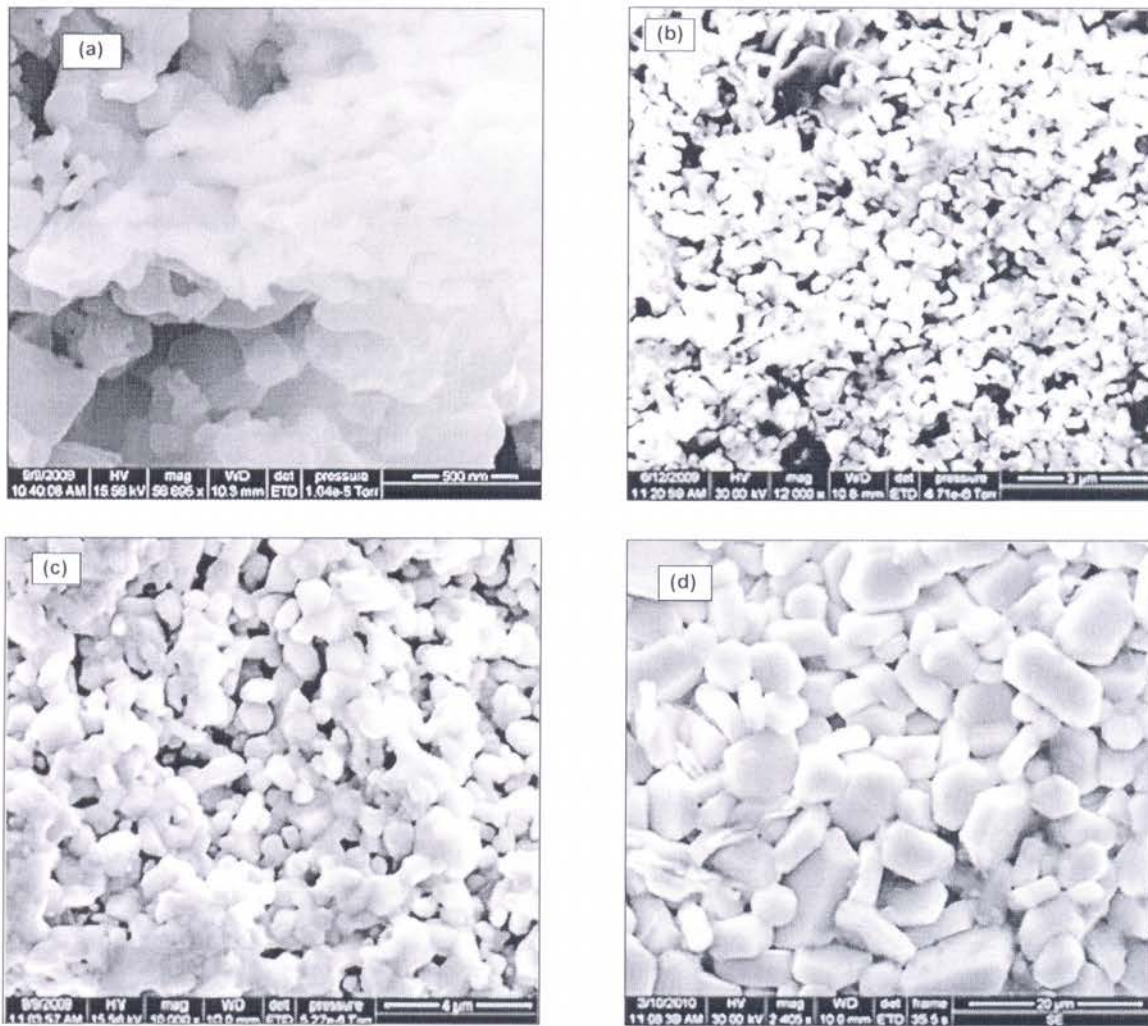


Fig. 4 – HR-SEM image of (a) powder calcined at 900°C, (b) pellet sintered at 1000°C, (c) 1050°C and (d) 1150°C

Table 1 : Effect of temperature on the magnetic properties of strontium hexaferrite

	Saturation magnetization (Ms) (emu.g ⁻¹)	Coercive field (Hc) (Oe)
Powder calcined at 900°C	46.93	6693
Pellet sintered at 1150°C	54.88	4734

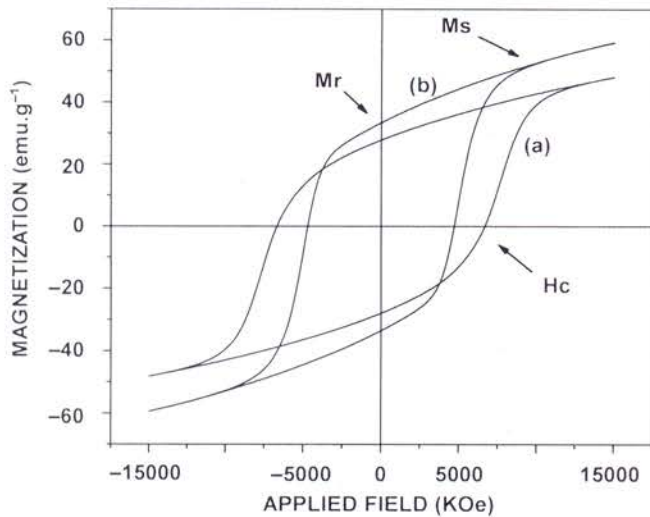


Fig. 5 – Magnetization curve of SrFe₁₂O₁₉: (a) powder calcined at 900°C for 3 h, (b) pellet sintered at 1150°C for 3 h

SrFe₁₂O₁₉ prepared by sol-gel technique increases with the calcination temperature (800°-900°C). The coercivity increases with the calcination temperature. Chen and Chen²⁰ have also reported the reduction in coercivity of strontium hexaferrite prepared by co-precipitation method as we have observed in the present investigation. In the co-precipitation synthesis of strontium hexaferrite by Hessien *et al.*,²¹ the crystallization occurs when the precursor is calcined at 1000°C. However, in the present investigation the crystallization is observed for the sample calcined at 900°C.

Conclusions

In the present investigation, the strontium hexaferrite was synthesized by sol-gel based combustion technique using D-fructose as a fuel, thermal properties were analyzed by TG/DTA, phase formation was confirmed by XRD and particle morphology was investigated by HR-SEM. Single phase strontium hexaferrite was formed at relatively low temperature of about 900°C (XRD). Calcined particles are mixed plate like hexagonal and hexagonal in shape. From the above results, it is concluded that the powder is well crystallized at relatively low temperature using D-fructose

as a fuel. Changes of the crystalline structure are well agreed with the results of TG/DTA and XRD. This paper proves the phase transitions at different calcined temperatures; the formation process of the strontium ferrite and Fe₂O₃ plays an important role in the formation of SrFe₁₂O₁₉. The temperature treatment strongly influences the microstructure of the produced strontium hexaferrite. The formed ultrafine pure SrFe₁₂O₁₉ has good magnetic saturations (46.93-54.88 emu.g⁻¹) and wide intrinsic coercivities (4734-6693 Oe).

Acknowledgements : The authors thank SRM University for providing the facilities available in Nanotechnology Center.

References

1. V. Babu and P. Padaikathan, *J. Magn. Magn. Mater.*, **3**, 1-4 (2001).
2. S. Alamolhoda, S. A. Seyyed Ebrahimi and A. Badiei, *J. Magn. Magn. Mater.*, **303**, 69-72 (2006).
3. H. Kojima, pp. 305-91 in: *Ferromagnetic Materials*, Vol. 3, Ed. E. P. Wohlfarth, North-Holland, Amsterdam, The Netherlands (1982).
4. X. S. Liu, W. Zhong, B. X. Gu and Y. W. Du, *J. Appl. Phys.*, **92**, 1028-32 (2002).
5. C. M. Fang, F. Kools, R. Metselaar, G. de With and R. A. de Groot, *J. Phys. Condens. Mater.*, **15**, 6229-37 (2003).
6. B. B. Ghate, D. P. H. Hasselman and R. M. Spriggs, *Ceram. Int.*, **1**, 105-10 (1975).
7. W. A. Kazmareck, B. Idzikowski and K. H. Muller, *J. Magn. Magn. Mater.*, **921**, 177-81 (1998).
8. F. Haberey and A. Kockel, *IEEE Trans. Magn.*, **12**, 983-85 (1976).
9. K. Haneda, C. Miyakana and H. Kojima, *J. Am. Ceram. Soc.*, **57**, 354-57 (1974).
10. A. Ataie and S. Heshmati-Manesh, *J. Eur. Ceram. Soc.*, **21**, 1951-55 (2001).
11. Z. B. Guo, W.-P. Ding, W. Zhong, J.-R. Zang and Y.-W. Do, *J. Magn. Magn. Mater.*, **175**, 333-36 (1997).
12. H. Sato and T. Umeda, *J. Mater. Trans.*, **34**, 76-81 (1993).
13. K. Oda, T. Yoshio, K. O. Oka and F. Kanamaru, *J. Mater. Sci. Lett.*, **3**, 1007-10 (1984).
14. A. Gruskova, V. Jancarik, J. Slama and M. Usakova, *Acta Phys. Polonica A*, **113**, 557-60 (2008).
15. M. Kakihana, *J. Sol-Gel Sci. Tech.*, **6**, 7-55 (1996).
16. S. Kanagesan, S. Jesurani, R. Velmurugan and C. Kumar, *J. Manu. Eng.*, **5**, 133-36 (2010).
17. X. Tang, B. Y. Zhao and K. A. Hu, *J. Mater. Sci.*, **41**, 3867-71 (2006).
18. A. Ataie, S. Heshmati-Manesh and H. Kazempour, *J. Mater. Sci.*, **37**, 2125-28 (2002).
19. L. A. Garcia-Cerda, O. S. Rodriguez-Fernandez and P. J. Resendiz-Hernandez, *J. Alloys Comp.*, **369**, 182-84 (2004).
20. D. H. Chen and Y. Y. Chen, *Mater. Res. Bull.*, **37**, 801-10 (2002).
21. M. M. Hessien, M. M. Rashad and K. El-Barawy, *J. Magn. Magn. Mater.*, **320**, 336-43 (2008).