# Magnetic Properties of Sr-Ferrite Nano-Powder Synthesized by Sol-Gel Auto-Combustion Method

M. Ghobeiti-Hasab, Z. Shariati

*Abstract*—In this paper, strontium ferrite ($SrO.6Fe_2O_3$) was synthesized by the sol-gel auto-combustion process. The thermal behavior of powder obtained from self-propagating combustion of initial gel was evaluated by simultaneous differential thermal analysis (DTA) and thermo gravimetric (TG), from room temperature to 1200°C. The as-burnt powder was calcined at various temperatures from 700-900°C to achieve the single-phase Sr-ferrite. Phase composition, morphology and magnetic properties were investigated using X-ray diffraction (XRD), transmission electron microscopy (TEM) and vibrating sample magnetometry (VSM) techniques. Results showed that the single-phase and nano-sized hexagonal strontium ferrite particles were formed at calcination temperature of 800°C with crystallite size of 27 nm and coercivity of 6238 Oe.

*Keywords*—Hard magnet, Sr-ferrite, Sol-gel auto-combustion, Nano-powder.

## I. INTRODUCTION

FERRITES are iron containing complex oxides with technically interesting magnetic properties. Soft ferrites are the magnetic materials that do not retain their magnetism after being magnetized. They have narrow hysteresis loops possessing high saturation magnetization but very small coercivity. They are extensively used in the cores of transformers where they must respond to a rapidly oscillating field. Hard ferrites are permanent magnets because they can retain their magnetism after being magnetized. For hard ferrites the hysteresis loop is broad having high coercivity. These materials are commonly used as permanent magnets. Sr-ferrite is a hard ferrite with wide applications in memory storage and magnetic recording media [1].

The magnetic properties such as saturation magnetization (Ms), remanence (Mr) and coercivity (Hc) are determined from the hysteresis loop (Fig. 1). The shape and width of the hysteresis loop of a ferrite depend not only on the chemical composition but also on the particle size. Sr-ferrite as a hard magnet is required to have high saturation magnetization and coercivity for its use in magnetic recording media. Hard magnetic materials are characterized by domain walls which move hardly when a magnetic field is applied. The ultra-fine powders with single-domain behavior have high saturation magnetization and coercivity [2].

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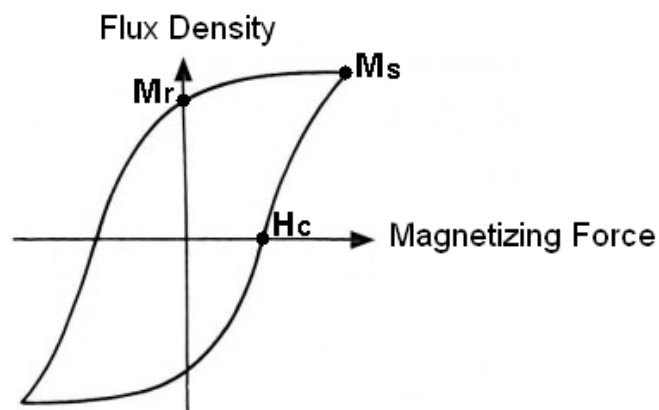
Fig. 1 Typical hysteresis loop showing saturation magnetization (Ms), remanence (Mr) and coercivity (Hc)

Conventional solid state reactions for preparing Sr-ferrite powder require a high calcining temperature (1200-1300°C) of milled mixtures of ferric oxide with strontium carbonate. Furthermore, the milling process can introduce impurities into the material. Recently, the sol-gel auto-combustion method has been considered because of its utilization of the heat released from the self-propagating exothermic reactions of the chelating agents with the nitrate oxidant. This method has been successful in producing homogeneous nano-sized ferrite crystallites at much lower calcination temperatures. Low temperatures may prevent grain growth and keep high coercivity [3]. In this work, Sr-ferrite nano-powder has been synthesized by the sol-gel auto-combustion method and the influence of calcination temperature on its phase composition and magnetic properties has been studied.

## II. EXPERIMENTAL

Appropriate amounts of $Fe(NO_3)_3.9H_2O$ and $Sr(NO_3)_2$ (molar ratio of Fe/Sr:10) were dissolved in a minor amount of distilled water and then citric acid (molar ratio of citric acid/metallic nitrates:1), trimethylamine as pH adjusting agent (pH:7) and n-decyl trimethyl ammonium bromide surfactant (molar ratio of surfactant/strontium:0.4) were added. 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 (Fig. 2). As-burnt powder was calcined at 700-900°C in a resistance furnace with air atmosphere for 1 hour to form single-phase Sr-ferrite.

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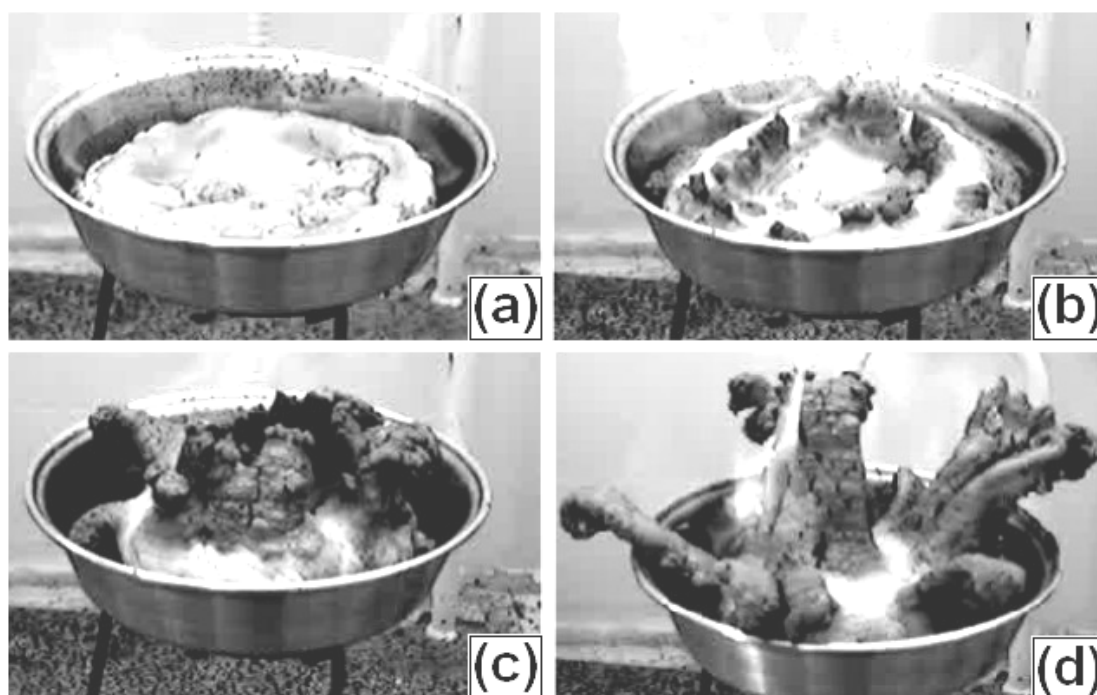International Journal of Materials and Metallurgical Engineering
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Fig. 2 (a) Swelling up the gel (b) starting the self-propagating ignition (c) progressing the ignition (d) finishing the process

The thermal behavior of as-burnt powder was evaluated by simultaneous differential thermal analysis (DTA) and thermo gravimetric (TG). X-ray diffraction (XRD, Cu-Kα radiation) was used for phase identification of the as-burnt and calcined powder. The average crystallite size of Sr-ferrite powders obtained by calcination of as-burnt powder at different temperatures were calculated by X-ray line broadening technique for profile of the main peak using the Scherrer's equation [4]:

$$D = 0.9\lambda / \beta \cos\theta \qquad (1)$$

In this equation, D is the average crystallite size in nm, λ is the radiation wavelength (0.154 nm for Cu Kα), β (in radian) is the width of the main peak at the half of its intensity, and θ (in degree) is the peak position. The morphology of Sr-ferrite powder was observed by transmission electron microscopy (TEM). Vibrating sample magnetometry (VSM) was used to characterize the magnetic properties of the powders, such as coercivity and saturation magnetization at room temperature.

## III. RESULTS AND DISCUSSION

The DTA-TG curves of as-burnt powder in air atmosphere are shown in Fig. 3. The first exothermic peak at about 400°C is attributed to decomposition of the residual organic matters and the second exothermic peak at about 800°C belongs to the crystallization of Sr-ferrite phase. The TG curve also shows two distinct stages of weight loss at mentioned temperatures, which are due to the evaporation of residual organic matters

and decomposition of $SrCO_3$ to SrO and $CO_2$ and the formation of Sr-ferrite, respectively. After 800°C no reaction could be detected from the curves, which means the completion of the Sr-ferrite formation.

The XRD patterns of as-burnt powder and the powders calcined at 700, 750, 800, 850 and 900°C are shown in Fig. 4. It is seen that the as-burnt powder consists of $\gamma$-$Fe_2O_3$, $\alpha$-$Fe_2O_3$ and $SrCO_3$ phases. After calcination it seems that $\gamma$-$Fe_2O_3$ and $SrCO_3$ are transformed to $\alpha$-$Fe_2O_3$ and SrO, respectively, and then $SrO.6Fe_2O_3$ phase is formed by reaction of SrO with $\alpha$-$Fe_2O_3$. At 800°C, only the Sr-ferrite phase could be detected.
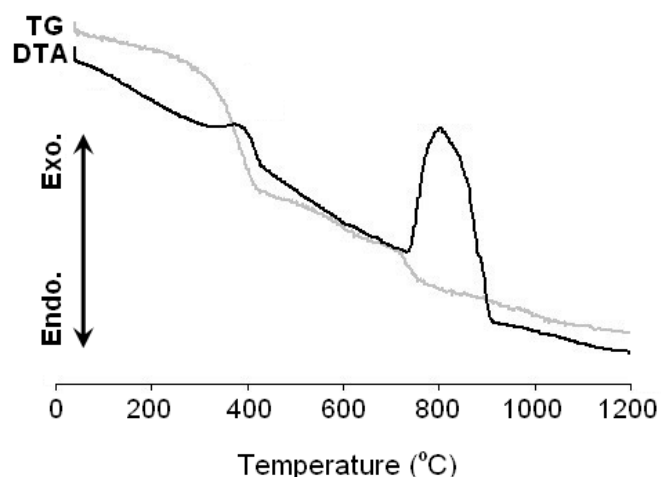


Fig. 3 DTA/TG curves for the as-burnt powder

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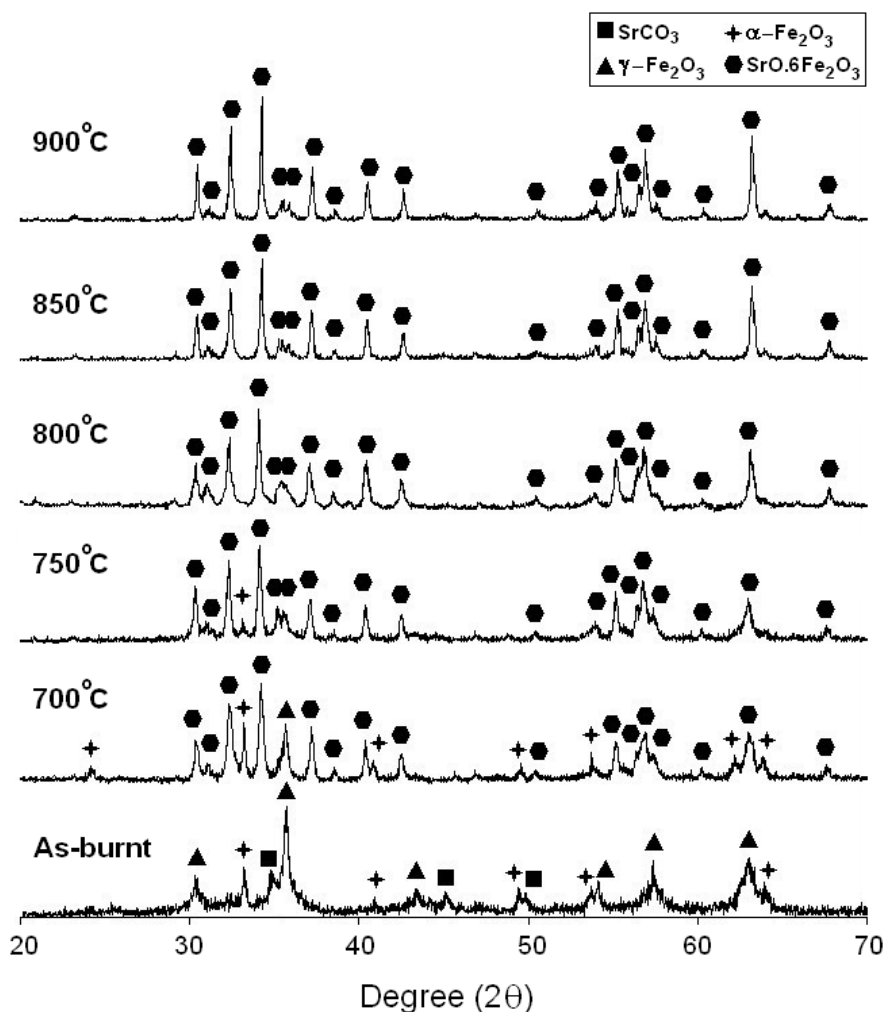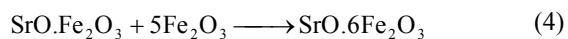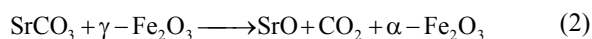Vol:8, No:10, 2014

Fig. 4 XRD patterns of as-burnt powder and powders calcined at various temperatures

Equations (2)-(4) show the reactions which take place after calcination of as-burnt powder [5].

$$SrCO_3 + \gamma - Fe_2O_3 \longrightarrow SrO + CO_2 + \alpha - Fe_2O_3 \qquad (2)$$

$$SrO + \alpha - Fe_2O_3 \longrightarrow SrO.Fe_2O_3 \qquad (3)$$

$$SrO.Fe_2O_3 + 5Fe_2O_3 \longrightarrow SrO.6Fe_2O_3 \qquad (4)$$

According to the XRD patterns presented in Fig. 4, the peaks width decreases with the increase in calcination temperature, demonstrating the increase in crystallite size.

Fig. 5 shows the average crystallite size of the Sr-ferrite powders synthesized at various calcination temperatures. These results reveal that the crystallite growth rate increases with increasing the calcination temperature. At 800°C, average crystallite size was estimated 27 nm.

Fig. 6 shows the saturation magnetization and coercivity of the Sr-ferrite nano-powder as a function of calcination temperature. The saturation magnetization demonstrates an initial decrease when the calcination temperature is increased from 700 to 750°C, followed by a steady increase as the calcination temperature increases in the range of 750-900°C.

The higher value of saturation magnetization observed for the powder calcined at 700°C in comparison with 750°C is due to the existence of the $\gamma$-Fe$_2$O$_3$ phase [6]. Any further increase in the calcination temperature causes to increasing in the saturation magnetization due to the increase of crystallite size and growth of magnetic domains. The maximum saturation magnetization value of 63.44 emu/g was obtained for the powder calcined at 900°C.

It is also shown in Fig. 6 that as the calcination temperature is increased from 700 to 800°C, an increase in coercivity is observed, but when the temperature is increased beyond 800°C, a decrease in coercivity is noticed. The increase in coercivity in the range of 700-800°C is due to the development of Sr-ferrite phase as temperature increases up to 800°C. The decrease in coercivity for temperatures above 800°C is attributed to the increase in crystallite size. Maximum coercivity value of 6238 Oe was obtained for the powder calcined at 800°C.

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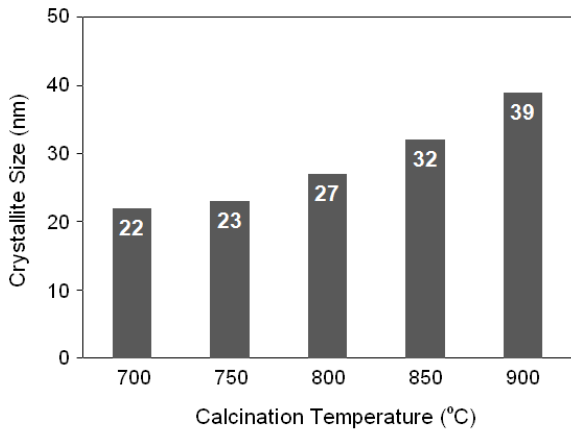Vol:8, No:10, 2014

Fig. 5 Average crystallite size of the Sr-ferrite powder synthesized at various calcination temperatures
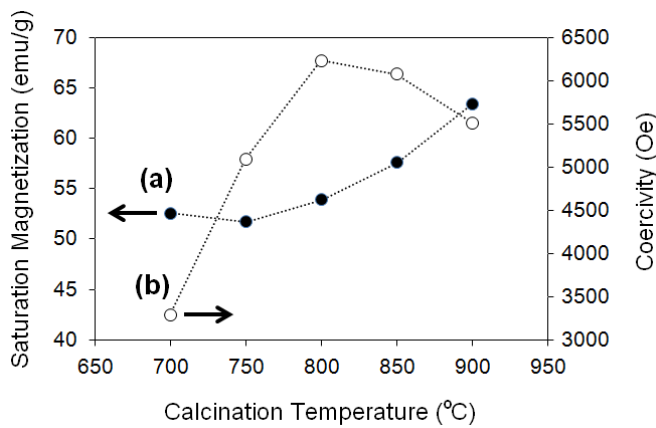


Fig. 6 (a) Saturation magnetization (b) coercivity of Sr-ferrite nano-powder as a function of calcination temperature
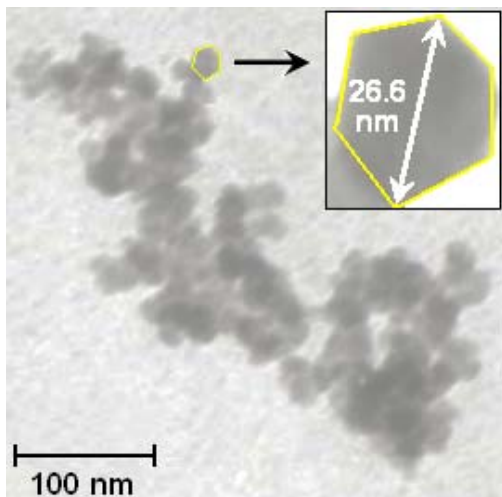


Fig. 7 TEM photograph of Sr-ferrite powder prepared at calcination temperature of 800°C

In Fig. 7, TEM photograph of the Sr-ferrite powder calcined at 800°C for 1 hour is shown. As it is seen, the size of the hexagonal-shape crystallites is 25-30 nm, which is much smaller than the single-domain crystal particle size of the Sr-ferrite (270 nm) [7].

## IV. CONCLUSION

Sr-ferrite $(SrO.6Fe_2O_3)$ nano-powder was successfully synthesized by a sol-gel auto-combustion method in the presence of n-decyltrimethylammonium bromide cationic surfactant. At the calcination temperatures below 800°C the intermediate phases like $\gamma$-$Fe_2O_3$ and $\alpha$-$Fe_2O_3$ are still detected. Single-phase of $SrO.6Fe_2O_3$ formed after the as-burnt powder was calcined at 800°C for 1 hour. In this sample, the average size of hexagonal-like crystallites was about 27 nm. The results indicated that calcination temperature had significant effect on the magnetic properties. A maximum coercivity value of 6238 Oe was obtained for the sample calcined at 800°C and high saturation magnetization value of 63.44 emu/g was observed in the sample calcined at 900°C.

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