

The Influence of Reaction Parameters on Magnetic Properties of Synthesized Strontium Ferrite

M. Bahgat, F. M. Awan, H. A. Hanafy

Abstract—The conventional ceramic route was utilized to prepare a hard magnetic powder (M-type strontium ferrite, $\text{SrFe}_{12}\text{O}_{19}$). The stoichiometric mixture of iron oxide and strontium carbonate were calcined at 1000°C and then fired at various temperatures. The influence of various reaction parameters such as mixing ratio, calcination temperature, firing temperature and firing time on the magnetic behaviors of the synthesized magnetic powder were investigated. The magnetic properties including Coercivity (Hc), Magnetic saturation (Ms), and Magnetic remnance (Mr) were measured by vibrating sample magnetometer. Morphologically the produced magnetic powder has a dense hexagonal grain shape structure.

Keywords—Hard magnetic materials, ceramic route, strontium ferrite, magnetic properties.

I. INTRODUCTION AND BACKGROUND

THE magnetic materials market is forecast to witness a significant increase in demand, driven by the evolution of new end-use applications, and consistent demand from existing end-use markets, including industrial and consumer electronics, data storage, military and aerospace, medical, power generation, and telecommunications. New technological innovations, improved electrical, magnetic, and mechanical performance of soft and hard magnetic materials, and readily available improved materials represent the major factors influencing demand for magnetic materials [1], [2].

During the twentieth century, several permanent magnet materials were discovered. Techniques to effectively manufacture these magnets have been established. Device designs using such magnets in various active and passive applications have been successfully exploited. The energy product, which is a key figure of merit of permanent magnets, has been enhanced, starting from ≈ 1 MGOe for steels discovered during the early part of the century, increasing to ≈ 3 MGOe for hexagonal ferrites [3].

Magnetic materials are widely used as components in various applications of industrial and medical equipment. A very well-established class of magnetic materials is made from magnetic ceramic materials, or ferrites [4]-[6], which are essential in devices for storing energy in a static magnetic field. Major applications involve the conversion of mechanical

to electrical energy. The applications of magnetic materials in information technology have been growing continuously [7].

The ferrite materials may be classified into three different classes; spinel ferrites, garnet ferrites and hexagonal ferrites [8]. The magnetic spinel has the general formula of MFe_2O_4 , where M is the divalent metal ion, usually Ni, Co, Mn, or Zn.

During the last few years spinel ferrites have drawn a major attention because of their technological importance in magnetic recording, magnetic fluid and catalyst. The garnet ferrites are the basis of materials for many high-technology devices for magneto-optic, microwave and memory applications [6]. The ferrites used for permanent-magnet purposes are the hexagonal ferrites, also called hard ferrites or M-type ferrites. For many applications a permanent magnet is the best choice because it provides a constant field without the continuous expenditure of electric power and without generation of heat. In 1952 this class of ferrite having permanent magnetic properties was discovered. These were the so-called hexagonal ferrites with formula $\text{M}(\text{Fe}_{12}\text{O}_{19})$ where M is usually barium Ba, Strontium Sr or Lead Pb [9]-[11].

Strontium hexaferrite ($\text{SrFe}_{12}\text{O}_{19}$, Sr-ferrite) is one of the well-known materials for permanent magnets. The magnetic properties of sintered Sr-ferrite depend on its microstructure (size and shape of the particles). In order to fabricate a sintered magnet with superior properties it is necessary to inhibit the grain growth during sintering and also to keep the microstructure homogeneous [12], [13].

It is a hard magnet with high coercivity, which originates from high magnetocrystalline anisotropy with single easy magnetization axis [14]. It has been recognized that it can be used as permanent magnets, recording media, telecommunication, and as components in microwave, higher-frequency, and magneto-optical devices [15]-[18]. It is also used as a dielectric or magnetic filler in the electromagnetic attenuation materials (EAM). EAM are used to minimize the electromagnetic interference (EMI), a specific type of environmental pollution. EMI has a serious problem due to huge growth in the utilization of electrical and electronic devices in the industrial, commercial and military applications [19]. Sr-ferrite powders are ideal fillers for the development of EAM due to their low cost, high stability, large electrical resistivity and high microwave magnetic loss.

Various processing techniques have been employed for fabrication of the strontium ferrites, including the traditional sol-gel process [20], [21], the solid-state method [22], the salt melting method [23], ball milling [24], [25], self-propagating high temperature synthesis [26], and the chemical co-

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precipitation method [27]. However, the simple ceramic process has been extensively used in industrial manufacturing [28].

II. EXPERIMENTAL WORK

A. Raw Material

Strontium carbonate SrCO_3 (99% purity, Chemical) and steelmaking by-product Iron oxide were used in the experiments. The Iron oxide samples are characterized with X-ray diffraction analysis (XRD), X-ray fluorescence (XRF) and scanning electron microscope (SEM). It was found that iron oxide (Fe_2O_3) is about 93% purity. For SEM the sample to be examined was fixed directly on adhesive tape and then examined under scanning microscope. The SEM photos for iron oxide samples are shown in Fig. 1. It was observed that grain coalescence with very low micropores and many macropores took place in a dense structure.

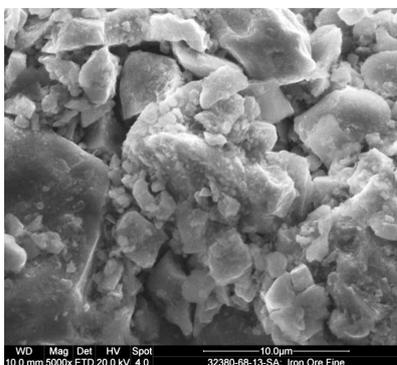


Fig. 1 SEM micrographs of iron oxide

B. Experimental System

In this study, M-type strontium ferrites were prepared by the conventional ceramic method. The initial materials, SrCO_3 and iron oxide were mixed together in the composition of $\text{SrFe}_{12}\text{O}_{19}$ in different Sr/Fe ratios 1/12, 1/11 and 1/10. The mixtures of these raw materials were dry-milled for 6 h. The blended powder was pre-sintered (calcination) at 1000°C for 4, 8 and 12 hrs, then cooled down to room temperature. These pre-sintered samples were re-milled in a dry atmosphere for 1 hr and then fired at 1100, 1200 and 1300°C for 2, 4 and 6 hrs in air atmosphere, and cooled down to room temperature.

The produced magnetic powder was characterized with X-ray diffraction analysis (XRD), X-ray fluorescence (XRF), and scanning electron microscope (SEM). Hc, Ms, and Mr were measured by VSM with applied magnetic field 15 KOe at 300 K.

III. RESULTS AND DISCUSSION

The XRD pattern for fired samples is shown in Fig. 2. It was observed that after calcination at 1000°C for 12 hrs, and then firing at 1100°C for 2 hrs, strontium carbonate decomposed to SrO and Strontium ferrite phase is completely formed.

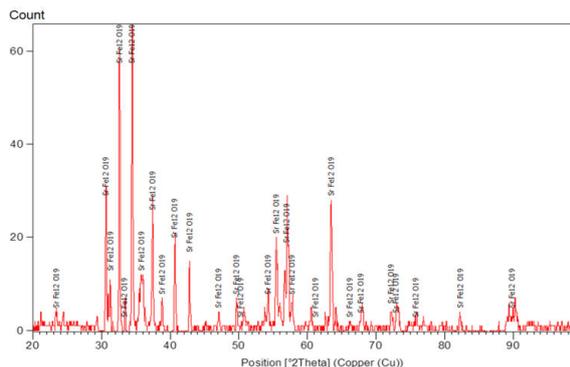


Fig. 2 XRD pattern for iron oxide/strontium oxide mixture after calcination at 1000°C for 12 hrs and then firing at 1100°C for 2 hrs

The magnetic properties were measured by vibrating sample magnetometer (VSM) for all samples. The influence of various parameters such as Sr/Fe mixing stoichiometry, calcination time, firing time and firing temperature on phase formation, microstructure and magnetic properties of the prepared Strontium ferrite were investigated.

A. Influence of Mixing Stoichiometry

Strontium carbonate and iron oxide were mixed together in the composition of $\text{SrFe}_{12}\text{O}_{19}$ in different Sr/Fe ratios 1/12, 1/11 and 1/10 and then calcined at 1000°C for 12 hrs and then fired at 1200°C for 4 hrs. As shown in the XRD pattern (Fig. 3) the Sr-ferrite phase is successfully formed completely in the three samples. There are no peaks for unreacted Fe_2O_3 or SrO. The grain size (1-3 μm) and grain shape of the synthesized strontium ferrite came very similar with well uniformed and homogeneous hexagonal crystalline shape as shown in Fig. 4. Coercivity (Hc), Magnetic saturation (Ms), and Magnetic remnance (Mr) were measured for all samples at 300 K. As shown in Fig. 5 the Coercivity, magnetic saturation and magnetic remnant increased gradually with increasing the mixing ratio up to the stoichiometric value (Sr/Fe = 1/12).

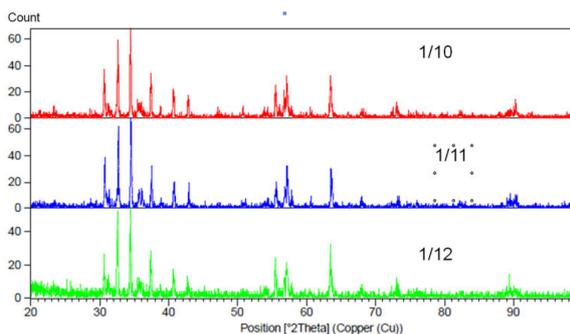


Fig. 3 XRD pattern for iron oxide and strontium oxide mixed together in different Sr/Fe ratios 1/12, 1/11 and 1/10 and then calcined at 1000°C for 12 hrs and then fired at 1200°C for 4 hrs

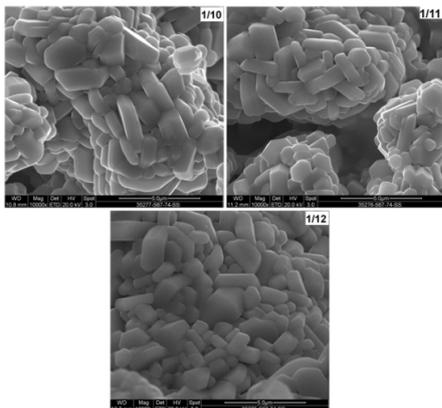


Fig. 4 SEM micrographs for iron oxide and strontium oxide mixed together in different Sr/Fe ratios 1/12, 1/11 and 1/10 and then calcined at 1000 °C for 12 hrs and then fired at 1200 °C for 4 hrs

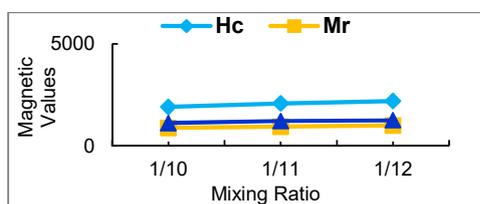


Fig. 5 The magnetic values Coercivity (Hc), Magnetic saturation (Ms), and Magnetic remnance (Mr) of the synthesized magnetic material at different Sr/Fe ratios

B. Influence of Calcination Conditions

Strontium carbonate and iron oxide were mixed together in Sr/Fe ratio 1/12 and then calcined at 1000 °C for 4, 8 and 12 hrs and then fired at 1200 °C for 4 hrs. As shown in the XRD pattern (Fig. 6) it can be seen that the Sr-ferrite phase is successfully formed completely in the three samples. The grain size (1-3 μ) and grain shape of the synthesized strontium ferrite came very similar with well uniformed and homogeneous hexagonal crystalline shape as shown in Fig. 7. Regarding the measured magnetic values, as shown in Fig. 8 the Coercivity, magnetic saturation and magnetic remnant increased gradually with increasing the calcination time from 4 up to 12 hrs.

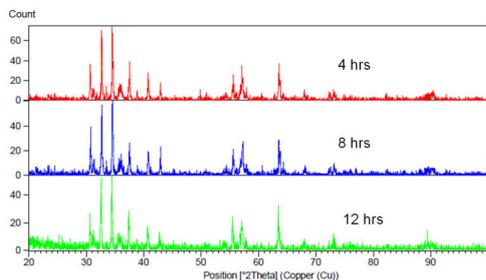


Fig. 6 XRD pattern for iron oxide and strontium oxide mixture after calcination at 1000 °C for 4, 8 and 12 hrs and then firing at 1200 °C for 4 hrs

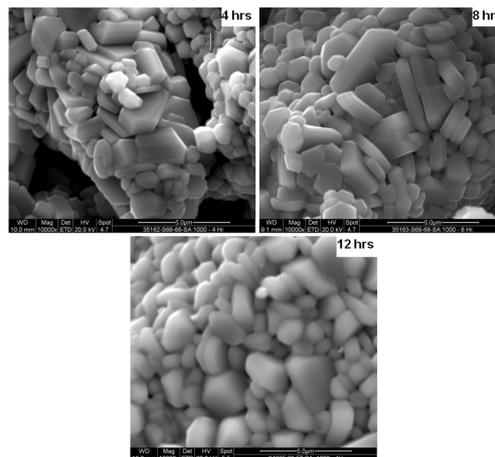


Fig. 7 SEM micrographs for iron oxide and strontium oxide mixture after calcination at 1000 °C for 4, 8 and 12 hrs and then firing at 1200 °C for 4 hrs

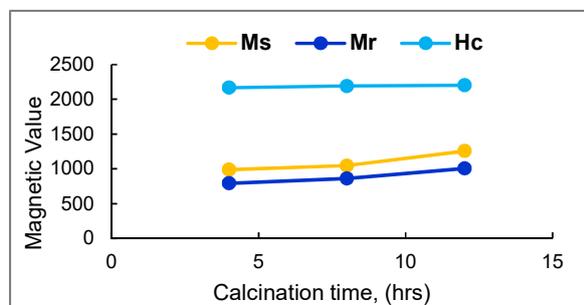


Fig. 8 The magnetic values Coercivity (Hc), Magnetic saturation (Ms), and Magnetic remnance (Mr) of the synthesized magnetic material at different calcination time

C. Influence of Firing Time

Strontium carbonate and iron oxide were mixed together in Sr/Fe ratio 1/12 and then calcined at 1000 °C for 12 hrs and then fired at 1100-1300 °C for 2, 4 and 6 hrs. As shown in the XRD pattern (Fig. 9) for samples fired at 1200 °C, it can be seen that at different reaction times the Sr-ferrite phase is successfully formed completely in the various samples. Also there are no peaks for unreacted Fe₂O₃ or SrO. The morphological examination for these samples is shown in Fig. 10. There is no clear change in the grain size with increasing the firing time. Grain shape of the synthesized strontium ferrite came very similar with well uniformed and homogeneous hexagonal crystalline shape. For measured Hc, Ms, and Mr as shown in Fig. 11 the Coercivity, magnetic remnance and magnetic saturation increased with increasing the firing time up to 4 hrs and then decreased again with longer firing time. This is might be owing to the formation of multi-domain and the easy movement of the domain walls result in domains misalignment that decrease the hard magnetic character.

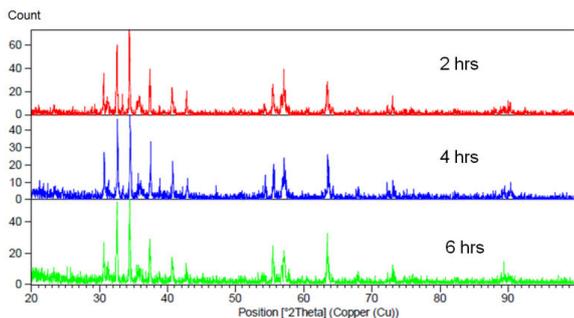


Fig. 9 XRD pattern for iron oxide and strontium oxide mixture after calcination at 1000 °C for 12 hrs and then firing at 1200 °C for 2, 4 and 6 hrs

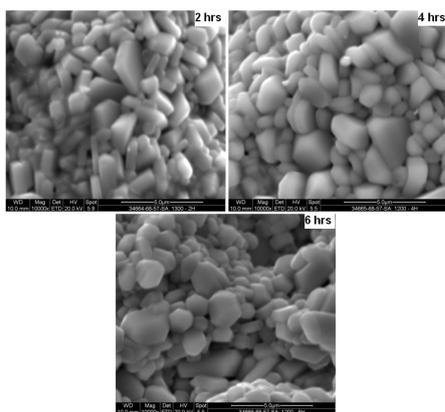


Fig. 10 SEM micrographs for iron oxide and strontium oxide mixture after calcination at 1000 °C for 12 hrs and then firing at 1200 °C for 2, 4 and 6 hrs

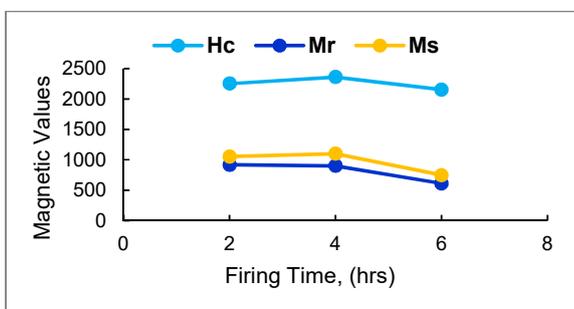


Fig. 11 The magnetic values Coercivity (Hc), Magnetic saturation (Ms), and Magnetic remnance (Mr) of the synthesized magnetic material at different firing time

D. Influence of Firing Temperature

Strontium carbonate and iron oxide were mixed together in Sr/Fe ratio 1/12 and then calcined at 1000 °C for 12 hrs and then fired at 1100-1300 °C for 2, 4 and 6 hrs. As shown in the XRD pattern (Fig. 12) for samples fired at 1100, 1200 and 1300 °C for 2 hrs, it can be seen that at different reaction temperatures the Sr-ferrite phase is successfully formed completely in the various samples. The morphological examination for these samples is shown in Fig. 13. The grain size is increased drastically with increasing the firing

temperature from 1100 to 1300 °C. Grain shape of the synthesized strontium ferrite became well crystalline hexagonal structure with increasing the firing temperature. For magnetic characterizations, as shown in Fig. 14 the Coercivity, magnetic remnance and magnetic saturation increased with increasing the firing temperature up to 1200 °C and then decreased again with higher firing temperature. This is might be owing to the formation of multi-domain and the easy movement of the domain walls result in domains misalignment that decrease the hard magnetic character.

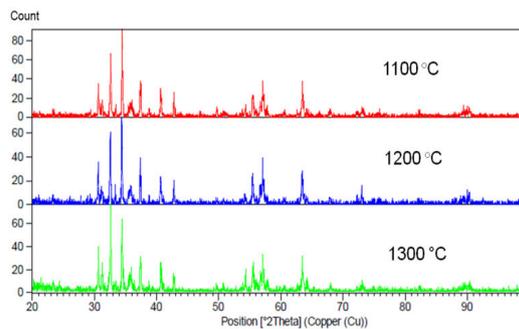


Fig. 12 XRD pattern for iron oxide and strontium oxide mixture after calcination at 1000 °C for 12 hrs and then firing at 1100, 1200 and 1300 °C for 2 hrs

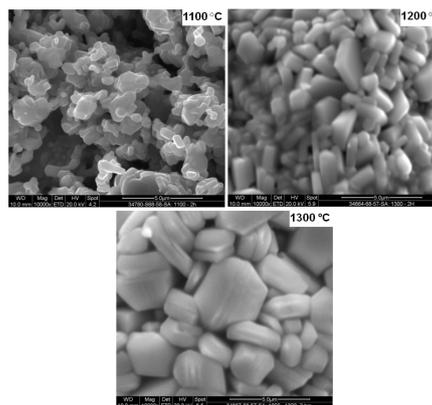


Fig. 13 SEM micrographs for iron oxide and strontium oxide mixture after calcination at 1000 °C for 12 hrs and then firing at 1100, 1200 and 1300 °C for 2 hrs

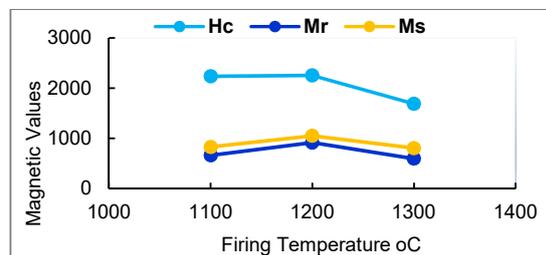


Fig. 14 The magnetic values Coercivity (Hc), Magnetic saturation (Ms), and Magnetic remnance (Mr) of the synthesized magnetic material at different firing temperatures

IV. CONCLUSIONS

- 1) Hard magnetic material in powder shape (Sr-ferrite) was successfully synthesized through ceramic route using steelmaking by-product
- 2) The measured magnetic values (H_c , M_s and M_r) enhanced gradually with;
 - Increasing the mixing ratio (Sr/Fe) up to stoichiometric ratio
 - Increasing the calcination time up to 12 hrs
 - increasing the firing time up to 4 hrs
 - Increasing the firing temperature up to 1200 °C

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