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ABSTRACT

This study examines the sustainable production of strontium hexaferrite by recycling mill scale, a by-product of steel manufacturing, as a source of iron. Strontium, designated as a critical raw material by the European Union, is essential for high-demand magnetic applications. In this study, various additives, including Al_2O_3, Cr_2O_3, Co_3O_4, and La_2O_3, were incorporated during the synthesis process to enhance the magnetic properties of the resulting strontium hexaferrite. Additives were included in the structure using the mechanochemical method. The results show that the incorporation of these additives has a significant influence on the coercivity, remanence, and overall magnetic anisotropy of $SrFe_{12}O_{19}$.

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INTRODUCTION

Permanent magnets are essential in modern technologies, including computers, motors, cars, and generators. Ferrite magnets, despite not possessing the high magnetic properties of rare-earth element-containing magnets like NdFeB and SmCo, are significant in industry.[1,2] Strontium, identified by the European Union as a "critical raw material,"[3] is utilized in the production of strontium hexaferrite, which combines strong magnetic properties with high thermal stability.[4,5]

Sr-ferrite magnet production began in the early 20th century and has evolved. Recycling has become increasingly important due to the growing demand and decreasing availability of natural resources.[6] In recent years, several studies have investigated the recycling of strontium ferrite.[7–9] For example, Ghorbanighoshchi *et al.* investigated recycling strategies to recover rare earths from magnet scraps,[8] while Berja *et al.* demonstrated that recycled $SrFe_{12}O_{19}$ powders, processed through wet milling and sintering, can meet industrial performance standards.[9]

Mill scale is a flaky, dense layer formed on the surface of steel during hot rolling or continuous casting, typically consisting of a mixture of various iron oxides.[10] One promising direction is the use of mill scale, a by-product of steel production that accounts for roughly 4% of its output and is rich in iron oxides.[11,12] The following reaction represents the synthesis of strontium hexaferrite.

$$SrCO_3 + 6Fe_2O_3 \rightarrow SrFe_{12}O_{19} + CO_2 \quad (1)$$

The study aims to enhance the magnetic properties of recycled ferrites and offer an environmentally friendly alternative to conventional production methods. It utilizes mill scale, a by-product of steel production with a 72% Fe content, to produce strontium hexaferrite, thereby reducing reliance on primary raw materials and promoting a circular economy. Another advantage of strontium hexaferrite is the ability to tune its properties during synthesis by adding small amounts of other oxides. Additives have been shown to improve magnetic performance while maintaining decent physical properties.[13,14] The study also aims to optimize synthesis

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conditions and raw material ratios, benefiting industries like electric motors and advanced electronics that rely on efficient magnetic materials.[4]

EXPERIMENTAL METHOD

In this study, strontium hexaferrite ($SrFe_{12}O_{19}$) production utilized $SrCO_3$ (Strontianite, hereafter referred to as SC) and mill scale (hereafter referred to as MS from now on), a by-product of steel production. $SrCO_3$ serves as the source of strontium, while mill scale is evaluated as a source of iron obtained from local steel producers. Various oxide additives were introduced to improve strontium hexaferrite's magnetic and morphological properties. The oxide additives included Chromium oxide (Cr_2O_3), Lanthanum oxide (La_2O_3), Cobalt oxide (Co_3O_4), and Alumina (Al_2O_3).

Initially, the mill scale (MS) was prepared using a jaw crusher for size reduction. If the powder remains coarse, manual crushing was then used to reduce the particle size to under 300 μm. The crushed material was then placed in mill jars filled with hexane and stainless-steel balls with various diameters between 4.40 and 14.80 mm. The mixture was milled for 12 h at a vibration frequency of 25 Hz using a high-energy vibrating ball mill (Unal Makina) to reduce the particle size to below 10 μm effectively. Subsequently, $SrCO_3$ was added in a weight ratio of 1:6 to the mill scale. After adding $SrCO_3$, the mixture was further pulverized in the ball mill for an additional 16 hours to ensure homogeneity in size. This homogenized mixture was then subjected to annealing at 1190 °C for 6 h to form the $SrFe_{12}O_{19}$ structure.[11] After annealing, the resulting powder was pressed under 5 tons of pressure in a mold and sintered at 1190 °C for 5 min, followed by 2 h at 1000 °C.[4,5]

For further research of improving the properties, various oxide additives were introduced to the $SrFe_{12}O_{19}$ powder to enhance its magnetic and structural properties. These oxide additives (Cr_2O_3 at 1.47%, La_2O_3 at 3.48%, Co_3O_4 at 1.47%, Al_2O_3 7.64% by total weight) were included in the mixture at the same time with $SrCO_3$, and the mixture was homogenized by milling for 16 h. Following the annealing process, compaction and sintering were performed as before. For magnetization measurements, the doped magnets were subjected to a magnetic field of 10^5 Oersted (Oe).

RESULTS AND DISCUSSION

The XRD analysis of the raw mill scale, presented in Fig. 1(a), reveals that the material primarily comprises the maghemite (γ-Fe_2O_3) phase along with a significant amount of wuestite (FeO). Initially, the presence of wuestite was carefully evaluated alongside potential trace impurities common in industrial by-products; however, the diffraction peaks were conclusively indexed to the FeO phase. As mentioned earlier, these oxides are unsuitable for strontium hexaferrite production [Eq. (1)]. The intended hematite phase forms after annealing the mill scale for 6 h at 1190 °C [Fig. 1(b)].

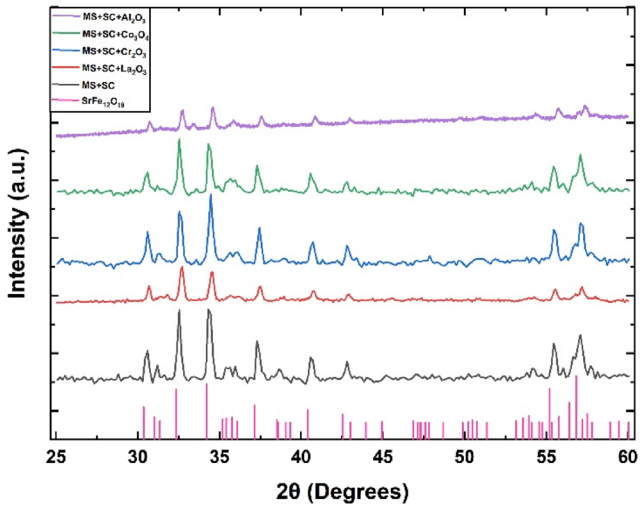


FIG. 2. The XRD pattern of the $SrFe_{12}O_{19}$ powders synthesized from the mill scale with different additives (MS and SC; mill scale and strontionate, respectively).

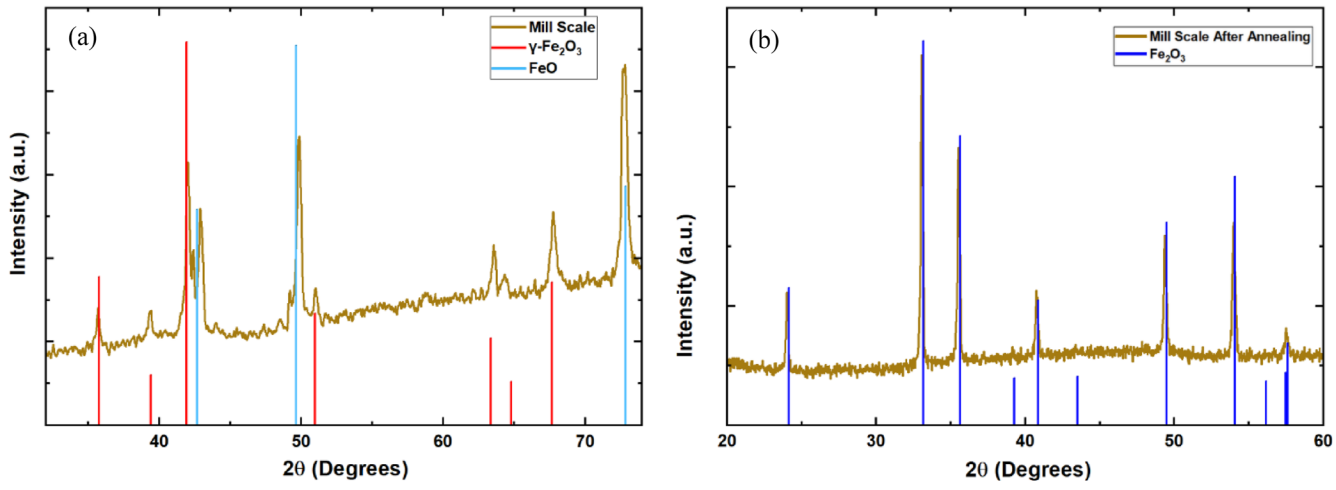


FIG. 1. The XRD analysis of the mill scale as the iron source before (a) and after annealing 6 h at 1190 °C (b).

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$SrFe_{12}O_{19}$ synthesized from mill scale was doped with Al_2O_3, Cr_2O_3, La_2O_3, and Co_3O_4 to modify the magnetic properties. Al_2O_3 incorporation enhances magnetic anisotropy and coercivity but lowers Curie temperature and grain size due to the smaller radius of Al^{3+} compared to Fe^{3+}, while also reducing remanent magnetization and altering particle morphology.[14] Cr_2O_3 doping similarly increases coercivity and anisotropy, though it decreases lattice size and retentivity.[5] La_2O_3 addition improves coercivity, retentivity, and anisotropy, but promotes coarse-grain growth.[3,13] In contrast, Co_3O_4 reduces grain size and has a positive effect on both coercivity and remanence.[4]

XRD data for the oxide-doped powders are shown in Fig. 2. The shift in the peaks is due to the replacement of Fe^{3+} ions in strontium ferrite by doped ions in the crystal lattices. The crystallite size was calculated from the width and length of the peaks by using the Profex.[16] Calculated crystallite sizes for MS + SC, MS + SC + Al_2O_3, MS + SC + Co_3O_4, MS + SC + Cr_2O_3, and MS + SC + La_2O_3 are 65, 56, 54, 42, and 40 nm, respectively.

The powder synthesis procedure significantly affects the particle size and morphology of metal oxides. In this study, the particle size is expected to be reduced to below 10 μm using 12 + 16 h of ball milling. Figure 3 represents the grain size distribution and morphology of ball-milled powders. The average particle size of Co_3O_4, Al_2O_3, Cr_2O_3, La_2O_3-doped, and as-made $SrFe_{12}O_{19}$, respectively, was measured as 1.83, 1.96, 2.23, 2.04, and 2.10 μm with standard deviation 0.49, 0.90, 0.66, and 0.62. The results of the present study confirm that the ball milling process is suitable for reducing metal oxides to sizes below 10 μm. Among the various oxides, Co_3O_4-doped samples had the most homogeneous and compact structure, based on the minimal standard deviation.

The correlation between particle size distribution and additive oxides suggests that an appropriate choice of additives can significantly improve powder and structural uniformity. These results align with earlier research on the impact of milling conditions on the morphology and distribution of strontium hexaferrite.[17,18] Chen *et al.* demonstrated that extended milling durations enhance uniformity and reduce agglomeration,[19] while Kim *et al.* emphasized that milling parameters, including speed and additives, critically influence the final particle characteristics.[20]

Additionally, Table I displays the lattice parameters of $SrFe_{12}O_{19}$ and illustrates how the doped ions impact the structure's size. All contributions reduced the c parameter, but compared to the literature, the increase in the MS + SC sample may have been caused by the Co element in the mill scale. Al^{3+} and Cr^{3+} ions replace the Fe^{3+} ions in the structure, as observed in previous studies. Since the sizes of Cr^{3+} and Fe^{3+} are similar, the substitution cannot be directly observed; however, a decrease in the c parameter confirms the substitution in the hexaferrite lattice.[15,21] Additionally, the radius of Co^{2+} ions is very similar to that of Fe^{3+} ions, resulting in a slight decrease in contribution rates that is more pronounced than expected in previous studies.[4,22] Unlike other contributions,

TABLE I. The lattice parameters of the $SrFe_{12}O_{19}$ powders synthesized from the mill scale with different additives.

Composition	a (Å)	c (Å)	GoF
$SrFe_{12}O_{19}$	5.998	23.384	...
MS + SC	5.872	23.017	1.19
MS + SC + Co_3O_4	5.874	23.005	1.20
MS + SC + La_2O_3	5.883	23.034	1.25
MS + SC + Cr_2O_3	5.876	23.030	1.14
MS + SC + Al_2O_3	5.850	22.935	1.15

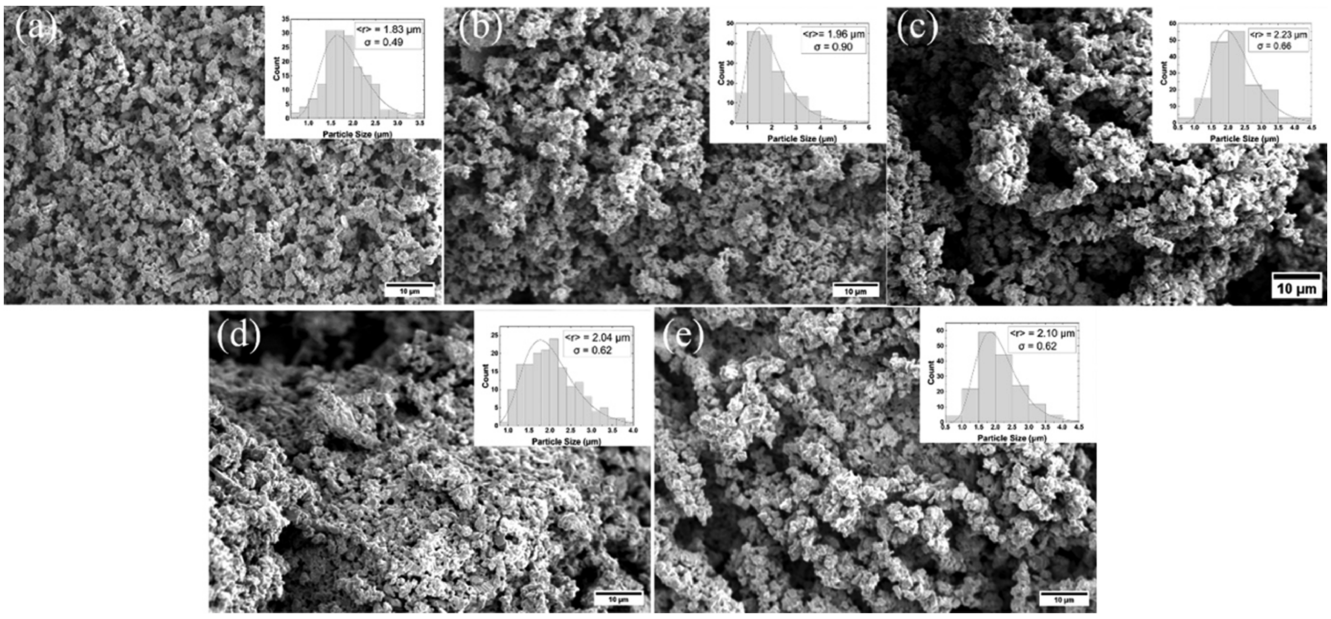


FIG. 3. SEM images of (a) Co_3O_4, (b) Al_2O_3, (c) Cr_2O_3, (d) La_2O_3 doped, and (e) as made $SrFe_{12}O_{19}$.

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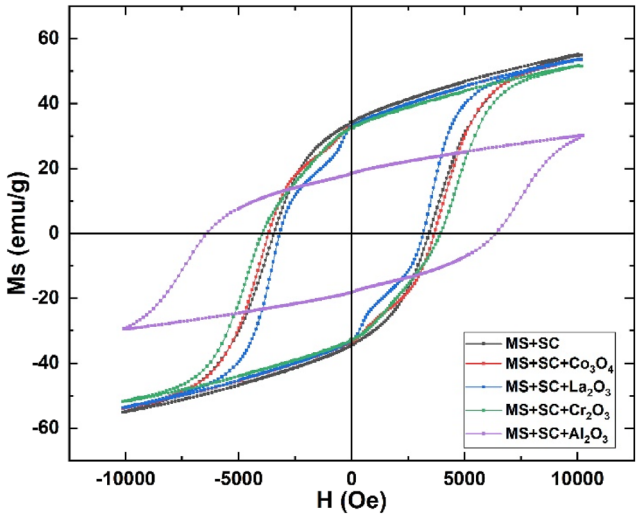


FIG. 4. The hysteresis loops of the powders.

La^{3+} ions replace Sr^{2+} ions, not Fe^{3+}. Since there is little difference in size between the two, a parameter has remained almost the same as in previous studies.[23]

VSM analysis was also performed to investigate the magnetic properties of the synthesized powders (Fig. 4). The MS + SC + La_2O_3 sample showed a shoulder in the demagnetization curve. While such phenomena can indicate secondary magnetic phases, our XRD analysis (Fig. 2) confirms the presence of the $SrFe_{12}O_{19}$ phase without detectable magnetic impurities. This behavior is therefore attributed to the wide size distribution of powder particles [Fig. 3(d)].

Several magnetic properties have been summarized in Table II. Except for the alumina-doped sample, it has been seen that the coercivity and remanent magnetization values were similar. Additionally, the Mr/Ms values were between 0.613 and 0.630, indicating an isotropic nature. The MS + SC + Al_2O_3's remanent magnetization (Mr) is lower, and coercivity (Hc) is higher than others. According to Icin *et al.*,[15] as the Al^{3+} amount increases in the structure, coercivity rises due to the needle-shaped grain structure [Fig. 3(b)], inducing shape anisotropy and remanent magnetization decreases. Compared to previous studies[24–26] that utilized other production methods, upcycled Sr ferrite magnets have been successfully synthesized with an average surface magnetic flux of ~600 G.

TABLE II. The magnetic properties summary of the samples shows saturation magnetization (Ms), coercivity (Hc), and remanence magnetization (Mr), as well as their ratio.

Composition	Mr (emu/g)	Ms (emu/g)	Mr/Ms	Hc (Oe)
MS + SC	33.03	53.59	0.616	3670
MS + SC + Co_3O_4	34.02	55.06	0.618	3448
MS + SC + La_2O_3	32.88	53.40	0.615	3175
MS + SC + Cr_2O_3	32.44	51.49	0.630	3955
MS + SC + Al_2O_3	18.50	30.14	0.614	6401

CONCLUSIONS

The study suggests that mill scale recycling can be a sustainable resource for the magnet industry, promoting environmentally friendly production of strontium ferrite. XRD analysis confirmed the transformation of the initial mill scale, with a maghemite phase, into hematite following heat treatment, allowing for the formation of strontium hexaferrite. In all doped structures, the desired $SrFe_{12}O_{19}$ phase was observed. Metal oxide particle sizes in the ball milling process were effectively reduced to below 10 μm, with the most uniform structure observed in Co_3O_4-doped samples. VSM analysis showed that all the samples exhibited strong ferromagnetic behavior, with slight (except Al_2O_3) variations in remanent magnetization and coercivity based on the type of dopant.

The Mr/Ms ratio suggested an isotropic nature. Despite the decrease in remanent magnetization for the Al_2O_3-doped sample, the overall magnetic properties of the synthesized strontium hexaferrite were comparable to those produced by conventional methods, validating the efficiency of this study.

In conclusion, the successful synthesis of strontium hexaferrite from mill scale and doping with various metal oxides demonstrates the applicability of recycling for producing functional magnetic materials with adjustable properties.

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AUTHOR DECLARATIONS

Conflict of Interest

The authors have no conflicts to disclose.

Author Contributions

All authors contributed equally to this work.

Nilay Gunduz Akdogan: Conceptualization (equal); Investigation (equal); Methodology (equal); Validation (equal); Visualization (equal); Writing – original draft (equal); Writing – review & editing (equal). **Duru Kalkavan**: Formal analysis (equal); Visualization (equal); Writing – original draft (equal). **Shabnam Ghorbanighoshchi**: Formal analysis (equal); Visualization (equal); Writing – original draft (equal). **Ozan Akdogan**: Conceptualization (equal); Formal analysis (equal); Methodology (equal); Validation (equal); Visualization (equal); Writing – original draft (equal); Writing – review & editing (equal).

DATA AVAILABILITY

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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