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Exploring the Effects of Annealing Temperature on Structural and Magnetic Properties of Low-Level Neodymium-Substituted Strontium Hexaferrite Synthesized Via Combustion Method

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ABSTRACT: This study investigates the impact of annealing temperature on the structural and magnetic attributes of Nd-substituted strontium hexaferrite ($Sr_{0.9}Nd_{0.1}Fe_{12}O_{19}$). The research demonstrated that as the annealing temperature increased, diffusion accelerated, thereby inducing a transformation into the $SrFe_{12}O_{19}$ phase and enhancing hard magnetic properties. The annealing process also significantly impacted the bonding structure, specifically affecting the presence of N-O bonds. Peak intensities escalated until 1100°C, subsequently declining due to the emergence of a composite structure ($SrFe_{12}O_{19}$+α-Fe_2O_3) arising from the Nd substitution. Importantly, the coercive field of the compound substantially increased post-annealing, attaining a peak at 1100°C (5.21 kOe) from 0.52 kOe pre-annealing, indicating that annealing amplifies resistance to demagnetization. Furthermore, both remanent magnetization and saturation magnetization values improved, reaching maximum values at 1100°C, measuring 40.2 emu/g and 61.5 emu/g, respectively.

Keywords: Combustion synthesis, Hexaferrite, Magnetic properties, Rare-earth substitution.

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1. INTRODUCTION

Strontium hexaferrites, also known as $SrFe_{12}O_{19}$, are a class of magnetic materials with unique properties that make them invaluable in numerous applications (Zhang, et al., 2023; Zhivulin, et al., 2023). Derived from the hexagonal ferrite family, these compounds exhibit extraordinary magnetic properties, including high uniaxial magnetic anisotropy and coercivity. They are primarily used in permanent magnets due to their ability to maintain a strong magnetic field (Lim, et al., 2023; Rambabu, et al., 2023; Verma, et al., 2023). Furthermore, strontium hexaferrite have been widely adopted in various sectors, such as electronics, telecommunications, and the automotive industry, because of their high chemical stability, relatively low cost, and ease of production (Gulbadan, et al., 2023; Için, et al., 2022; Kolev, et al., 2022; Serrano, et al., 2021; Zahid, et al., 2021). Lately, extensive research is being directed towards understanding the impact of doping and substitution in strontium hexaferrites in order to enhance their magnetic properties and temperature stability (Elanthamilan, et al., 2023; R. S. Liu, et al., 2023; M. Zhang, et al., 2022; W. H. Zhang, et al., 2022; Wenhao Zhang, et al., 2022).

The unit cells in the crystal structure of strontium hexaferrite magnets are hexagonal and exhibit strong magnetic properties. The dense packing structure of strontium hexaferrite magnets provides a suitable environment for permanent magnet properties. Considering the crystal structure of the magnet, this ferrite magnet has a complex magnetoplumbite-type structure, in which oxygen ions coordinate iron ions in a tetrahedral (FeO_4), trigonal bipyramidal (FeO_5), and octahedral (FeO_6) manner (Li, et al., 2023; Rambabu, et al., 2021; Serrano, et al., 2021; Shariff, et al., 2021). Essentially, studies have been related to the physical properties, characterization, and measurements of strontium hexaferrite magnets. $SrFe_{12}O_{19}$ have shown good electrical properties and a strong anisotropy. M-type strontium hexaferrite magnets crystallize in 64 ions in 11 different symmetry regions in the magnet unit cage. In the densely packed layers, Fe^{3+} ions are distributed in 5 crystallographic interstices. These are; three octahedral (2a, 12k, and 4f2), one tetrahedral (4f1), and one trigonal bipyramidal (2b) site 3 parallel (2a, 12k and 2b) and 2 anti-parallel (4f1 and 4f2) subcages match with O^{2-} ions to form the ferrimagnetic structure (Bohlender, et al., 2019; Gultom, et al., 2020; Oura, et al., 2018; Wenhao Zhang, et al., 2022).

The magnetic moments of ions in crystal structures determine the magnetic behavior of materials. The magnetic properties of these magnetic materials can be altered by doping or impurity incorporation into the crystal structure. Co-doping in $SrFe_{12}O_{19}$ materials causes a change in magnetic properties. This dopant causes changes in the positions of the Fe^{3+} ions, increasing their net magnetic moments and thus enhancing the magnetic behavior of the material. Cobalt provides high-performance magnetic materials by adding high coercive field and excellent magnetic properties to $SrFe_{12}O_{19}$ materials (Granja-Banguera, et al., 2022; Jianfeng, et al., 2022; Ruoshui Liu, et al., 2023). When aluminum (Al) dopant is added to $SrFe_{12}O_{19}$ materials, it becomes more resistant to temperature changes and its magnetic properties become more stable. This dopant causes a decrease in the net magnetic moment created by Fe^{3+} ions in the structure, but the coercive field of the material increases (Gorbachev, et al., 2023; Joshi, and Ruban Kumar, 2023; Manjunatha, et al., 2023). The effect of the Cu dopant or replacement depends on the addition ratio. At low ratios, the Cu dopant improves the remnant magnetization and saturation magnetization properties, while at high ratios, the Cu dopant weakens these properties (Ateia, et al., 2023; Ghimire, et al., 2018; Iqbal, and Ashiq, 2008; Muhammad Javed, et al., 2009; Qiao, et al., 2007).

The use of rare earth elements to modify the magnetic and structural properties of hexaferrites has attracted increasing attention due to the diverse applications that these materials can offer. In

particular, the ability of rare earth elements to induce significant changes in the magnetic and structural characteristics of hexaferrites makes them invaluable tools in the development of advanced magnetic materials (Liu, et al., 2019; Luo, et al., 2015; Singh, et al., 2014; Wang, et al., 2004; Wang, et al., 2005). Hexaferrites, an important class of magnetic materials, have gained prominence due to their unique combination of high magnetic permeability, low cost, and excellent temperature stability. However, their magnetic and structural properties can be tailored to a significant extent using rare earth elements, which can introduce new functionalities to these materials. The ionic radii and the valence state of rare earth elements, which are larger and more varied than those of iron, can significantly affect the crystal structure, leading to a modification of the magnetic properties. For instance, the substitution of rare earth elements, such as Neodymium (Nd) (Bercoff, et al., 2009) and Samarium (Sm) (K, et al., 2020), into the hexaferrite lattice can significantly enhance the magnetic anisotropy and coercivity. This can be extremely beneficial in applications such as permanent magnets, where high coercivity and remanent magnetization are desired. Furthermore, such substitutions can also improve the Curie temperature, making these materials suitable for high-temperature applications. On the structural front, rare earth substitutions can lead to the formation of different types of crystal structures. Depending on the type and concentration of the rare earth element used, one can achieve various types of crystal structures, such as spinel, garnet, or perovskite. This structural versatility enables the design of magnetic materials with desired magnetic properties.

In this study, we investigate the structural and magnetic property changes that occur when rare earth elements (REEs) are substituted for strontium (Sr). Previous research has primarily focused on the substitution of REEs for Sr in Fe-based compounds; however, in this investigation, we extend our analysis to explore the effects of REEs replacing Fe sites. The primary aim of this study is to gain a comprehensive understanding of how these dual substitutions impact the crystal structure and magnetic characteristics of the materials. By delving into this unexplored territory, we aim to contribute valuable insights to the field of materials science, providing a basis for the design and development of novel functional materials with tailored properties for various technological applications. In the context of this research, we've undertaken a comprehensive investigation into the effects of annealing temperature on the structural and magnetic characteristics of a $Sr_{0.9}Nd_{0.1}Fe_{12}O_{19}$ composition, where 0.1 Nd has been substituted for Sr. The main aim of this research is to better comprehend how these characteristics behave when subjected to different annealing temperatures. Additionally, it aims to clarify how substituting Nd impacts the magnetic properties of strontium hexaferrite.

2. MATERIALS AND METHODS

Within the scope of this study, strontium hexaferrite (SrM) powder with a 0.1 Nd additive was produced using the solution combustion synthesis technique (Figure 1). In the $SrFe_{12}O_{19}$ composition that enables the emergence of the hard magnetic phase in strontium hexaferrites, Nd was added in place of Sr. In the production of Nd-doped strontium hexaferrites, the Sr ratio was reduced by 0.1, and Nd was added in its place. Thus, the chemical compound aimed to be formed can be expressed as $Sr_{0.9}Nd_{0.1}Fe_{12}O_{19}$. In the production of the $Sr_{0.9}Nd_{0.1}Fe_{12}O_{19}$ compound, precursor chemicals, i.e. strontium nitrate $Sr(NO_3)_2$, iron nitrate ($Fe(NO_3)_2.9H_2O$), neodymium nitrate ($Nd(NO_3)_3.6H_2O$), were obtained from Sigma Aldrich. These compounds were purity of 99.9%. Citric acid, was also obtained from Sigma Aldrich and used as the necessary fuel for combustion to take place in solution combustion synthesis. The stoichiometric ratio was prepared according to the reaction given in

Equation 1. As a result of previous experimental studies, the stoichiometric ratio between Sr and Fe has been determined as 1:10.

$$Sr(NO_3)_2+Fe(NO_3)_2.9H_2O+Nd(NO_3)_2.6H_2O+C_6H_{10}O_8 \rightarrow SrNdFe_{12}O_{19}+H_2O+CO_2+N_2 \quad (1)$$

As part of the production process for strontium hexaferrite via the auto-combustion method, nitrate compounds and citric acid as a fuel were accurately weighed according to the ratios determined to produce 5 grams of $SrFe_{12}O_{19}$ and added to 30 ml of pure water. The mixture was stirred until a homogeneous solution was formed (approximately 2 hours) at 300 rpm. Ammonia (NH_3) was added to raise the pH of the resulting homogeneous mixture to 7. The solution was further stirred until the pH reached equilibrium (approximately 1 hour). Following this process, the solution was placed on a heating plate at 350 °C, and gel formation was initiated. After the gel formation process was completed, auto-combustion took place due to an exothermic reaction, resulting in the production of strontium hexaferrite precursor powders. The nano-sized powders produced by the exothermic combustion were initially ground in an agate mortar to break up partial agglomerations, and then subjected to high-energy ball milling to completely dissolve the lumps. In a high-energy ball mill, powders were gently milled using a 1:5 ball to powder ratio at 300 rpm in a WC mill with 10mm diameter WC balls. After the mild grinding process, an annealing heat treatment was applied to form the $SrFe_{12}O_{19}$ structure. To study the impact of phase changes occurring at different temperatures on the magnetic properties of strontium hexaferrite powders produced by auto-combustion synthesis from mixtures prepared according to $Sr_{0.9}Nd_{0.1}Fe_{12}O_{19}$ composition, an annealing process was carried out at atmospheric conditions starting from 900 °C, respectively at 1000, 1100, and 1200 °C for 1 hour. Due to the annealing process at high temperatures, a mild grinding process was applied for 5 minutes to break up the partial agglomerations that occurred between the powders. Various methods were employed to examine and analyze strontium hexaferrite magnets in detail. These methods included X-ray diffraction (XRD), Fourier transform infrared spectrophotometer (FTIR), vibration sample magnetometer (VSM). Phase changes in the structure of the produced strontium hexaferrite powders, changes in the crystal structure, were examined with an X-ray diffractometer. These analyses were conducted in the Central Research Laboratory of Karadeniz Technical University on a PANalytical X'pert Powder^3 model XRD device. FTIR analyses were performed in the wave number range of 500-4000 cm^{-1} with a Shimadzu brand IRSprit model FTIR device. The magnetic properties (coercivity, magnetic saturation, remanent magnetization) of strontium hexaferrite powders were measured using a Lakeshore brand 7304 Model vibrating sample magnetometer (VSM) at room temperature.

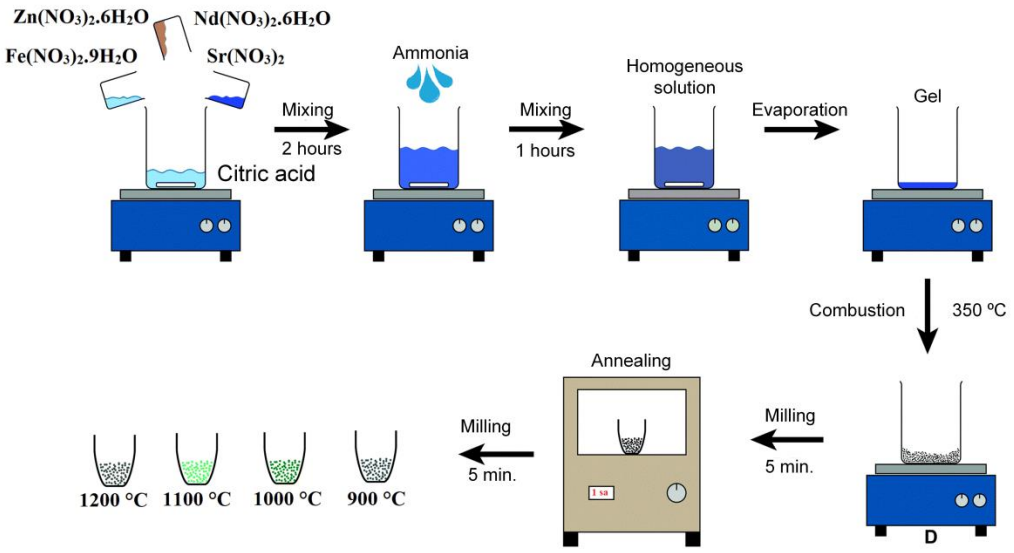

Figure 1. Schematic representation of the process flow diagram used in the study

3. RESULTS AND DISCUSSION

This research investigated the effects of substituting Nd for Sr on the phase structure of strontium hexaferrite, and how these effects evolve with temperature. The first stage of the study was a comprehensive analysis of the $Sr_{0.9}Nd_{0.1}Fe_{12}O_{19}$ composition's phase structure, both pre and post-annealing at temperatures ranging from 900 to 1200 °C, with the heat treatment lasting an hour. This experimental setup enabled the study of the effect of Nd-substitution at elevated temperatures, a critical aspect that affects the phase formation and properties of the material. In Figure 2, the graphical representation of the phase structural changes, it was observed that the initial phase formed in the strontium hexaferrite powders doped with 0.1 Nd, before annealing, is predominantly Fe_3O_4. This finding indicates that Nd substitution initially influences the phase composition by favoring the formation of Fe_3O_4. However, this phase composition does not remain constant. It undergoes dramatic changes with the onset of the annealing process, even at the minimum annealing temperature. The prevalent phase appears to be the $SrFe_{12}O_{19}$ phase, distinguished by its hard magnetic properties and hexagonal crystal lattice structure. This transition reveals the temperature-sensitive nature of the phase transformations, and the pivotal role annealing temperature plays in determining the resultant phase and hence, the magnetic properties of the material. With increasing annealing temperatures, there's a complete transformation of all peaks into $SrFe_{12}O_{19}$, a consequence of enhanced diffusion. The elevated temperature facilitates the mobility of atoms, enabling the formation of the $SrFe_{12}O_{19}$ phase, indicative of Nd's role in enhancing the diffusion process. The α-Fe_2O_3 phase, observed at $2\theta=33°$, persists even after the annealing process at 900 °C for an hour. Despite possessing relatively low magnetic properties, it remains unreacted in the structure, suggesting that the kinetic parameters (temperature and time) for the formation of the $SrFe_{12}O_{19}$ phase were not sufficiently met at this annealing temperature. As the temperature increases to between 1000-1200 °C, the α-Fe_2O_3 phase diminishes but a small impurity peak at $2\theta=36°$ persists in the structure, indicating the tenacity of the phase even at elevated temperatures. Furthermore, the primary diffraction peaks of the $SrFe_{12}O_{19}$ phase at $2\theta=32°$ and 34° show a decrease in width and an increase in intensity with an increase in annealing temperature. These variations in peak intensity and width suggest that the $SrFe_{12}O_{19}$ crystal size experiences growth along with the annealing temperature. The augmentation in crystal size at higher annealing temperatures has been documented in several studies, reinforcing the understanding

of temperature-dependent crystal growth. A study conducted by Urbano-Peña and colleagues suggested (Urbano-Peña, et al., 2019) that at temperatures above the phase temperature, the atomic movement within the structure is accelerated, leading to quicker phase transformations. In scenarios where phase transformation is rapid, grain growth is more likely. These findings are parallel to our observations, strengthening the argument that grain growth accompanies fast phase transformations.

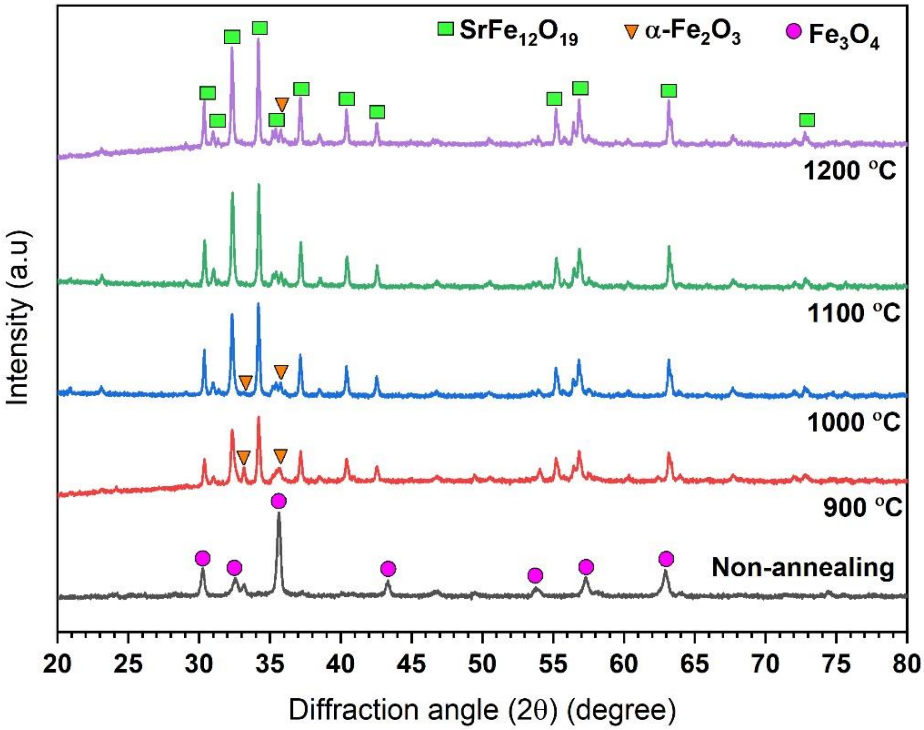

Figure 2. X-ray diffraction pattern of Nd substitution strontium hexaferrite powders

In this study, Fourier Transform Infrared Spectroscopy (FTIR) analyses were used to study the bond changes of neodymium (Nd) substituted strontium hexaferrite powders with respect to the annealing temperature. FTIR is a robust spectroscopic technique that provides valuable insights into the molecular structure and chemical bonding of materials. The results, as depicted in Figure 3, offer a detailed view of the impact of Nd substitution and annealing temperature on the material's bonding. The FTIR analyses were carried out at room temperature, spanning a broad spectral range of 400-1500 cm^{-1}. Room temperature measurements ensure the stability of the sample and prevent additional thermal effects from influencing the results. Since all the FTIR spectra resulted in similar vibrations, each graph was juxtaposed for a comparative analysis. This comparative approach offers a clearer understanding of the subtle differences arising due to Nd substitution and varying annealing temperatures. The $SrNdFe_{12}O_{19}$ compositions containing 0.1 Nd, synthesized using nitrate precursors and citric acid, exhibited absorption peaks of metal oxides within approximately 750-500 cm^{-1} in the FTIR spectrum. These metal oxide peaks are pivotal indicators of the nature of the chemical bonds present within the sample. Moreover, an absorption peak possibly related to N-O was observed at around 1450 cm^{-1}, signifying the potential presence of nitrogen-containing compounds. However, an interesting transformation occurred upon annealing the $Sr_{0.9}Nd_{0.1}Fe_{12}O_{19}$ between 900-1200 °C. The N-O related absorption peak vanishes, revealing a structure dominated by metal-oxygen bonds. This change underscores the importance of the annealing temperature in affecting the bond changes in Nd-substituted strontium hexaferrite powders. The absorption peaks at 566 and 599 cm^{-1} are associated with M-O (Sr-O or Fe-O) lattice vibrations, acting as spectral fingerprints for these metal-oxygen

bonds. The differences in vibrations are due to the dissimilar atomic weights of Sr and Fe, with lower (560 cm^{-1}) and higher (599 cm^{-1}) vibrations corresponding to Sr-O and Fe-O bonds respectively. Furthermore, the intensity of these absorption peaks showed a distinctive pattern based on the annealing temperature. The peak intensity increased until reaching 1100°C, after which it started to decrease. This variation in intensity is attributed to the composite nature of the powders' composition, as a result of Nd substitution, featuring $SrFe_{12}O_{19}$ and α-Fe_2O_3 phases. As the proportion of the α-Fe_2O_3 phase grows with Nd substitution, a new type of Fe-O bond emerges in the structure. An increment in this phase would lead to an increase in vibrations caused by the new Fe-O bond, intensifying the absorption peak seen at 599 cm^{-1}. These findings offer crucial insights into the spectral behavior of the Nd substituted strontium hexaferrite powders, underlining the instrumental role of Nd substitution and annealing temperature in modulating the material's bonding structure.

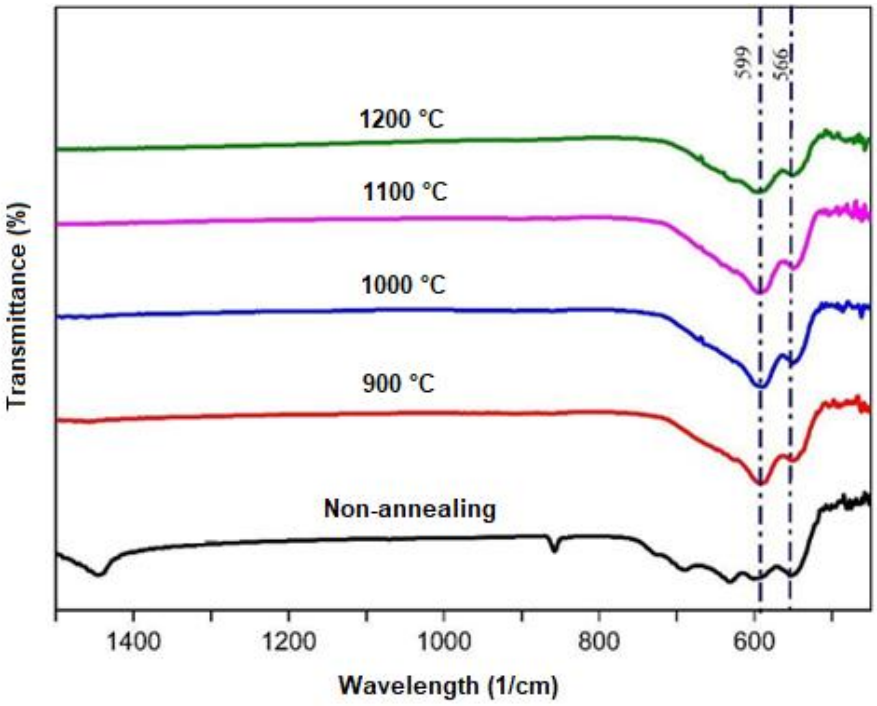

Figure 3. FTIR spectrum of annealed at various temperature Nd substituted strontium hexaferrite powders

This study also provides an in-depth analysis of the magnetic properties of $Sr_{0.9}Nd_{0.1}Fe_{12}O_{19}$, a neodymium (Nd) substituted strontium hexaferrite compound. These properties, which include the coercive field (H_c), remanent magnetization (M_r), and saturation magnetization (M_s), are essential in understanding the behavior of the material under different conditions. The hysteresis loops of the Nd-substituted strontium hexaferrite powders are shown in Figure 4. All magnetic properties of produced magnetic powders are presented in Table 1. Before annealing, the H_c of $Sr_{0.9}Nd_{0.1}Fe_{12}O_{19}$ was recorded to be 0.52 kOe. The coercive field represents the material's resistance to demagnetization. At this initial stage, the remanent magnetization, which indicates the material's magnetization in the absence of an external magnetic field, stood at 18.2 emu/g. Meanwhile, the saturation magnetization, referring to the maximum magnetization that the material can attain, was observed to be 59.5 emu/g. The annealing process, which involves heating the material and allowing it to cool slowly, has significant effects on the magnetic properties of the strontium hexaferrite. As the compound was annealed at various temperatures (900°C, 1000°C, 1100°C, and 1200°C), remarkable changes were observed. At 900°C, the coercivity increased significantly to 5.04 kOe, suggesting improved

resistance to demagnetization. The remanent and saturation magnetization values altered to 32.4 emu/g and 49.7 emu/g, respectively. Further increase in the annealing temperature to 1000°C resulted in a slight decrease in H_c (5.02 kOe), while both M_r and M_s improved (39.1 emu/g and 60.6 emu/g, respectively), suggesting an overall enhancement in magnetic performance. At 1100°C, the coercivity further grew to 5.21 kOe, indicating increased stability in the magnetic state of the material. The remanent and saturation magnetization values followed suit, reaching 40.2 emu/g and 61.5 emu/g, respectively. However, upon reaching an annealing temperature of 1200°C, the coercivity fell to 4.42 kOe, while the remanent and saturation magnetization declined slightly to 38.9 emu/g and 59.2 emu/g, respectively. This points to an optimal annealing temperature range for maximizing the magnetic properties of $Sr_{0.9}Nd_{0.1}Fe_{12}O_{19}$. The observed changes in the magnetic properties of the $Sr_{0.9}Nd_{0.1}Fe_{12}O_{19}$ compound are intrinsically linked to the underlying structural modifications occurring during the annealing process. It is important to highlight that the structure and magnetic properties of a material are closely interconnected. With an increase in annealing temperature up to 1100°C, there is a noticeable enhancement in the compound's H_c, M_r and M_s. This improvement is primarily attributed to the formation and growth of the hard magnetic $SrFe_{12}O_{19}$ phase. As the Nd-substituted strontium hexaferrite is annealed, diffusion processes are enhanced, which leads to the development and enlargement of the $SrFe_{12}O_{19}$ phase. This hard magnetic phase, characterized by a hexagonal crystal lattice structure, is crucial for the elevated magnetic properties observed. The growth of the $SrFe_{12}O_{19}$ phase leads to an increase in the remanent magnetization and the saturation magnetization. This is because the $SrFe_{12}O_{19}$ phase, being a hard magnetic phase, possesses superior magnetic properties compared to the original Nd-substituted ferrite. The increase in coercivity, which indicates better resistance to demagnetization, is also a typical feature of hard magnetic materials. However, when the annealing temperature surpasses the optimal range, around 1200°C in this case, there is a decline in the magnetic properties. This drop can be linked to the occurrence of grain growth induced by the elevated temperatures. With the grain size exceeding the single-domain size, multi-domain particles are formed. The transition from single domain to multi-domain particles usually results in a decline in magnetic properties due to the emergence of demagnetizing fields within the material. Therefore, the slight reduction in coercivity and magnetization at 1200°C is associated with the onset of multi-domain behavior due to grain growth. Thus, the careful control of annealing temperature is critical to balance the beneficial growth of the hard magnetic $SrFe_{12}O_{19}$ phase and mitigate the negative impacts of excessive grain growth, allowing for the optimization of the magnetic properties of the $Sr_{0.9}Nd_{0.1}Fe_{12}O_{19}$ compound.

Table 1. Magnetic properties of strontium hexaferrite powders

	$Sr_{0.9}Nd_{0.1}Fe_{12}O_{19}$		
	H_c kOe	M_r emug^{-1}	M_s emug^{-1}
Non-annealing	0.52	18.2	59.5
900	5.04	32.4	49.7
1000	5.02	39.1	60.6
1100	5.21	40.2	61.5
1200	4.42	38.9	59.2

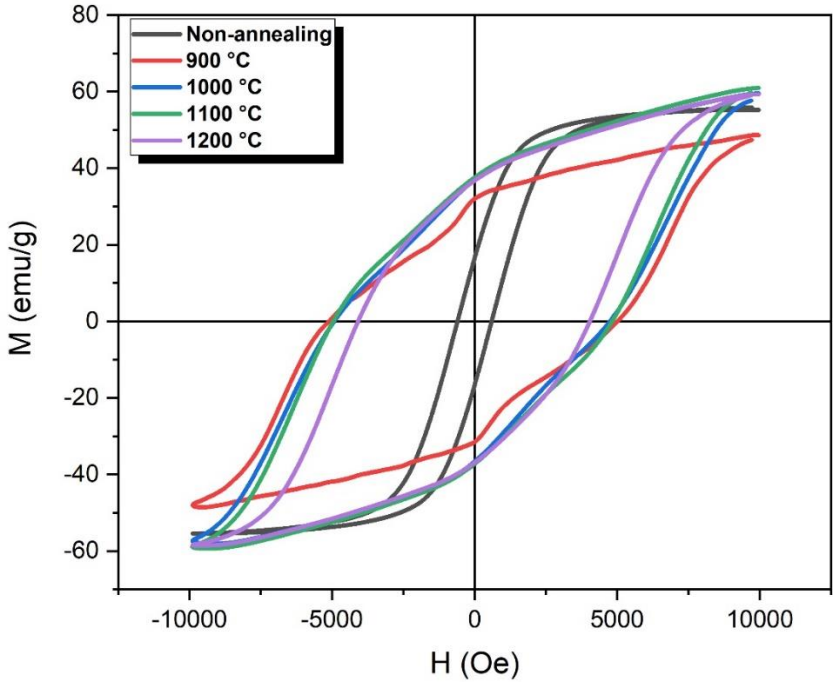

Figure 4. Hysteresis loops of Nd substitution strontium hexaferrite powders

4. CONCLUSION

Building upon the substantial body of research emphasizing the modification of strontium hexaferrites' magnetic properties via ion substitution, this study has endeavored to delve deeper into the nuanced impacts of Nd substitution on the structural, and magnetic characteristics of a $Sr_{0.9}Nd_{0.1}Fe_{12}O_{19}$ composition. A comprehensive exploration of these properties across a range of annealing temperatures has been conducted to elucidate the dynamic interplay between material composition, processing conditions, and resultant properties. Some important observed results were summarized below:

- Increased annealing temperatures expedite diffusion, leading to a transformation into the $SrFe_{12}O_{19}$ phase and enhancement in hard magnetic properties.
- The non-annealed samples displayed absorption peaks related to both metal oxides and N-O, while after the annealing process, the N-O related peak disappeared. This indicates a significant impact of the annealing temperature on the bonding structure, particularly affecting the presence of the N-O bond.
- There was an escalation in peak intensities up to 1100°C annealing temperature, after which a decline was observed due to the composite structure ($SrFe_{12}O_{19}$+α-Fe_2O_3) emerging from the Nd substitution, consequently modifying the Fe-O bond in the structure.
- The coercive field of the $Sr_{0.9}Nd_{0.1}Fe_{12}O_{19}$ compound increased significantly with annealing, reaching its peak at 1100°C (5.21 kOe) from a mere 0.52 kOe before annealing. This indicates that annealing effectively enhances the compound's resistance to demagnetization.
- Annealing also improved the remanent magnetization and saturation magnetization values, demonstrating the highest values at 1100°C with 40.2 emu/g and 61.5 emu/g, respectively.

5. ACKNOWLEDGEMENTS

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6. CONFLICT OF INTEREST

Authors approve that to the best of their knowledge, there is not any conflict of interest or common interest with an institution/organization or a person that may affect the review process of the paper.

7. AUTHOR CONTRIBUTION

In this study, all authors have contributed equally. Esin Demir: Investigation; Sefa Emre Sünbül: Original Draft and software; Kürşat İÇİN: supervisor, analysis, original draft.

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