Magnetic properties of polycrystalline Y-type hexaferrite $\text{Ba}_{2-x}\text{Sr}_x\text{Ni}_2(\text{Fe}_{1-y}\text{Al}_y)_{12}\text{O}_{22}$ using Mössbauer spectroscopy

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ABSTRACT

Synthesis of the polycrystalline \( \text{Ba}_{2-x}\text{Sr}_x\text{Ni}_2(\text{Fe}_{1-y}\text{Al}_y)_{12}\text{O}_{22} \) \((x = 0.0, 1.5, y = 0.00, 0.01, \text{and} 0.03) \) was accomplished by employing the polymerizable complex method. The samples were investigated for the crystallographic and magnetic properties by x-ray diffraction (XRD), vibrating sample magnetometer (VSM), and Mössbauer spectrometer. Based on the results of the refined XRD patterns, all samples were confirmed to have a rhombohedral structure with space group \( \text{R}-3m \). Moreover, six distinguished sublattices were identified, which were four octahedral sites \((18h\text{VI}, 3h\text{VI}, 6c\text{VI}, \text{and} 3a\text{VI}) \) and two tetrahedral sites \((6c\text{IV}, 6c\text{IV}) \). The lattice constant of \( a_0 \) and \( c_0 \) decrease via \( \text{Sr}, \text{Al} \) substitution because the ionic radius of \( \text{Sr}^{2+} \) (1.12 Å) is smaller than that of \( \text{Ba}^{2+} \) (1.34 Å), and the ionic radius of \( \text{Al}^{3+} \) (0.535 Å) is smaller than that of \( \text{Fe}^{3+} \) (0.645 Å). The zero-field-cooled (ZFC) measurement was applied at 100 Oe between 4.2 and 295 K and it revealed the spin transition temperature \((T_s) \). Substitution of \( \text{Sr} \) ions increased \( T_s \). \( \text{Al} \) ions were further substituted after the substitution of \( \text{Sr} \) ions, and \( T_s \) increased above room temperature. The Mössbauer spectra fit six distinguish sublattices: \( 18h\text{VI}, 3h\text{VI}, 6c\text{VI}, 6c\text{IV}, 6c\text{IV}, \text{and} 3a\text{VI} \). The isomer shift of all samples indicated that the charge state of \( \text{Fe} \) ions is \( \text{Fe}^{3+} \). The Mössbauer spectra obtained with respect to temperature change confirmed the changes in the magnetic hyperfine field curves at \( T_s \).

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I. INTRODUCTION

The Y-type hexaferrite has certain physical characteristics, such as the magnetoelectric (ME) effect based on spin-current model, multiferroicity, spin structure according to temperature and magnetic field. Especially, the ME effects are observed based on strong super-exchange interactions between \( \text{Fe} \) ions. Therefore, the various magnetic properties of hexaferrite have been extensively studied.\(^{2-4} \) However, the temperature applied to the operating devices is low due to their weaker magnetic energy as compared to the lattice energy.\(^{2-4} \) A high magnetic ordering temperature is required for utilizing the advantages of these hexaferrites. Some Y-type hexaferrites show a magnetoelectric effect near room temperature such as \( \text{Ba}_2\text{Mg}_2\text{Fe}_{12}\text{O}_{22} \), \( \text{Ba}_2\text{Sr}_2\text{Zn}_2(\text{Fe}_{11}\text{Al}_1)_{12}\text{O}_{22} \).\(^{5,6} \) It has been reported that the helimagnetic structure is stabilized by partially substituting \( \text{Ba} \) ions with \( \text{Sr} \) ions.\(^{7} \) In \( \text{Ba}_{0.5}\text{Sr}_{1.5}\text{Co}_2\text{Fe}_{12}\text{O}_{22} \), the helical magnetic structure was observed at 255 K and higher than the 215 K observed in \( \text{Ba}_2\text{Co}_2\text{Fe}_{12}\text{O}_{22} \).\(^{8,9} \) The magnetic properties changed due to the super-exchange interaction from ionic radius difference of \( \text{Ba} \) ions and \( \text{Sr} \) ions.\(^{10} \) And a small \( \text{Al} \) doped Y-type hexaferrite has shown ME effect with enhanced ME susceptibility.\(^{10-12} \) \( \text{Al} \) ion substitution of \( \text{Fe} \) ions at \( \text{Ba}_2\text{Co}_{1.3}\text{Mg}_{0.5}\text{Fe}_{12}\text{O}_{22} \) and \( \text{Ba}_{0.5}\text{Sr}_{1.5}\text{Co}_2\text{Fe}_{12}\text{O}_{22} \) have been reported to increase the spin transition temperature. Non-magnetic \( \text{Al} \) ion doping results in a change in \( T_s \) and Curie temperature by reducing the spin moment per unit cell and adjusting the spin exchange strength.\(^{10,11} \)

In this study, we investigated the magnetic properties by substituting \( \text{Sr} \) ions and small amounts of \( \text{Al} \) ions for \( \text{Ba}_2\text{Ni}_2\text{Fe}_{12}\text{O}_{22} \). We synthesized \( \text{Ba}_2\text{Sr}_x\text{Ni}_2(\text{Fe}_{11}\text{Al}_1)_{12}\text{O}_{22} \) using the polymerizable complex method. Mössbauer spectroscopy was performed to...
confirm the behavior of Fe ions resulting from the substitution of Sr and Al ions. Mössbauer spectroscopy can provide detailed information on each site, site occupancy, and hyperfine interaction. Furthermore, the VSM experiment for measuring the change in magnetic structure and the magnetization at room temperature was also conducted to confirm the magnetic properties.

II. EXPERIMENTS

The polycrystalline $\text{Ba}_{2-x}\text{Sr}_x\text{Ni}_2(\text{Fe}_{1-y}\text{Al}_y)_2\text{O}_{22}$ ($x=0.0, 1.5, y=0.00, 0.01, 0.03$) samples were synthesized by the polymericizable complex method. $\text{BaCO}_3$, $\text{SrCO}_3$, $\text{Ni(NO}_3)_2\cdot6\text{H}_2\text{O}$, $\text{Fe(NO}_3)_3\cdot9\text{H}_2\text{O}$, and $\text{Al(NO}_3)_3\cdot9\text{H}_2\text{O}$ were used as starting materials; these were prepared at stoichiometric ratios corresponding to $x$ and $y$, respectively. Further, these were dissolved in distilled water as a solvent. Total metal and citric acid were added at a ratio of 1:2.5 and subsequently mixed at 70°C for 2h. The mixture was heated to 120°C and dried until a homogeneous and stable gel complex was formed. Subsequently, the gel was calcined at 320°C for 2h. The mixture was then heated to 80°C for 1h. The mixture was then heated to 120°C and dried until a homogeneous and stable gel complex was formed. Subsequently, the gel was calcined at 320°C for 1h. The mixture was heated to 120°C and dried until a homogeneous and stable gel complex was formed. Subsequently, the gel was calcined at 320°C for 2h in air. Lastly, the obtained precursor was ground, palletized into cylindrical pellets and sintered at 1100°C for 10h in air to obtain the $\text{Ba}_{2-x}\text{Sr}_x\text{Ni}_2(\text{Fe}_{1-y}\text{Al}_y)_2\text{O}_{22}$ ($x=0.0, 1.5, y=0.00, 0.01, 0.03$) samples.

The crystal structure of the samples was investigated on the Rigaku Ultima IV by XRD using Cu-Ka radiation ($\lambda = 1.5406$ Å), and the results were analyzed by the Rietveld refinement method using the FULLPROF program. We used the VSM to measure the magnetic properties of the samples at various temperatures from 4.2 to 295 K under the maximum applied field of 15 kOe. Additionally, the site preference and hyperfine structure were investigated by the Mössbauer spectrometer with a $^{57}$Co source in a Rh matrix, which was calibrated using an $\alpha$-Fe foil.

III. RESULTS AND DISCUSSION

The XRD patterns were measured with a step size of 0.04 from 10° to 80°, and the lattice constants were confirmed via analysis using the FULLPROF program, as shown in Fig. 1. All samples made up of the rhombohedral structure with space group $R-3m$, and structural parameters of these samples are shown in Table I. The lattice constants of $a_0$, $c_0$ and unit cell volume were decreased by substituting the Sr ion from $\text{Ba}_2\text{Ni}_2\text{Fe}_{12}\text{O}_{22}$ ($x=0.0, y=0.00$). It was further decreased by substituting Al ions. It could be explained that the ionic radius of $\text{Sr}^{2+}$ (1.12 Å) is smaller than that of $\text{Ba}^{2+}$ (1.34 Å). In addition, the ionic radius of $\text{Al}^{3+}$ (0.535 Å) is smaller than that of $\text{Fe}^{3+}$ (0.645 Å). The Fe ions were found located at six crystallographic sites; these were corresponding four octahedral sites (18a), six tetrahedral sites (6b), one octahedral site (18c), and two tetrahedral sites (6c). The Ni, Al ions were located at four octahedral sites which preferentially occupied the octahedral site.

Figure 2 shows the magnetic hysteresis curves up to 15 kOe at 295 K for $\text{Ba}_{2-x}\text{Sr}_x\text{Ni}_2(\text{Fe}_{1-y}\text{Al}_y)_2\text{O}_{22}$ samples. The magnetization

![FIG. 1. Refined XRD patterns of $\text{Ba}_{2-x}\text{Sr}_x\text{Ni}_2(\text{Fe}_{1-y}\text{Al}_y)_2\text{O}_{22}$ ($x=0.0, 1.5, y=0.00, 0.01, 0.03$). The red circles represent the experimental observed data and the black lines represent calculated patterns. The green bar is the position of the allowed Bragg reflections.](image-url)

| $x, y$ | Chemical formula | $a_0$ (Å) | $c_0$ (Å) | $V$ (Å$^3$) |
|-------|------------------|-----------|-----------|------------|
| x=0.0, y=0.00 | $\text{Ba}_2\text{Ni}_2\text{Fe}_{12}\text{O}_{22}$ | 5.8498 | 43.3946 | 1286.042 |
| x=1.5, y=0.00 | $\text{Ba}_{0.5}\text{Sr}_{1.5}\text{Ni}_2\text{Fe}_{12}\text{O}_{22}$ | 5.8307 | 43.2527 | 1273.459 |
| x=1.5, y=0.01 | $\text{Ba}_{0.5}\text{Sr}_{1.5}\text{Ni}_2(\text{Fe}_{0.99}\text{Al}_{0.01})_{12}\text{O}_{22}$ | 5.8293 | 43.2382 | 1272.449 |
| x=1.5, y=0.03 | $\text{Ba}_{0.5}\text{Sr}_{1.5}\text{Ni}_2(\text{Fe}_{0.97}\text{Al}_{0.03})_{12}\text{O}_{22}$ | 5.8550 | 43.1985 | 1269.406 |

TABLE I. Lattice parameters of $\text{Ba}_{2-x}\text{Sr}_x\text{Ni}_2(\text{Fe}_{1-y}\text{Al}_y)_2\text{O}_{22}$ ($x=0.0, 1.5, y=0.00, 0.01, 0.03$).
of samples at 15 kOe was decreased by substituting Sr ion from Ba$_2$Ni$_2$Fe$_{12}$O$_{22}$ (x=0.0, y=0.00). When Al ion is substituted by 0.01, the magnetization was 20.4 emu/g at 295 K. The magnetization clearly decreased when Al ions were substituted by 0.03. This can be attributed to a decrease in the total spin moment due to substitution of the non-magnetic Al$^{3+}$ ions, which preferentially occupy the octahedral site with up-spin corresponding to 18$b_{IV}$, 6$c_{IV}$, and 3$a_{IV}$. Mössbauer parameters of Ba$_2$Sr$_{1-x}$Ni$_x$(Fe$_{0.97}$Al$_{0.03}$)$_{12}$O$_{22}$ samples are shown in Table II. The $H_{hf}$ decreased with increasing Sr substitution, and decreased when Al ions were further substituted. The relative site occupancy of Fe ions occurred at the same ratio as in the Sr ion substitution. When the Al ion was substituted, the relative area ratios of the up-spin octahedral sites, corresponding to 3$b_{IV}$, 18$b_{IV}$, and 3$a_{IV}$, decreased with increasing Al substitution. This is because Al ions preferentially occupied the octahedral sites leading to a reduction in the relative area ratio at the Fe ion site. This further causes a decrease in magnetization. The charged state of the Fe ions was determined to be Fe$^{2+}$ because the values of the isomer shift were less than 0.4 mm/s. Furthermore, we estimated

The temperature dependence of the zero-field-cooled (ZFC) magnetization curves were measured under 100 Oe from 4.2 to 295 K, as shown in Fig. 3. Inhibition of low-field magnetization suggests a transition of the magnetic structure, and this temperature is the spin transition temperature ($T_S$). In $x=0.0$, $y=0.00$ sample, the $T_S$ when the magnetic structure changes from helical magnetic structure to ferrimagnetic structure is around 47 K. Further, the $T_S$ is increased when Sr and Al ions are substituted. The Ba$_2$Sr$_{1.5}$Ni$_x$(Fe$_{0.97}$Al$_{0.03}$)$_{12}$O$_{22}$ ($x=1.5, y=0.03$) sample can be understood to be the state of the magnetic structure prior to changing to a ferrimagnetic at room temperature, resulting in a ME effect. This is the same result as the magnetization anomalies at a low field in the magnetic hysteresis curve. This feature is identical to the results observed with other Y-type hexaferrites such as Ba$_{0.5}$Zn$_{1.5}$Fe$_{12}$O$_{22}$, Ba$_2$Mg$_{0.5}$Fe$_{12}$O$_{22}$. Mössbauer spectroscopy can provide information on the distribution of ions and their magnetic structure when they have a variety of sites such as hexaferrite. Figure 4 shows analyzed Mössbauer spectra of all samples at 295 K. All Mössbauer spectra show six superposition of Fe sites. These were fitted using the least squares method with six sextets of six Lorentzian lines corresponding to 3$b_{VI}$, 6$c_{IV}$, 6$c_{VII}$, 18$b_{IV}$, 6$c_{V}$, and 3$a_{IV}$. The 18$b_{IV}$ site has the strong magnetic hyperfine field ($H_{hf}$), followed by 3$b_{IV}$, 6$c_{IV}$, 6$c_{VII}$, 6$c_{VI}$, and 3$a_{IV}$.
FIG. 4. Mössbauer spectra of Ba$_{2-x}$Sr$_x$Ni$_2$(Fe$_{1-y}$Al$_y$)$_{12}$O$_{22}$ ($x=0.0, 1.5, y=0.00, 0.01,$

and 0.03) at 295 K.

FIG. 5. Mössbauer spectra of Ba$_2$Ni$_2$Fe$_{12}$O$_{22}$ ($x=0.0, y=0.00$) between 4.2 and 295 K.

TABLE II. Mössbauer parameter of the Ba$_{2-x}$Sr$_x$Ni$_2$(Fe$_{1-y}$Al$_y$)$_{12}$O$_{22}$ ($x=0.0, 1.5, y=0.00, 0.01,$

and 0.03) at 295 K: magnetic hyperfine field $H_{hf}$, isomer shift $\delta$, and the occupation number of Fe ion $N_{Fe}(i)$.

| Site   | $x$, $y$ | $H_{hf}$ (kOe) | $\delta$ (mm/s) | $N_{Fe}(i)$ | $18\delta_{VII}$ | $3\delta_{VII}$ | $6\delta_{IV}$ | $6\delta_{IV}$ | $6\delta_{VIl}$ | $3\delta_{Vll}$ |
|--------|----------|----------------|-----------------|-------------|----------------|---------------|---------------|---------------|----------------|---------------|
| x=0.0, y=0.00 | | 480.13 | 0.20 | 8.69 | 473.51 | 0.22 | 464.19 | 0.18 | 427.23 | 0.32 | 425.01 | 0.04 | 385.65 | 0.14 |
| x=1.5, y=0.00 | | 455.73 | 0.20 | 8.69 | 451.80 | 0.16 | 443.17 | 0.21 | 402.78 | 0.09 | 397.42 | 0.07 | 377.48 | 0.25 |
| x=1.5, y=0.01 | | 461.60 | 0.23 | 8.61 | 460.83 | 0.16 | 451.94 | 0.20 | 418.92 | 0.19 | 403.08 | 0.13 | 390.39 | 0.15 |
| x=1.5, y=0.03 | | 462.05 | 0.23 | 8.44 | 457.62 | 0.16 | 444.25 | 0.20 | 413.50 | 0.33 | 406.77 | 0.03 | 379.40 | 0.08 |

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where $C_{hi}$ represents the compositions of the Fe ions and $S(i)$ is the relative area each site.

Figure 5 shows the fitted temperature dependence of the Mössbauer spectra for Ba$_2$Ni$_2$Fe$_{12}$O$_{22}$ from 4.2 to 295 K. The site occupancy of the Fe ions was calculated by the absorption area ratio. The relative area ratios of Fe ions for about six sub-lattices were constant during the super-exchange interaction, as shown in Fig. 6. A varying slope in the temperature dependence of $H_{hf}$ curve is confirmed around $T_s$, which is consistent with the $T_s$ confirmed in the ZFC curve in Fig. 3. Consequently, it was confirmed that the value of the isomer shift according to temperature change from 4.2 to 295 K was determined by the high spin state of Fe$^{3+}$. 

IV. CONCLUSION

We investigated the magnetic properties of Y-type hexaferrite Ba$_2$Sr$_2$Ni$_2$(Fe$_{1-x}$Al$_x$)$_{12}$O$_{22}$ that was synthesized via the polymerizable complex method. All samples employed for this study were rhombohedral with space group R-3m. The lattice constants and the unit cell volume decreased with increasing substitution of Sr ions and additional Al ions. The $H_{hf}$ of six sites decreased with increasing $x$ and $y$ as analyzed Mössbauer spectra. Based on the ZFC curve, Ba$_2$Ni$_2$Fe$_{12}$O$_{22}$ ($x=0.0, y=0.00$) sample confirmed $T_s$ at around 47 K, which could also be confirmed by the changing slope of $H_{hf}$ from the temperature dependence of Mössbauer spectra. Moreover, Ba$_{0.5}$Sr$_{1.5}$Ni$_2$(Fe$_{0.97}$Al$_{0.03}$)$_{12}$O$_{22}$ sample suggests that it is a magnetic structure in which the ME effect is expressed under a low external magnetic field at room temperature; this can be observed in the ZFC and magnetic hysteresis curves.

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