

FULL PAPER

Temperature variation of the magnetic properties of mechanochemically synthesized $(\text{Fe}_2\text{O}_3)_{1-x}(\text{Al}_2\text{O}_3)_x$ solid solutions through the Mössbauer spectroscopy

Dong Luo¹, Hayato Nakaishi¹, Takeshi Yabutsuka¹, Shinji Kitao², Makoto Seto² and Shigeomi Takai^{1,†}

¹Graduate School of Energy Science, Kyoto University, Yoshida-Honmachi, Sakyo-ku, Kyoto 606–8501, Japan

²Institute for Integrated Radiation and Nuclear Science, Kyoto University, Asashiro-Nishi, Kumatori-cho, Sennan-gun, Osaka 590–0494, Japan

Mössbauer spectroscopy experiments were carried out on corundum-type structured $(\text{Fe}_2\text{O}_3)_{1-x}(\text{Al}_2\text{O}_3)_x$ solid solutions ($x = 0\text{--}0.67$) synthesized by the mechanical alloying method to clarify the contribution of temperature and aluminum concentration to the magnetic properties. The Mössbauer spectra at room temperature gradually change from a sextet (weak ferromagnetism) to a doublet (paramagnetism) with increasing aluminum contents. As the temperature decreases down to 10 K, the sextet component develops for lower aluminum substitution, while the doublet profile changes to sextet for higher aluminum region. The absolute values of quadrupole splitting for the weak ferromagnetic component slightly increase with the aluminum substitution, primarily due to the reduction of the symmetry of the crystal field. The isomer shift δ decreases with the aluminum concentration due to the shrinkage of the lattice, and it also decreases with increasing temperature for the enhanced lattice vibration. Additionally, the hyperfine magnetic field B_{hf} weakens with the aluminum substitution presumably due to a diminished interaction between the nuclear magnetic moments, which can be observed in the lower temperatures.

Key-words : Hematite, Mössbauer spectra, Solid solutions, Temperature variation, Magnetic properties

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1. Introduction

$\alpha\text{-Fe}_2\text{O}_3$ (hematite) attracts great interest from the scientific and technological points of view as its abundance and environmentally friendly nature in the past decades.^{1–4} While $\alpha\text{-Fe}_2\text{O}_3$ is a promising candidate for anode material of lithium-ion battery (LIB) due to its low cost, high theoretical capacity ($1007\text{ mA}\cdot\text{h}\cdot\text{g}^{-1}$), in addition to environmental friendliness,^{5–12} it undergoes significant volume changes during the charge/discharge process, which lead a poor cycle stability.^{10–14} Although $\alpha\text{-Al}_2\text{O}_3$ (corundum) is appropriate to stabilize the electrode structure by forming a solid solution with $\alpha\text{-Fe}_2\text{O}_3$ due to their structural similarity and inertness in electrode reactions, its solubility range is strictly limited around the end-member compositions.^{15,16} Despite extensive efforts to extend the solid solution range, a wide spreading intermediate composition range ($0.3 < x \leq 0.75$) has still been in the two-phase coexistence.^{17–20}

In recent years, we have successfully synthesized corundum-type solid solution $(\text{Fe}_2\text{O}_3)_{1-x}(\text{Al}_2\text{O}_3)_x$ across the entire compositional range using the mechanical alloy-

ing technique starting from $\gamma\text{-Fe}_2\text{O}_3$ and $\gamma\text{-Al}_2\text{O}_3$.^{10,21,22} X-ray diffraction confirmed the formation of single-phase solid solutions from the lattice parameter variations consistent with Vegard's law.¹⁰ To further characterize the obtained solid solutions, high-resolution transmission electron microscopy (HR-TEM) was performed, revealing typical grain sizes of 10–20 nm with the corresponding lattice spacing to the composition.²¹ Extended X-ray absorption fine structure (EXAFS) measurements indicated that increasing aluminum substitution led to a decrease in the radial distribution of Fe–Fe distances, while the Fe–O distances are not primarily altered.²¹ Additionally, the low-temperature heat capacity measurements demonstrated the influence of aluminum substitution and suggested the heat capacity anomaly below 25 K.²² The neutron diffraction revealed the temperature and compositional dependence of lattice and magnetic structures of $(\text{Fe}_2\text{O}_3)_{1-x}(\text{Al}_2\text{O}_3)_x$.²³

We have also carried out the room-temperature Mössbauer spectroscopy on this system and reported that the spectra changed from a sextet of a weak ferromagnetic state into a doublet of paramagnetic with substituting aluminum ions.²¹ We thought that the substitution of non-magnetic aluminum ions would diminish the spin interactions between iron ions forming the paramagnetic states. In terms of the temperature variation of the magnetic states

[†] Corresponding author: S. Takai; E-mail: stakai@energy.kyoto-u.ac.jp

for pristine $\alpha\text{-Fe}_2\text{O}_3$, reducing temperature, the Néel transition from paramagnetic to weak ferromagnetic phase occurs at 955 K, below which the magnetic spin aligns close to the basal plane of the hexagonal lattice.^{23–27} Then, it flips to antiparallel along the c -axis, forming an anti-ferromagnetic phase at 263 K (Morin transition).^{28,29} The temperature-dependent Mössbauer spectroscopy would provide a deeper understanding of the spin interaction of the newly found $(\text{Fe}_2\text{O}_3)_{1-x}(\text{Al}_2\text{O}_3)_x$ solid solutions, in addition to clarifying the phase relation of the solid solution system.

The present study aims to explore the magnetic properties in the $(\text{Fe}_2\text{O}_3)_{1-x}(\text{Al}_2\text{O}_3)_x$ solid solutions, emphasizing the effects of temperature variation and spin dilution caused by aluminum substitution. The temperature and compositional contributions are discussed based on some deduced magnetic parameters, including hyperfine magnetic field (B_{hf}), isomer shift (δ), and quadrupole splitting (ε).

2. Experimental

$(\text{Fe}_2\text{O}_3)_{1-x}(\text{Al}_2\text{O}_3)_x$ solid solutions were synthesized through the high-energy ball milling method as described previously.^{10,21,22} $\gamma\text{-Fe}_2\text{O}_3$ (Sigma Aldrich, 99+ %) and $\gamma\text{-Al}_2\text{O}_3$ (Strem Chem., 96 %) reagent powders were mixed in stoichiometric ratios and placed into a silicon nitride milling vessel, along with ten Si_3N_4 milling balls (10 mm in diameter) without any solvents. A planetary ball mill (Fritsch, Premium Line P-7) was operated at 800 rpm was employed in segmented intervals for a total of 240 min of milling (12 cycles for 20 min of operation). X-ray diffraction (Rigaku, Ultima IV) with $\text{CuK}\alpha$ radiation (40 kV, 40 mA) was used to confirm the formation and crystallinity of the solid solution within the 2θ range of 20 to 80°.

For Mössbauer spectroscopy, 25 mg of $(\text{Fe}_2\text{O}_3)_{1-x}(\text{Al}_2\text{O}_3)_x$ solid solution powder was mixed with boron nitride and polyethylene, then molded into a thin pellet with a 10 mm diameter. Mössbauer spectroscopy was performed in conventional transmission mode using a ^{57}Co in Rh γ -ray source (nominal activity of 1.85 GBq) at various temperatures (10, 20, 40, 77, 100, 200, and 300 K). Liquid helium and liquid nitrogen were employed as coolants below and above 77 K, respectively. The Doppler velocity scale was calibrated using an iron foil reference, and the spectral profiles were analyzed in Velocity Buffer File (VBF) mode. The fitting process was carried out by RECOIL program employing Voigt-based fitting function,^{30,31} to yield isomer shift, hyperfine magnetic field, and quadrupole splitting.

3. Results and discussion

Figure 1 represents the X-ray diffraction patterns of the $(\text{Fe}_2\text{O}_3)_{1-x}(\text{Al}_2\text{O}_3)_x$ solid solutions with $x = 0\text{--}0.67$ synthesized through mechanical alloying. All diffraction patterns exhibit the single-phased corundum-type structure, and the diffraction peaks shift toward higher 2θ angles with the aluminum substitution, which is ascribed to the substitution of smaller aluminum ions instead of iron. This behavior is consistent with the previous study.¹⁰

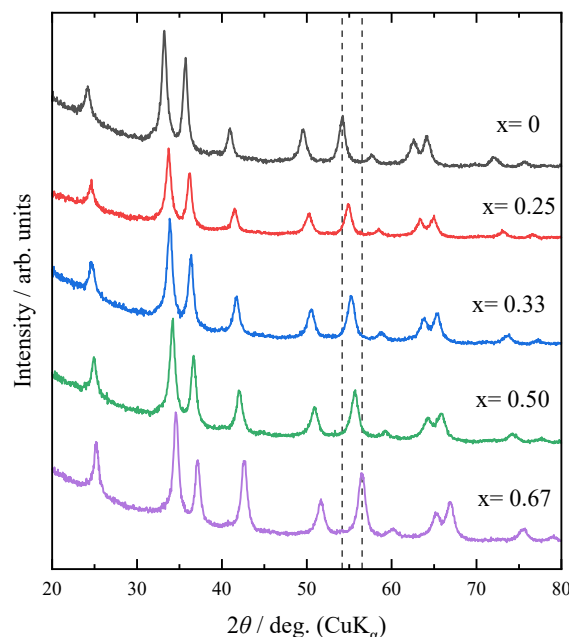


Fig. 1. X-ray diffraction patterns for mechanically alloyed $(\text{Fe}_2\text{O}_3)_{1-x}(\text{Al}_2\text{O}_3)_x$ solid solutions. Two dashed lines emphasize the peak shift of the (116) reflection for $x = 0$ and 0.67.

Figure 2 shows the measured Mössbauer spectra of $(\text{Fe}_2\text{O}_3)_{1-x}(\text{Al}_2\text{O}_3)_x$ solid solutions in the temperature range from 10 to 300 K. For the ball-milled sample of $x = 0$ (a), the Mössbauer spectrum at room temperature exhibits a sextet pattern, which agrees with the reported $\alpha\text{-Fe}_2\text{O}_3$ reagent,³² although the peaks show a smaller intensity with broadened shape. This can be attributed to the significantly smaller particle size of the samples synthesized via ball milling,³³ and the possible lattice defects introduced at the milling to disrupt spin interactions.^{34,35} As the temperature decreases, the peak profiles of the Mössbauer spectrum of $x = 0$ become distinct, presumably due to the decrease in thermal fluctuation of magnetic spins.^{35–37} Then, the sextet pattern persists in the measured temperature range for $x = 0$.

Comparing the room-temperature spectra of the $(\text{Fe}_2\text{O}_3)_{1-x}(\text{Al}_2\text{O}_3)_x$ solid solutions in Figs. 2(a)–2(e), the profile gradually changes from sextet to doublet with aluminum substitution. As reported in the previous paper,²¹ this profile change is predominantly attributed to the dilution of spin interactions for the non-magnetic aluminum substitution.^{38,39} As temperature decreases down to 10 K, the highly aluminum-substituted samples such as $x = 0.50$ or 0.67 show the gradual change in spectra from almost doublet to sextet profile. This transition is thought to result from the suppression of thermal fluctuation of spin orientation at lower temperatures, facilitating the stabilization of a certain ordered magnetic state. For intermediate compositions as $x = 0.25$ and 0.33, the Mössbauer spectra exhibiting a coexistence of doublet and sextet profiles at room temperature show the gradual change into a single sextet profile with decreasing temperature. The peak profiles of sextet at 10 K appear to be asymmetric shape, which

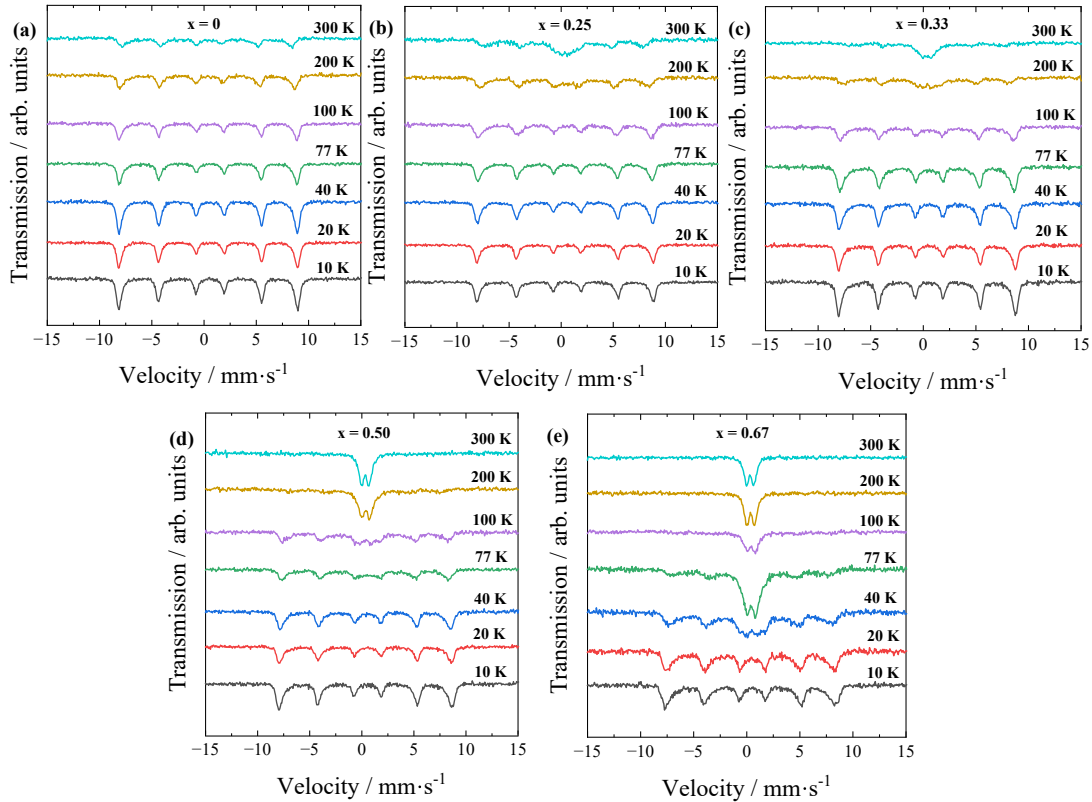


Fig. 2. Mössbauer spectra of the mechanochemically synthesized $(\text{Fe}_2\text{O}_3)_{1-x}(\text{Al}_2\text{O}_3)_x$ solid solutions. (a) $x = 0$, (b) 0.25, (c) 0.33, (d) 0.50, and (e) 0.67 measured at variable temperatures.

becomes apparent for the highly aluminum-substituted samples. This might be due to the increased distribution of magnetic local structures by the enhanced ratio of the non-magnetic aluminum ions.

The measured Mössbauer spectra were fitted by a combination of doublet and sextet profiles to yield quadrupole splitting ε , isomer shift δ , and hyperfine magnetic field B_{hf} . Representative fitting procedure for $x = 0.5$ is shown in Fig. 3. While single doublet and sextet profiles were applied to the data collected at 300 K (a) and 10 K (c), respectively, a combination of doublet and sextet was applied to those at 40 K (b). As observed in Figs. 3(b) and 3(c), the peak profile of sextet seems to be an asymmetric shape, although we fitted the peak by using a single Voigt function for simplicity. The parameters obtained through the fitting are listed in Table 1.

Figure 4 presents the compositional dependence of the quadrupole splitting for $(\text{Fe}_2\text{O}_3)_{1-x}(\text{Al}_2\text{O}_3)_x$ at various temperatures derived from (a) the sextet and (b) doublet components. The negative values of ε for the sextet component over the measured range suggest that the Morin transition disappears, and the weak ferromagnetic state persists down to 10 K, and the Morin transition disappears.^{35,40)} Considering that the antiferromagnetic phase is absent even in $x = 0$, this behavior would be due to the smaller particle size caused by the mechanical alloying process. Since the Morin transition temperature reduces with decreasing particle size,⁴¹⁾ 10–20 nm in size of the present samples²¹⁾ is sufficiently small to keep the weak ferromagnetism. It

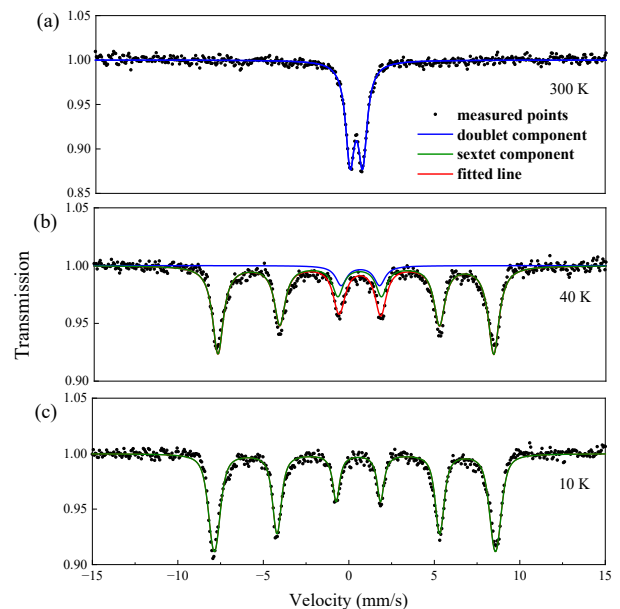


Fig. 3. Observed and fitted of Mössbauer spectra for the synthesized $(\text{Fe}_2\text{O}_3)_{1-x}(\text{Al}_2\text{O}_3)_x$ solid solution ($x = 0.5$) using RECOIL software, (a) 10 K, by sextet, (b) 40 K, by overlapped with sextet and doublet, and (c) 300 K, by doublet.

should be noted that the absolute value of ε slightly increases with the aluminum substitution. Substituting iron sites with the non-magnetic aluminum ions randomly, the symmetry of the crystal field decreases to enhance the

Table 1. Quadrupole splitting (ε), isomer shift (δ) and hyperfine magnetic field (B_{hf}) obtained by fitting Mössbauer spectra

x	Temperature (K)	ε ($\text{mm}\cdot\text{s}^{-1}$)		δ ($\text{mm}\cdot\text{s}^{-1}$)		B_{hf} (T)
		Sextet	Doublet	Sextet	Doublet	
0	10	−0.1750(38)		0.4893(38)		52.751(34)
	20	−0.1849(42)		0.4916(42)		52.656(37)
	40	−0.1840(41)		0.4944(41)		52.518(36)
	77	−0.1883(52)		0.4865(52)		52.265(46)
	100	−0.1961(75)		0.4850(75)		52.308(66)
	200	−0.1877(12)		0.4318(12)		51.112(11)
	300	−0.2218(20)		0.3881(20)		49.264(18)
0.25	10	−0.1971(52)		0.4779(52)		52.106(45)
	20	−0.1950(57)		0.4715(57)		52.072(50)
	40	−0.2040(56)		0.4743(56)		51.734(50)
	77	−0.2042(71)		0.4735(70)		51.283(62)
	100	−0.2041(14)		0.4780(14)		50.858(12)
	200	−0.2218(29)	1.8257(12)	0.4059(31)	0.5449(68)	49.157(20)
	300	−0.3564(10)	0.8738(13)		0.3253(13)	47.124(70)
0.33	10	−0.1919(50)		0.4685(49)		51.755(39)
	20	−0.2156(59)		0.4651(59)		51.643(46)
	40	−0.2089(64)		0.4653(64)		51.284(51)
	77	−0.2176(78)		0.4608(78)		50.637(63)
	100	−0.1920(16)	2.217(13)	0.4469(16)	0.5970(72)	50.517(11)
	200		1.2279(38)		0.4712(66)	48.608(37)
	300		0.9388(49)		0.3558(26)	46.080(54)
0.50	10	−0.2028(71)		0.4561(71)		50.973(57)
	20	−0.2232(91)		0.4551(91)		50.665(72)
	40	−0.2224(11)	2.2542(12)	0.4516(11)	0.6055(64)	50.129(76)
	77	−0.2449(23)	1.6410(10)	0.4459(24)	0.5132(58)	48.821(16)
	100	−0.2515(33)	1.3416(85)	0.4352(35)	0.4153(51)	48.059(23)
	200		0.8416(24)		0.3929(15)	
	300		0.739(15)		0.3445(93)	
0.67	10	−0.2299(16)		0.4380(50)		48.463(15)
	20	−0.2338(19)	1.0893(91)	0.4294(19)	0.5507(59)	47.828(12)
	40	−0.2535(27)	0.9934(22)	0.4164(25)	0.5158(18)	44.764(27)
	77	−0.2258(60)	0.9300(32)	0.4344(61)	0.4811(18)	43.975(71)
	100		0.8114(29)		0.4259(18)	
	200		0.7407(14)		0.3752(85)	
	300		0.6968(14)		0.3311(87)	

electric field gradient (EFG),⁴²⁾ resulting in an enhanced absolute value of ε . Since this compositional dependence is consistent across the entire temperature range, the contribution of the thermally activated spin fluctuation seems to be small.

In Fig. 4(b), the quadrupole splitting for the doublet shows a decreasing trend with the aluminum substitution in the temperature range 40–200 K. Considering that the doublet profile is characteristic of a paramagnetic component, higher aluminum substitution alters the local crystal field environment. In contrast to the sextet, the temperature dependence of ε from the doublet is significant. With decreasing temperature, spin fluctuation is suppressed to accentuate deviations from spherical symmetry, allowing the ε from the doublet component larger.

Figure 5 presents the compositional dependence of isomer shift δ deduced from the sextet profiles of $(\text{Fe}_2\text{O}_3)_{1-x}(\text{Al}_2\text{O}_3)_x$ solid solution. As the general trend, the isomer shift decreases with aluminum substitution. This can be attributed to the reduction in Fe–Fe interatomic distance, e.g. 2.87 Å ($x = 0$) to 2.75 Å ($x = 0.5$) for the nearest one,²³⁾ due to the substitution of smaller aluminum ions,

which densifies the electron cloud, especially the s -electrons near the iron atomic nucleus.¹⁰⁾ The nuclear shielding effect is then enhanced, which would lead to a decrease in the isomer shift.⁴³⁾ While the temperature dependence of δ is not apparent below 100 K, δ values obviously decrease with increasing temperatures above 100 K. This might be due to the increasing lattice vibration with temperature,^{44,45)} although the isomer shift from the sextet profile is available in the limited compositions.

Figure 6 plots the hyperfine magnetic field B_{hf} obtained at various temperatures. As for the compositional dependence, B_{hf} decreases with increasing aluminum content, and drops largely above $x = 0.5$. This behavior is applicable for all temperatures, and B_{hf} curve shifts lower with increasing temperature. In the previous room-temperature study,²¹⁾ the obtained data are restricted to the aluminum-poor region, although the entire behavior is obtained in the low-temperature measurements. At room temperature, 49 T of the deduced B_{hf} for $x = 0$ is smaller than that of the reported value of 52 T for $\alpha\text{-Fe}_2\text{O}_3$.⁴⁶⁾ Such a reduced hyperfine magnetic field is thought to be due to its smaller particle size and possibly induced lattice distortions.

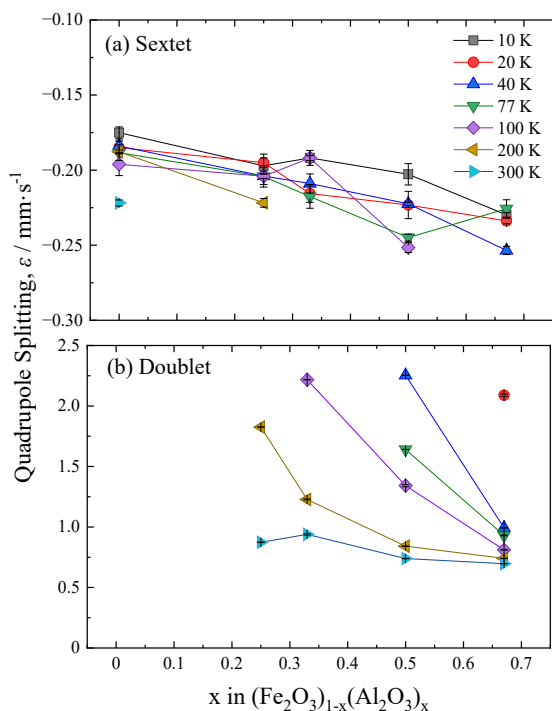


Fig. 4. Compositional dependence of the quadrupole splitting of $(\text{Fe}_2\text{O}_3)_{1-x}(\text{Al}_2\text{O}_3)_x$ under variable temperatures obtained from (a) sextet and (b) doublet component.

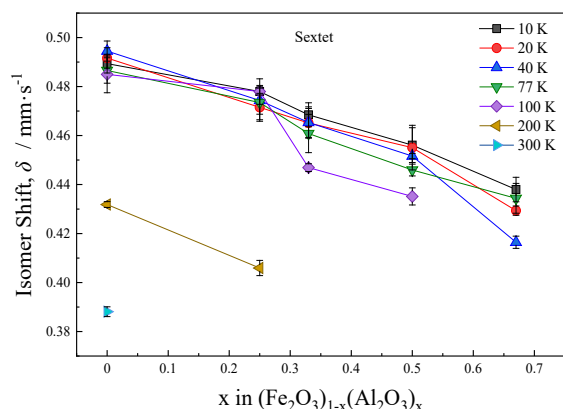


Fig. 5. Compositional dependence of isomer shift of $(\text{Fe}_2\text{O}_3)_{1-x}(\text{Al}_2\text{O}_3)_x$ solid solution obtained from the sextet component under variable temperatures.

tion.^{10,22,32,46)} In smaller particles, an enhanced proportion of the surface atoms weakens the spin interactions to reduce the hyperfine magnetic field, as reported for the milling time dependence of B_{hf} for hematite.⁴⁷⁾ In addition, when the thermal fluctuation of the magnetic moment becomes more pronounced in smaller particles above 100 K, the degree of the magnetic ordering is further reduced, leading to the overall reduction of the hyperfine magnetic field.^{47,48)}

Figure 7 represents the transient temperature from the weak magnetic to paramagnetic states, where the contributions of the sextet and doublet profiles in the Mössbauer spectra reach 50 % through the fitting. The paramagnetic state observed below room temperature is attributed to the

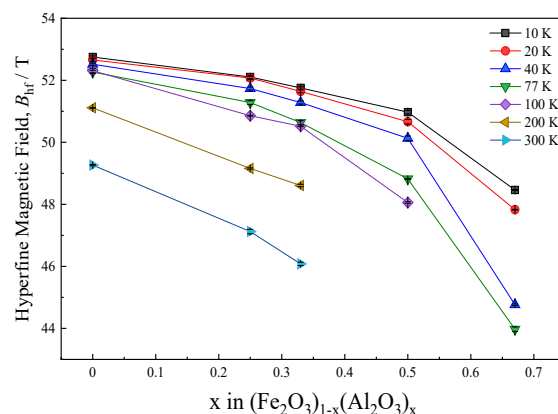


Fig. 6. Compositional dependence of the hyperfine magnetic field of $(\text{Fe}_2\text{O}_3)_{1-x}(\text{Al}_2\text{O}_3)_x$ under various temperatures.

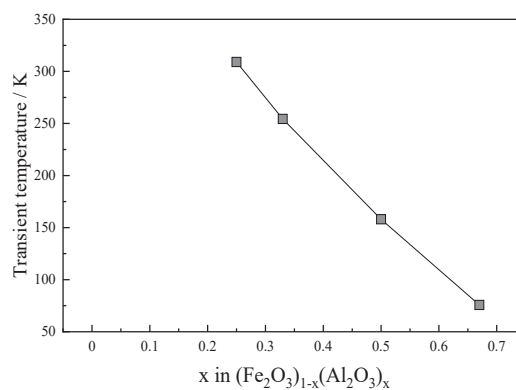


Fig. 7. Fifty percent contribution-temperature between doublet and sextet of Mössbauer spectrum for $(\text{Fe}_2\text{O}_3)_{1-x}(\text{Al}_2\text{O}_3)_x$.

dilution effect from aluminum substitution, which enables this gradual change in magnetic behavior even under weaker thermal fluctuation. With increasing aluminum substitution, the transient temperature decreases due to the disruption of the magnetic interactions in the Fe₂O₃ lattice. The reduction of magnetic moment with aluminum substitution was observed by the magnetic reflection of the neutron diffraction patterns.²³⁾ Unlike the Néel transition with cooperative phenomena, the change in the magnetic state from paramagnetic to weak ferromagnetic is rather diffused. The origin of the magnetic state transient is the reduction of averaged magnetic moment due to the aluminum substitution against the thermally activated spin fluctuation. Further analysis requires atomic simulation as future work.

In this study, we carried out a comprehensive study of Mössbauer spectroscopy under temperature and compositional variation. Through the low temperature measurements, spin interactions can be detected even at $x = 0.67$ by ignoring the effect of thermal fluctuation.

4. Conclusion

The magnetic properties of the $(\text{Fe}_2\text{O}_3)_{1-x}(\text{Al}_2\text{O}_3)_x$ solid solutions up to $x = 0.67$ were systematically investigated using temperature-dependent Mössbauer spectroscopy. As the temperature decreases from 300 to 10 K, the

Mössbauer spectra of the solid solutions gradually shift from a paramagnetic doublet to a weak ferromagnetic sextet. The quadrupole splitting of the sextet state exhibits an increase in absolute value with the aluminum substitution, which is not largely altered with temperature. On the other hand, those obtained from doublet show a large drop with aluminum substitution, which becomes notable with decreasing temperature. The isomer shift decreases with increasing aluminum substitution due to lattice contraction, although it reduces more significantly above 200 K for the lattice vibration. The hyperfine magnetic field diminishes progressively with increasing temperature, the trend of which becomes more pronounced with higher aluminum substitution. Substitution-induced dilution of spin–spin interactions reduces the magnetic moment, facilitating the disruption of the ferromagnetic state at a lower temperature. This study provides essential information about the magnetic properties of $(\text{Fe}_2\text{O}_3)_{1-x}(\text{Al}_2\text{O}_3)_x$ solid solutions and offers insights for future research.

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Author Contribution **Dong Luo:** Formal analysis, Writing – original draft, Writing – review & editing. **Hayato Nakaishi:** Formal analysis, Data curation. **Takeshi Yabutsuka:** Resources, Writing – review & editing. **Shinji Kitao:** Data curation, Formal analysis. **Makoto Seto:** Data curation. **Shigeomi Takai:** Conceptualization, Project administration, Resources, Supervision, Writing – review & editing.

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