

Effect of Mill-scale and Calcined Dolomite on High Al₂O₃ sinter and its Reduction Behaviour

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Keywords: Iron ore sintering, Al₂O₃, FeO, Mill-scale, MgO, Calcined dolomite

ABSTRACT

Al_2O_3 has been regarded as a harmful gangue component in iron ore, especially causing reduction degradation in industrial sintering of iron ore. The current study aimed to utilise low-grade iron ore containing high Al_2O_3 content in sintering process. In order to counteract the adverse effect of Al_2O_3 on reduction behaviour of iron ore sinter, mill-scale and calcined dolomite were chosen as FeO- and MgO-bearing materials. Firstly, mill-scale was added to high Al_2O_3 sinter and addition was optimised as 10 wt%, then calcined dolomite was added further. Magnetite and magnesio-ferrite were identified as the major phases in iron ore sinter when adding mill-scale and calcined dolomite. As the dosage of calcined dolomite increased beyond 4 wt% of in sinter mix, unreacted MgO particles were observed, which is ascribed to the limitation of MgO dissolution into magnetite matrix. Both mill-scale and calcined dolomite were effective in improving compressive load and Reduction Disintegration Index (RDI) of iron ore sinter although its reduction degree was highly decreased. The addition of both FeO and MgO-bearing materials to high Al_2O_3 sinter has a potential to improve the sinter quality, also contribute to the recycling of industrial by-product. However, the amount should be optimised in views of phase development and deterioration of reducibility.

INTRODUCTION

The price of raw materials being used in steel industry is gradually increasing in recent years. (Chai, 2019; Wang, 2020) Since several countries with limited mineral resource are largely depending on the import iron ores, ironmaking companies are attempting to substitute low-grade iron ores in their industrial process for high-grade iron ores. This urgent agenda is connected to the numerous studies (Yamaoka, 1974; Pimenta, 2002; Yang, 2005; Xue, 2020) in ironmaking research field to clarify the influence of gangue component on characteristics of iron ore sinter in sintering process where low-grade iron ores might be utilised.

Al_2O_3 has been known to be a representative of harmful gangue components affecting iron ore sinter. Several researchers (Yamaoka, 1974; Xiao, 2017) reported that Al_2O_3 increased the formation temperature of primary melt during the sintering process. Okazaki (2003) ported that Al_2O_3 inhibits the penetration of melt with high viscosity and suppresses assimilation between nuclei and adhering fines. This strongly affected the irregular pore distribution and further deteriorated the sinter strength, (Pimenta, 2002; Loo, 2003) which is inferior to the bed permeability of blast furnace. (Umadevi, 2014) Degradation of sinter matrix is also one of the critical problems when Al atom was entrapped in lattice of hematite and magnetite phase and resulted in rapid propagation of cracks (Sinha, 2009).

For counteracting the adverse effect of Al_2O_3 on iron ore sinter, several researchers (Yadav, 2002; Higuchi, 2004; Umadevi, 2012; Umadevi, 2013) considered the small addition of FeO and MgO. They found that increased FeO is directly connected to the magnetite (Fe_3O_4). Sinter volume was decreased during the reduction of hematite to magnetite, which caused the microstructure of sinter dense. (Biswas, 1981) This phenomenon is closely related to the improvement the overall sinter strength as shown by increase of Reduction Disintegration Index (Umadevi, 2012). Reduction behaviour of MgO in sintering process is similar to that of FeO because ionic radii of both cations are close. Panigrahy (1984) explained that MgO favours the formation of magnetite (Fe_3O_4) and Mg^{2+} ions replaced part of Fe^{2+} ions to stabilise the spinel structure. It implies that replacement of Fe^{2+} ions with Mg^{2+} ions can ensure the increase in excess Fe^{2+} content, which might contribute to the improvement of melting behaviour of iron ore sinter. (Iwanaga, 1982; Ono-Nakazato, 2002; Lee, 2004; Chuang, 2009)

In this study, combined addition of FeO- and MgO-bearing materials to sinter mix with high Al_2O_3 ore was investigated on the basis of reduction behaviour during sintering process. Mill-scale and calcined dolomite were selected to be the source of FeO and MgO, respectively. Mill-scale is FeO-based waste produced during hot-rolling, and it is regarded as reusable by-product in both ironmaking and steelmaking process. Dolomite contains both CaO and MgO so melting behaviour control as well as fluxing effect can be expected through addition to high Al_2O_3 sinter. Calcination of dolomite was conducted to exclude the endothermic reaction on reduction process. Mineralogical analyses by X-ray diffraction (XRD) and Scanned electron microscopy (SEM-EDS) were employed to figure out how the combined addition of mill-scale and calcined dolomite interact and affect to the sinter microstructure. Physical properties of sinters were measured by Compression test and Reduction Disintegration Index test to assess final sinter quality.

Materials preparation

All raw materials used in the study were supplied by POSCO, industrial steelmaking company (**Table 1**). To produce standard sinter mix being used in general ironmaking industrial factory, two kinds of iron ores were prepared: Ore A and Ore B. Ore A is Goethite, FeO(OH)-based ore with high SiO₂ content and Loss on ignition (LOI). Ore B is high-grade hematite-based iron ore, containing low gangue content. Unlike these two iron ores, Ore C, which is typical pisolitic ore, was used as an Al₂O₃ source because it has been classified as a high-Al₂O₃ ore in several ironmaking companies. Dolomite was calcined at 1000 °C before experiment.

TABLE 1 – Chemical composition of raw materials (wt%)

Raw materials	T.Fe	M.Fe	FeO	Al ₂ O ₃	CaO	SiO ₂	MgO	MnO	P ₂ O ₅	LOI
Ore A	57.00	0.07	0.17	1.59	0.06	5.39	0.08	0.02	0.08	10.38
Ore B	65.20	0.07	0.46	1.25	0.07	1.52	0.05	0.22	0.15	3.20
Ore C	57.10	0.06	0.20	3.14	0.04	5.35	0.05	0.03	0.14	8.59
Mill-scale	72.60	2.50	60.40	0.31	0.70	1.00	0.06	0.52	0.05	0.70
Calcined dolomite	-	-	-	-	56.19	-	38.78	-	-	2.53

To make uniform mixture, all samples were crushed under 50 µm and dried in oven for 24 hours. Ore A and Ore B were mixed with the mass ratio of 80 to 20 (Reference 1), which is simple blending of iron ores being typically utilised in actual ironmaking process. Reference 2 was prepared by mixing Ore A, Ore B and Ore C with the mass ratio of 48:12:40. Reference 2 has 2.31 wt% Al₂O₃ so that it is possible to compare it Reference 1 (1.76 wt% Al₂O₃) in terms of the adverse effect of Al₂O₃. Basicity, defined in this study as CaO/SiO₂, of two mixtures was adjusted to be 1.85 by adding a reagent-grade of CaO (purity 98%) while mill-scale and calcined dolomite were added without considering basicity. (**Table 2**) The sinter mixes were homogenised using a Turbula mixer for 3 hours and pressed into disk shape for 20 MP by a Hydraulic Work Press Machine.

TABLE 2 – Calculated chemical composition of sinter mix with addition of mill-scale and calcined dolomite (wt%)

Sample	T.Fe	M.Fe	FeO	Al ₂ O ₃	CaO	SiO ₂	MgO	MnO	P ₂ O ₅	CaO/SiO ₂
Reference 1	53.97	0.07	0.21	1.40	7.87	4.26	0.07	0.06	0.09	1.85
Reference 2	52.71	0.06	0.19	2.28	8.56	4.63	0.05	0.04	0.11	1.85
Mill5	53.66	0.17	3.06	2.19	8.18	4.45	0.05	0.06	0.11	1.85
Mill10	54.52	0.28	5.67	2.10	7.84	4.30	0.06	0.08	0.11	1.85
Mill15	55.30	0.38	8.05	2.02	7.53	4.15	0.06	0.10	0.10	1.85

Mill10-Dol2	53.54	0.28	5.57	2.06	8.64	4.22	0.70	0.08	0.10	2.05
Mill10-Dol4	52.61	0.27	5.47	2.02	9.41	4.15	1.33	0.08	0.10	2.27
Mill10-Dol6	51.70	0.27	5.37	1.99	10.16	4.07	1.93	0.08	0.10	2.50

Sintering process

A disk-shaped sinter mix was sintered in a vertical tube furnace. That is, it was sintered while heating for 7 min in a furnace at 1280 °C by preliminary experiment to optimise the fraction of SFCA phase. SFCA is abbreviation of Silico-Ferrite of Calcium and Aluminium, which is bonding phase in iron ore sinter as complex form of Calcium ferrite ($\text{CaO}\cdot\text{Fe}_2\text{O}_3$). This phase is known as desirable for high reducibility and sinter strength of iron ore sinter (Nicol, 2018). After heating, the sinter was cooled down inside the furnace for 30 min. Heating was conducted in an atmosphere consisting of CO, CO₂, and Ar with the ratio of 2:23:75 to maximise the formation of bonding phases according to Hsieh and Whiteman (1989).

Materials Characterization

All sinters were crushed to 500 µm and analysed by X-ray diffraction (XRD) using wavelength of 1.63Å (Cu-Kα) in the scanning range of 20 to 80 degree. Then, the sinter was mounted by cold resin and polished up to 1 µm grain size to employ Scanning electron microscopy (SEM). Elemental Dispersive (EDS) X-ray spectrometry was carried out to evaluate the elemental composition of each phase detected by XRD.

To assess the reduction behaviour of sinter, Compression Test and Reduction Disintegration Index (RDI) Test were performed. Compression Test was executed by Instron M5548 machine to characterise the superficial strength of sinter, measuring the maximum compressive load after gradual compression (Zhang, 2015). RDI Test was performed based on the international standard (ISO 4696 Part 2, 2015) Ten disk-shaped sinters were heated at 550 °C for 30 min in an atmosphere of 30 vol% CO and 70 vol% N₂ in a gold image furnace. After heating, the sinters were rotated inside Turbula Mixer at 60 rpm for 15 min. RDI_{2.8} was evaluated based on the weight of sinters retained on the 2.8 mm sieve. Reduction degree was calculated to describe the reducibility of a sinter based on ISO 7215:2015 where the sinter was heated at 900 °C for 3 hours in an atmosphere 30 vol% CO and 70 vol% N₂ gas atmospheres in a gold image furnace.

RESULTS

Effect of adding mill-scale to sinter mix containing high Al₂O₃ ore on sinter quality

Mill-scale was added to Reference 2 up to 15 wt% to evaluate the effect of FeO-bearing materials on sinter mix containing high Al₂O₃. Three phases were mainly identified by X-ray diffraction (**Figure 1**): hematite (Fe₂O₃), magnetite (Fe₃O₄) and SFCA ($\text{Ca}_{2.3}\text{Mg}_{0.8}\text{Al}_{1.5}\text{Fe}_{8.3}\text{Si}_{1.1}\text{O}_{20}$). Existence of high Al₂O₃ ore in the sinter did not show a meaningful difference in case XRD patterns of Reference 1 and Reference 2 were compared. As two sinters showed similar XRD patterns, iron ore sinter manufactured by blending high Al₂O₃ ore in this study is under acceptable range in a view of phase development. This is connected to the previous study that low Al₂O₃ content in the sinter showed positive effect on SFCA formation (Scarlett, 2004), rather than contribution to the formation of slag phase. Hematite was dominant among the phases of all the sinters while magnetite was drastically increased as 10 wt% of mill-scale was added to Reference 2 (Mill10), eventually exceeding the hematite at 15 wt% addition (Mill15). Although hematite and magnetite shared the same peak at 33~34 degree and 62.5~63.5 degree in the scanning range, the portion of magnetite in the peak intensity is dominant in Mill15. In addition, the fraction of SFCA was barely changed up to Mill10, but

it was decreased at Mill15. FeO in mill-scale is helpful to form bonding phases combined with Ca, Si, and Al, but excess Fe^{2+} prevents the formation of SFCA because reducing atmosphere is induced during sintering process (Wang, 2016). At reducing atmosphere, FeO dissolves into Fe_2O_3 to form magnetite. From reason that phase composition with sufficient SFCA and hematite phase is maintained, mill-scale addition can be optimised as 10 wt% into high Al_2O_3 sinter.

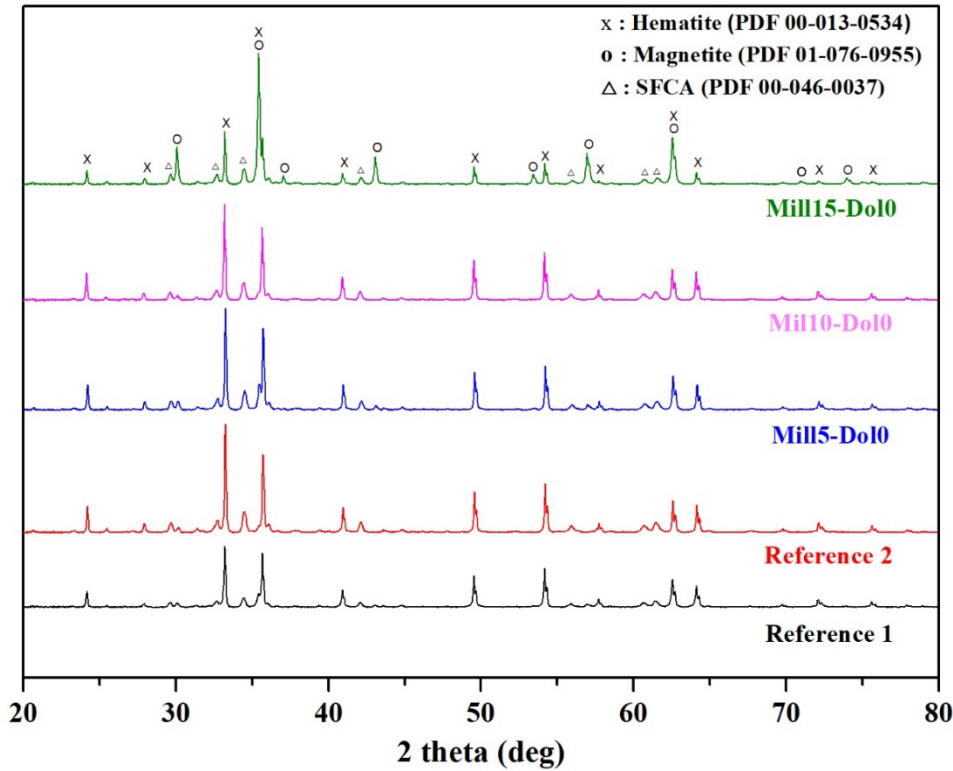


FIG 1 – Phase identification of the sinters to which mill-scale was added by X-ray diffraction

Figure 2 is the backscattered electron images of the sinters with increasing added amount of mill-scale, and EDS results of the selected points at **Figure 2** is shown in **Table 3**. White grains composed of hematite occupied the sinter matrix (Point 1, 3, 5, 7) and grey SFCA were partly observed as the acicular form (Point 4, 6, 8) or columnar form (Point 2, 6, 10). When 5 wt% and 10 wt% of mill-scale was added, sinters maintained their microstructure, and the ratio of hematite and SFCA was consistent from Reference 2. Meanwhile, the fraction of Fe was largely increased at Point 9 in Mill15, indicating that magnetite possessed more area than hematite. Acicular and columnar texture were decomposed as more Fe was diffused into magnetite matrix, and the boundaries between magnetite and SFCA were observed to be blurred. It is clearly indicated at Point 10, which portion of Fe in SFCA phase was notably increased compared to that at Point 6 and Point 8. It is not recommended for overall sinter quality, since the irregular morphology of SFCA is detrimental for reducibility and cold strength of iron ore sinter (Ying, 2006; Takayama, 2018).

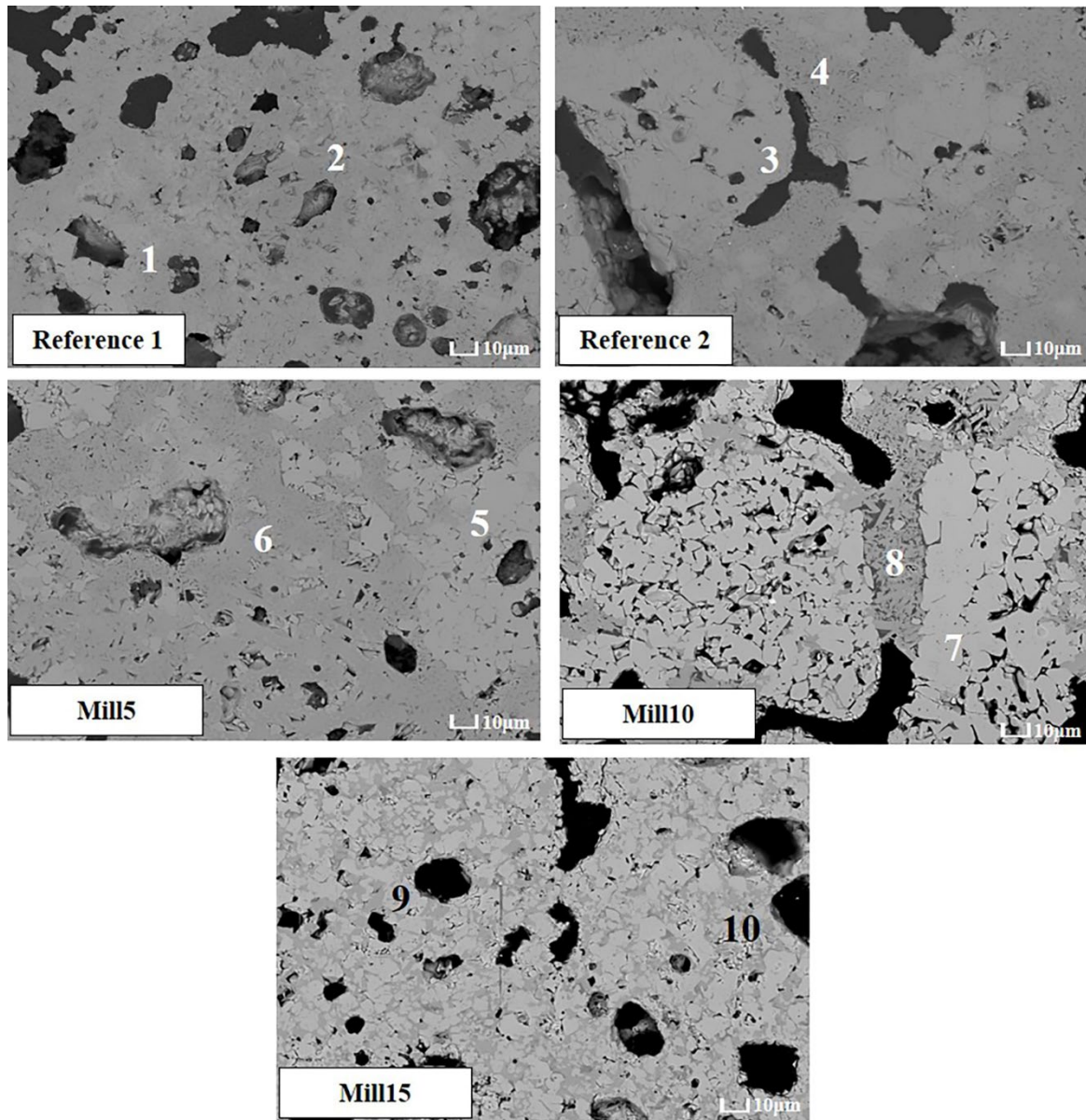


FIG 2 – Morphological features of the sinters to which mill-scale was added

TABLE 3 – Elemental distribution of selected points in the sinters to which mill-scale was added (at%)

Sinters	Pts.	Elemental composition						Identified phases
		Fe	Ca	Si	Al	Mg	O	
Reference 1	1	37.15	0.14	0.13	0.49	-	62.09	Fe ₂ O ₃
	2	33.81	4.10	2.69	1.87	-	57.53	SFCA
Reference 2	3	37.93	0.16	0.05	0.55	-	61.23	Fe ₂ O ₃
	4	26.17	7.95	3.29	2.42	-	59.86	SFCA
Mill5	5	34.93	2.29	0.91	0.48	-	61.39	Fe ₂ O ₃
	6	29.74	8.45	3.45	3.15	-	55.26	SFCA

Mill10	7	36.89	0.51	0.31	1.43	-	60.85	Fe ₂ O ₃
	8	31.34	7.75	3.29	2.35	-	55.29	SFCA
Mill15	9	42.28	0.05	0.12	0.38	-	57.16	Fe ₃ O ₄
	10	23.79	8.34	5.26	3.71	-	58.91	SFCA

Figure 3a depicts the reduction behaviour of the sinters as mill-scale was added to the sinter mix. Reduction degree was slightly decreased as high Al₂O₃ ore was blended, which is affected by Al₂O₃ content (Umadevi, 2009; Sinha, 2017). In case mill-scale was added to sinters, reduction degree was firstly increased at Mill5, but decreased then. Even it showed lower reducibility than that of Reference 2. It is obvious that FeO acted as a strong factor affecting the decrease in the reduction degree of sinters by increasing portion of magnetite in the sinter. These can be also applied into the change in compressive load and RDI_{-2.8} of the sinters as shown in **Figure 3b**. For Reference 2, compressive load was decreased while RDI_{-2.8} was increased compared to those of Reference 1. Both trends showed clear improvement after adding mill-scale. Several researchers have already reported that magnetite has a potential to prevent the low-temperature degradation (Umadevi, 2012; An, 2022), so adding mill-scale into high Al₂O₃ sinter can be the solution of reduction degradation.

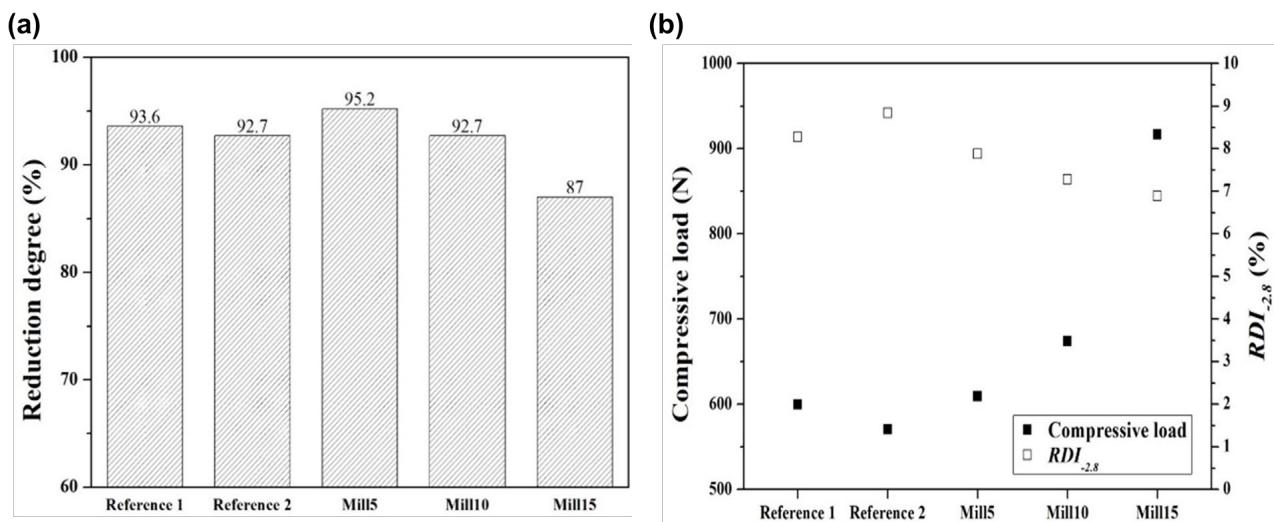


FIG 3 – (a) Change in reduction degree of the sinters to which mill-scale was added (b) Change in strength and disintegration behaviour of the sinters to which mill-scale was added

Combined effect of adding mill-scale and calcined dolomite to sinter mix containing high Al₂O₃ ore on sinter quality

Based on the obtained results in the previous section, calcined dolomite was added to the sinter mix (Mill10) up to 6 wt%. As shown in **Figure 4**, three major phases were identified as similar to the case in **Figure 2**, however, magnesio-ferrite (MgO·Fe₂O₃) was mainly observed instead of magnetite at identical peaks. It is required to be considered that several Mg²⁺ ions diffused into magnetite, and replaced the Fe²⁺ sites since the cations of Mg²⁺ and Fe²⁺ have the similar ionic radius. (Panigrahy, 1984) In addition, the fraction of SFCA was maintained at Mill10-Dol2, but it was gradually decreased as more than 4 wt% of calcined dolomite was added (Mill10-Dol4). Magnesio-ferrite was increased and became dominant at Mill10-Dol6. The portion of magnesio-ferrite was largest among all other sinters in this study although the total amount of FeO and MgO was lower than that in Mill15. That is, the effect of adding calcined dolomite on the stabilization of this magnesio-spinel structure was more remarkable than that of adding mill-scale.

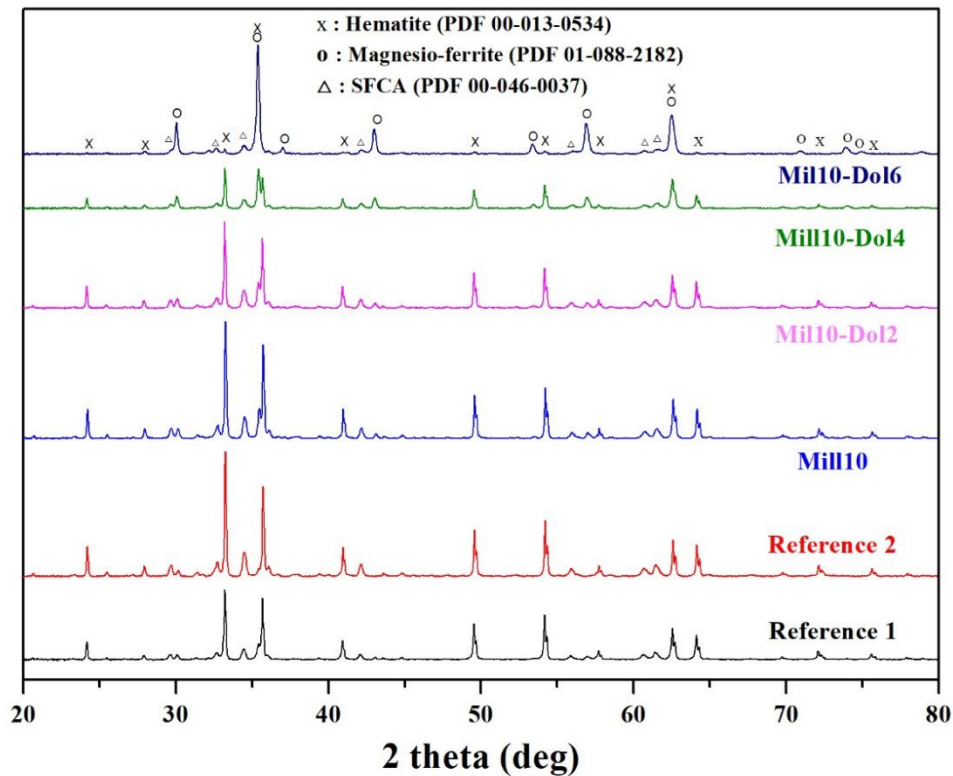


FIG 4 – Phase identification of the sinters to which mill-scale and calcined dolomite were added by X-ray diffraction

Figure 5 shows the backscattered electron images of the sinters when mill-scale and calcined dolomite were added. Mg was found to be distributed in Fe-enriched regions of all the sinters containing calcined dolomite. Mill10-Dol2 showed similar morphology to that of the sinter not containing dolomite (Mill10), but residual MgO in hematite matrix was concentrated at Mill10-Dol4 and Mill10-Dol6, which was indicated at Point 8. Residual MgO was surrounded by magnesio-ferrite (Point 9) and calcium silicate (Point 10) while other regions without MgO segregation showed typical microstructure consisting of magnesio-ferrite (Point 5) and SFCA (Point 7). Higuchi (2007) found similar phenomenon that rim structure consisting of magnetite solid solution was formed by the interaction between MgO and hematite or calcium ferrite, which is similar to the morphology of Mill10-Dol6 in Figure 5. Also, Al_2O_3 preferred to be absorbed into SFCA (Point 2, 4, 7) except Point 10. As addition of calcined dolomite was expanded, more Al_2O_3 became remained in Calcium silicate and caused the increase of slag phase.

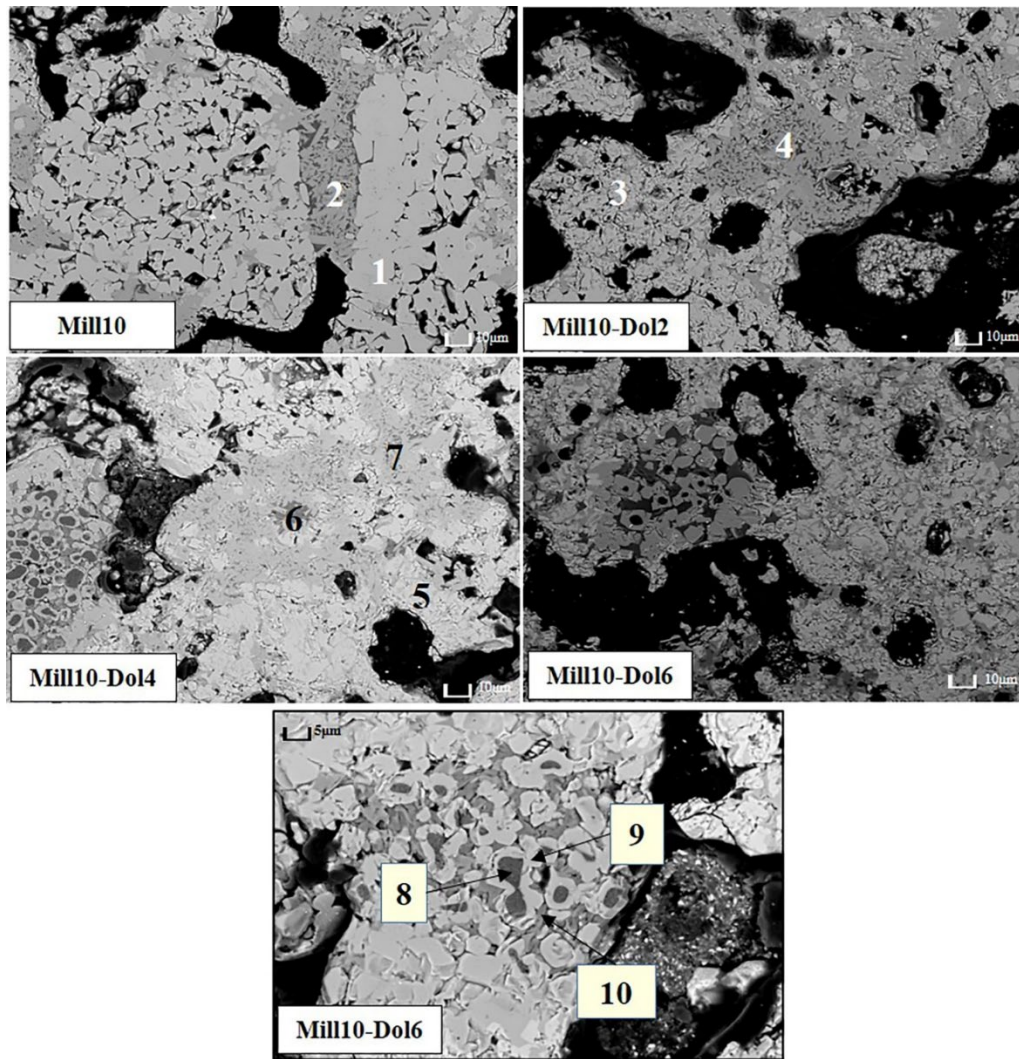


FIG 5 – Morphological features of the sinters to which mill-scale and calcined dolomite were added

TABLE 4 – Elemental distribution of selected points in the sinters to which mill-scale and calcined dolomite were added (at%)

Sinters	Pts.	Elemental composition						Identified phases
		Fe	Ca	Si	Al	Mg	O	
Mill10	1	36.89	0.51	0.31	1.43	-	60.85	Fe ₂ O ₃
	2	31.34	7.75	3.29	2.35	-	55.29	SFCA
Mill10-Dol2	3	37.88	1.32	0.25	0.89	0.94	58.72	Fe ₂ O ₃
	4	23.71	7.77	4.63	2.47	0.25	61.16	SFCA
Mill10-Dol4	5	26.13	0.81	0.19	1.37	12.28	59.21	Fe ₂ O ₃ , MgO·Fe ₂ O ₃
	6	1.47	21.31	14.88	0.50	0.12	61.73	2CaO·SiO ₂ , CaO·SiO ₂
	7	25.81	7.43	3.50	2.93	0.54	59.80	SFCA

Mill10-Dol6	8	4.47	0.25	0.19	0.08	44.13	50.88	MgO
	9	28.08	0.72	0.21	2.27	12.09	56.63	MgO·Fe ₂ O ₃
	10	3.95	14.84	12.32	3.09	4.03	61.77	CaO·SiO ₂

Figure 6a and **Figure 6b** show relationship between portion of magnetite and sinter strength of sinters when calcined dolomite was added together with mill-scale. There was no significant difference in reduction degree when calcined dolomite was added 2 wt% into Mill10, but it was declined from 4 wt% addition. Reduction degree in Mill10-Dol6 was calculated as 69.6%, which is not suitable in industrial sintering process (Umadevi, 2014). Both compressive load and RDI_{-2.8} were worsened compared to those of Mill10, but it changed to be improved along the calcined dolomite addition. Calcined dolomite seems to be acted as same as mill-scale to improve sinter strength and reduction degradation of high Al₂O₃ sinter, but further explanation is required to determine the individual role of calcined dolomite separated from that of mill-scale.

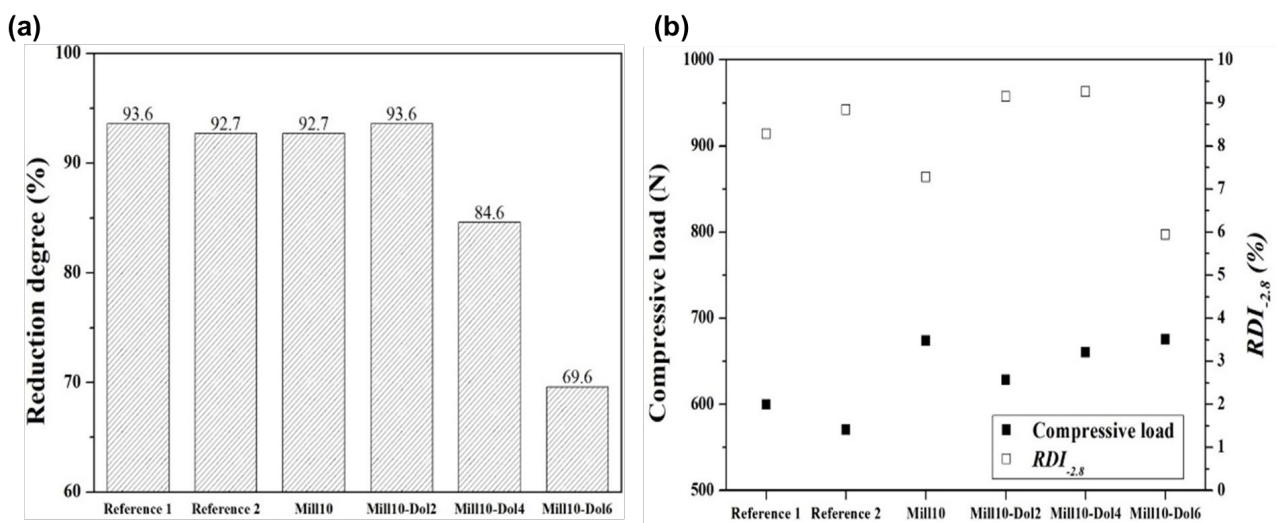


FIG 6 – (a) Change in reduction degree of the sinters to which mill-scale and/or calcined dolomite were added (b) Change in strength and disintegration behaviour of the sinters to which mill-scale and/or calcined dolomite were added

DISCUSSION

To clarify the mechanism of phase development of high Al₂O₃ sinter when mill-scale and calcined dolomite was added, the elemental distribution of Mill10-Dol6 was specified by EDS mapping as shown in **Figure 7**. The clusters consisting of Mg were surrounded by reddish part comprising Mg and Fe. MgO remained unreacted rather than forming magnesio-ferrite in the sintering process in case the large amount of calcined dolomite was added. It is clear that there is certain limitation in the dissolution of MgO into magnetite matrix. Or the relict MgO with high sintering temperature will block the further assimilation of bonding phases, further act as harmful to overall sinter qualities such as reducibility and toughness (Han, 2019; Shi, 2020). In case of Ca and Si, they were locally concentrated around MgO segregation and formed slag phase indicated by blue part. Excessive CaO supplied by calcined dolomite did not react with Fe to form Calcium Ferrite or SFCA. Instead, calcium silicate was formed in microstructure, so this slag phase would affect to the poor reducibility of high Al₂O₃ sinter.

Physical properties of high Al₂O₃ sinter with addition of mill-scale and calcined dolomite also can be explained based on phase development after iron ore sintering. Al was found to be homogeneously distributed in whole microstructure, respectively. It is desirable in terms of reduction degradation, since Al was reported to be entrapped in hematite matrix during reduction of hematite to magnetite (Sinha, 2009; Umadevi, 2009) to induce lattice distortion. Since Al₂O₃ exists in between the lattices

of hematite and magnetite and leads rapid crack propagation (Sinha, 2009), even distribution of Al in microstructure has an advantage on relaxing this crack propagation by forming dense matrix (Umadevi, 2012). It is similar to the result of Zhu *et al.* (2020), who found the decreased crack density of iron ore sinter to which MgO content was increased. MgO generally decreases the porosity of sinter (Umadevi, 2014) by stabilizing the spinel structure of magnetite so called magnesio-spinel (Panigrahy, 1984). Magnesio-spinel phase changes the magnetite matrix dense to resist against external pressure, further contributes to the improvement of compressive strength of iron ore sinter. This effect is dominant in this study, showing the increase of compressive load of iron ore sinter when calcined dolomite was utilised. However, MgO segregation should be considered in detail when controlling the addition of MgO-bearing material into high Al₂O₃ sinter. The portion of the phases where Al₂O₃ can diffuse would be decreased if the portion of MgO clusters is expanded. It implies the acceleration of reduction degradation caused by diffusion of Al into hematite-magnetite matrix. Thus, addition of calcined dolomite in high Al₂O₃ sinter in this study is recommended to be under 4 wt% to avoid MgO segregation shown in **Figure 7**.

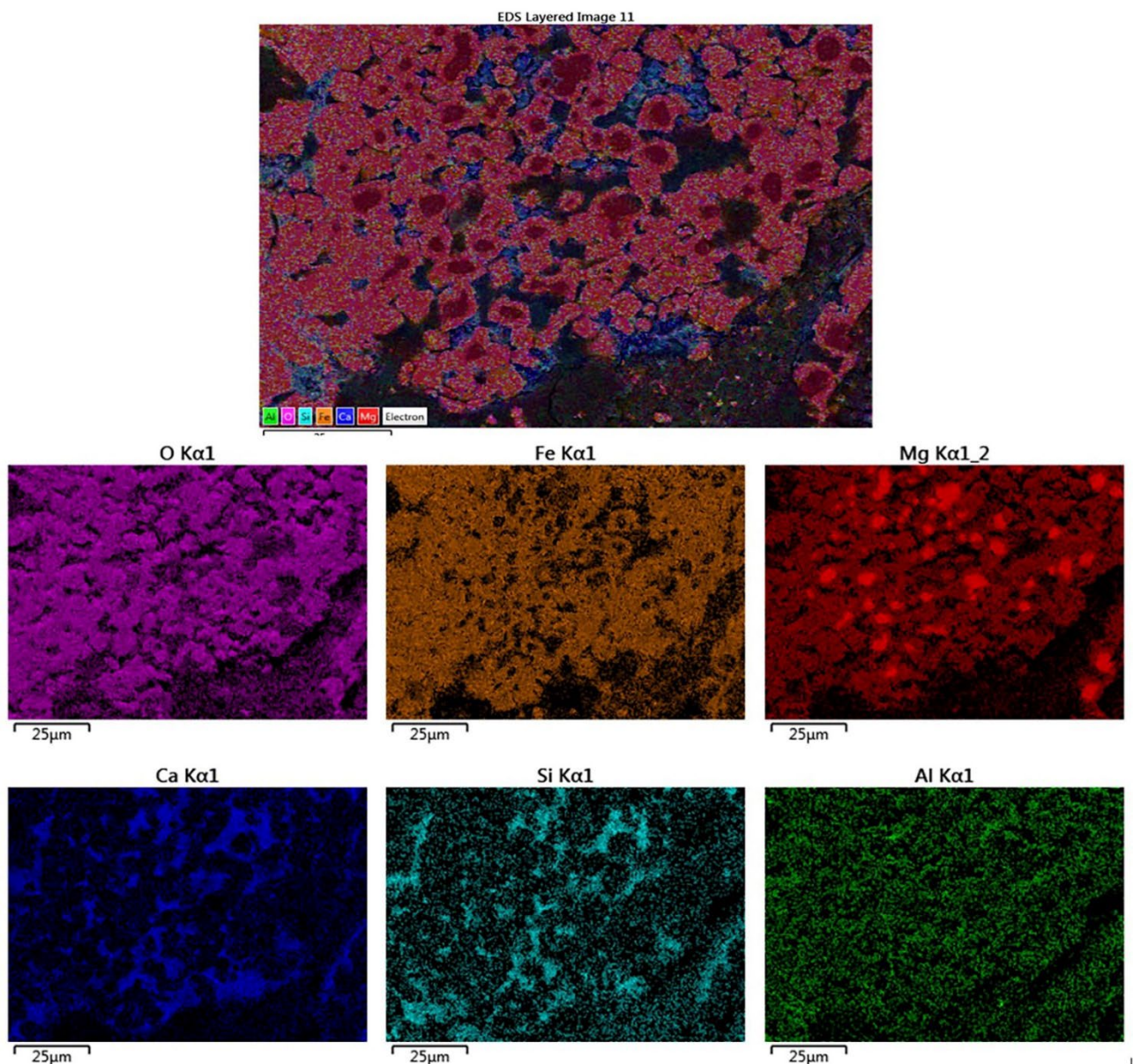


FIG 7 –Elemental distribution of selected area in the sinter to which 10 wt% of mill-scale and 6 wt% of calcined dolomite were added (Mill10-Dol6) by EDS mapping

CONCLUSIONS

To clarify the combined effect of adding mill-scale and calcined dolomite to sinter mix containing high Al_2O_3 ore on sinter quality, the reduction behaviour of iron ore sinters was investigated. From the findings, the following conclusions were obtained:

1. Magnetite became a dominant phase in the sinter with increasing mill-scale up to 15 wt% of mill-scale to sinter mix while magnesio-ferrite was formed instead of magnetite when calcined dolomite was added to sinter mix along with mill-scale. MgO stabilised the magnetite phase, so that magnesio-ferrite in iron ore sinter was remarkably increased while SFCA phase was decreased, respectively.
2. Several MgO clusters were observed as a residue with adding more than 4 wt% of calcined dolomite to sinter mix. Due to the limitation in the dissolution of MgO into magnetite matrix, MgO remained unreacted in the sintering process, resulting in the suppression of bonding phases including calcium ferrite. In addition, CaO supplied by calcined dolomite formed Calcium silicate as slag phase rather than forming Calcium ferrite or SFCA.
3. RDI_{-2.8} and compressive load of the iron ore sinters were improved while their reduction degrees were decreased when adding mill-scale and calcined dolomite. Calcined dolomite can play a same role as mill-scale to stabilise the magnesio-spinel for improving sinter strength and reduction degradation. It has a strong advantage of utilization of both FeO and MgO-bearing materials for counteracting the adverse effect of Al_2O_3 in high Al_2O_3 sinter, also decreasing FeO amount in final manufactured sinter. However, the total content of additives should be restricted under certain amount to avoid MgO segregation and maintain the overall sinter qualities.

ACKNOWLEDGEMENTS

The authors would like to thank Mr. Sang Yeol Lee for help with preparation of experimental procedures.

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