Effects of Basicity and $\text{Al}_2\text{O}_3$ Content on the Chemistry of Phases in Iron Ore Sinter Containing ZnO

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The effects of basicity and $\text{Al}_2\text{O}_3$ content on the chemistry of phases in iron ore sinter containing ZnO were investigated by Rietveld analysis of the X-ray diffraction (XRD) patterns. Bulk composition analysis was carried out using inductively coupled plasma-atomic emission spectroscopy (ICP-AES) and wet-chemical analysis. The composition of each phase was investigated using a scanning electron microscope-energy dispersive X-ray analyzer (SEM-EDX). It was found that ZnO exists in the franklinite and the silicoferrite of calcium and aluminum (SFCA) phases. With increasing ZnO content, the phase fraction of the franklinite increased, while the fraction of SFCA slightly increased. When ZnO content was fixed at 1 wt pct and basicity increased, the fraction of franklinite decreased and that of SFCA increased. Here, the solubility of ZnO in the SFCA increased. As the $\text{Al}_2\text{O}_3$ content increased, the fraction of franklinite decreased and that of SFCA increased, while ZnO content in the SFCA did not change significantly.

I. INTRODUCTION

RECYCLING by-products generated from the integrated iron- and steelmaking industry has drawn attention from the vantage point of environmental and economic issues. Charging of iron-containing industrial by-products, such as scrap and dust in the blast furnace (BF), is useful to reduce CO$_2$ emission from the ironmaking process as well as to increase economic competency.$^{[1–4]}$ For example, BF dust can be considered as a source of iron in BF operation, because it contains 15 to 40 wt pct of Fe. Recycling of BF dust has been generally applied to the sintering process.$^{[5–7]}$ BF dust is mostly composed of carbon, CaO, SiO$_2$, and iron oxides, but it contains some minor constituents such as $\text{Al}_2\text{O}_3$, MgO, ZnO, and so on. Among them, many researchers pointed out that zinc in iron ore sinter may cause accumulation of ZnO in the iron ore sinter and wear of the refractory material during the BF operation.$^{[8–10]}$

When iron ore sinter containing Zn is charged in the BF, or pulverized coal containing Zn is injected into the BF through tuyere nozzles, Zn vapor is generated in the relatively lower part of the BF. While traveling with the gas stream to the upper part of the BF, Zn vapor is oxidized and condensed in the charged burdens or the refractory materials. As this sequence is repeated, ZnO is accumulated in the iron ore sinter and the refractory materials.$^{[8,9]}$ As ZnO content increases in the refractory material, damage of the refractory material may occur.$^{[10]}$ If we consider the maximum use of various dust (not only BF dust but also basic oxygen furnace dust$^{[11,12]}$ and electric arc furnace dust$^{[13,14]}$), it is important to know the chemistry of iron ore sinter containing ZnO.

On the other hand, phases in the iron ore sinter are complex and mosaic structure is frequently observed. For the quantitative phase analysis of the iron ore sinter, the image analysis technique on the cross-sectional image has been generally applied$^{[15,16]}$ and the chemical composition of each phase has been determined using a scanning electron microscope-energy dispersive X-ray analyzer (SEM-EDX). However, the cross-sectional image does not provide adequate volume fractions of the phases so that an alternative technique for quantitative phase analysis should be considered. Hamilton
et al. suggested that Rietveld analysis of the X-ray diffraction (XRD) patterns of the bulk sample would be the most promising method for the quantitative phase analysis,[15] and several researchers have adopted this method for the quantitative phase analysis.[18–24] For example, Webster et al. investigated the phase transition at high temperatures using in-situ synchrotron XRD.[18–23] More recently, Takayama et al. quantitatively analyzed the phases in iron ore sinters from sinter pot tests.[24]

Generally, iron ore sinter is composed of hematite, magnetite, silicoferrite of calcium and aluminum (SFCA), dicalcium silicate (C2S), etc. Since Hancart et al.[25] reported that SFCA was the major bonding phase in the iron ore sinter, many researchers[26–28] have explored the composition range of SFCA. Inoue and Ikeda[26] suggested a substitution plane of SFCA; the substitution plane, called the “CCC plane,” was defined by the end members of CaO–3Fe2O3–CaO–3Al2O3–CaO–SiO2. Dawson et al.[27] reported the substitution plane as CaO–2Fe2O3–CaO–2Al2O3–CaO–3SiO2. Patrick and Pownceby[28] suggested the substitution plane as CaO–3Fe2O3–CaO–3Al2O3–4CaO–3SiO2. Especially, calcium-ferrite–based materials are sensitive to several factors such as the composition,[29] thermal history,[30] and gas atmosphere.[18,38,39] Therefore, it is important to control the factors and analyze the samples exactly.

In the iron ore sinter, Zn may form zinc ferrite or can be dissolved in other phases. However, no study has been reported yet on the phase equilibria and solubility of Zn in other phases of the iron ore sinter. In this study, the form of Zn in the iron ore sinter is identified by using the Rietveld analysis of the XRD patterns of the iron ore sinters and SEM-EDX analysis of the phases. With varying the bulk sample composition, the effects of basicity and Al2O3 content on the chemistry of the phases in the iron ore sinter are investigated.

II. EXPERIMENTAL

In the present study, mostly reagent grade chemicals (CaCO3, Fe2O3, SiO2, Al2O3, and ZnO, 99 pct purity, < 0.100 μm) were used. CaCO3 was calcinated to obtain CaO at 1273 K (1000 °C) for 2 hours. CaO was then crushed and particles of 53 to 100 μm were used. Commercial grade coke, which was provided by POSCO, was used as a carbonaceous material. Coke particles, which were crushed and sieved to the same size as CaO, were selected and used in the present experiments. The chemicals at a predetermined ratio were mixed and pelletized in a drum mixer at 250 rpm for 2 hours, while distilled water was sprayed by 7 wt pct. Then, iron ore green pellets of 2 to 4 mm were selected and used for the lab-scale sintering simulation experiments. Table I shows the nominal composition of the sample pellets, and Table II summarizes the chemical composition of the coke used in this study. After experiments, the chemical composition of the sample was analyzed using inductively coupled plasma-atomic emission spectroscopy (ICP-AES) and wet-chemical analysis.

Figure 1 shows a schematic illustration of the experimental apparatus. An electric resistance furnace of a maximum temperature of 1573 K (1300 °C) was used. A vertical alumina reaction tube (ID: 60 mm, OD: 70 mm, and length: 1000 mm) was placed in the furnace, and both sides of the tube were sealed with stainless cooling jackets. An alumina tube (ID: 47 mm, OD: 54 mm, and length: 314 mm) was attached to the bottom jacket with O-rings and another alumina tube (length: 100 mm) was placed above it. Between the two alumina tubes, a
stainless-steel mesh was placed to support the sample particles. At the bottom, alumina balls of 2 mm in diameter were charged to the height of 20 mm to prevent the dropping of molten phases during experiments. Then, pellets were charged in a 60-mm-thick layer. Finally, alumina balls of 2 mm in diameter were added on the top in a 20-mm thickness. Twenty millimeters above the sample particles, a heat exchanger composed of alumina balls of 10 mm in diameter was placed to prevent the chilling of the sample particles by rapid gas flows. To monitor temperature variation during experiments, two type-R thermocouples were buried in the sample pellets: one (TC-1) at a position 20 mm below from the top surface of the pellet layer and the other (TC-2) 20 mm below from TC-1.

Once the sample assembly was installed, it was heated to 1273 K (1000 °C) at a heating rate of +10 K/min (+10 °C/min) while flowing N₂ gas from the top of the reaction tube at a flowing rate of 300 mL/min STP. At 1273 K (1000 °C), 79 pct N₂-21 pct O₂ gas mixture was injected at a flowing rate of 20 L/min STP. Owing to the rapid chemical reaction between coke particles and oxygen, the temperature of the sample instantaneously increased. Figure 2 shows a typical result of the temperature variation of the sample. In 60 seconds, temperature monitored with TC-1 reaches a maximum temperature of 1573 K (1300 °C) and then decreases monotonically. Temperature monitored with TC-2 shows a similar variation with a time interval of 40 seconds. From the difference in temperature variations monitored with two thermocouples, the moving velocity of the combustion zone was estimated to be approximately 0.5 mm/s. When the temperature of TC-1 decreased to 1273 K (1000 °C), the electric current applied to the furnace was shut down and the sample was cooled to room temperature in the furnace. Due to the temperature profile obtained in this experimental
After the experiments, XRD analysis (D8 Advance, Bruker, Cu Kα target, 40 kV, 40 mA), wet-chemical analysis, and SEM-EDX analysis were carried out to identify the phases and compositions of the samples. In XRD analysis, the 2θ range was varied from 20 to 120 deg at the scan speed of 0.8 deg/min. X’Pert HighScore Plus 4.7 (Malvern Panalytical, Netherlands) was used to refine the phases and to perform Rietveld analysis.

III. RESULTS AND DISCUSSION

A. Effect of ZnO

To investigate the behavior of Zn in the iron ore sinters, ZnO content in the raw mixtures was varied from 1 to 3 wt pct (samples 1 through 3), while the basicity was fixed at 2 and the Al₂O₃ content was 1 wt pct. Figure 3 shows the identification results of XRD patterns of samples 1 through 3, and the comparison between the calculated and measured XRD patterns. At the bottom of each figure, differences between the calculated and measured profiles are shown. Commonly, Fe₂O₃, Fe₃O₄, FeO, SFCA, C₂S, franklinite, and Al₂O₃ were identified. No zincite (ZnO) was detected in the present samples. Since the binary system of franklinite and Fe₃O₄ is isomorphous, the XRD patterns for franklinite and Fe₃O₄ are almost identical. Therefore, the phase ratio was calculated with the values of lattice parameters of franklinite and Fe₃O₄.[40] In XRD patterns, various calcium ferrite phases, such as CaFe₂O₄ (CF),[41] Ca₃Fe₂O₅ (C₂F),[42] and CaFeO₇ (CF₂)[43] were also detected, but the amount of each phase was less than 0.6 wt pct. Therefore, these phases were excluded from the quantitative analysis. The formation behavior of complex calcium ferrites depends on oxygen partial pressure.[18,38] Among various complex calcium ferrites, SFCA was the main mineral phase. It is considered that high oxygen partial pressure leads to a favorable condition for forming the SFCA. SFCA was generated by the reaction between the liquid phase and magnetite during sintering, as suggested by Webster et al.[19] The Rietveld analysis results are summarized in Table III, and the phase fractions are shown as a function of bulk ZnO content in Figure 4. In this study, the weighted profile R factor (Rwp) and goodness-of-fit values on the Rietveld analysis are within the acceptable ranges of 1.59 to 1.97 and 1.30 to 1.55, respectively. With increasing ZnO content, the phase fractions of SFCA, Fe₃O₄, C₂S, FeO, and franklinite increased, whereas that of Fe₂O₃ decreased. Chemical analysis results are summarized in Table IV. It can be expected that some amount of ZnO could be reduced by coke during the experiments, but no significant change in ZnO content was noticed in this study. Besides, from the XRD analysis, zincite was not detected. Accordingly, it was considered that ZnO was dissolved in some phases such as franklinite.

To identify each mineral phase, the cross-sectional images were taken by using backscattered electron (BSE) microscopy. Then, SEM-EDX analysis was carried out to investigate the chemical composition of the selected phases. Figure 5 shows the BSE micrographs for samples 1 through 3. It is found...
that needlelike SFCA, Fe₂O₃, and Fe₃O₄ are the dominant phases. FeO, franklinite, and Al₂O₃ were detected from XRD analysis but are too small to be investigated from the cross-sectional image. From

| Sample | SFCA | Fe₂O₃ | Fe₃O₄ | Franklinite | C₂S | FeO | Al₂O₃ |
|--------|------|-------|-------|-------------|-----|-----|-------|
| 1      | 47.4 | 29.9  | 7.2   | 1.1         | 9.1 | 3.7 | 1.6   |
| 2      | 50.1 | 19.8  | 9.5   | 3.2         | 10.1| 5.6 | 1.6   |
| 3      | 53.1 | 13.5  | 10.9  | 4.5         | 10.2| 6.5 | 1.4   |
| 4      | 67.8 | 8.6   | 3.1   | 0.4         | 8.7 | 8.3 | 3.2   |
| 5      | 79.2 | 2.2   | 1.5   | 0.2         | 9.1 | 6.3 | 1.5   |
| 6      | 61.2 | 20.4  | 7.2   | 0.4         | 4.9 | 4.6 | 1.3   |
| 7      | 83.2 | 6.3   | 3.6   | 0.2         | 2.1 | 3.2 | 1.4   |

Table III. Phase Fractions of Mineral Phases in the Samples Determined by Rietveld analysis (Weight Percent)

![Phase fractions graph](image)

Fig. 4—Phase fractions in the sinter with respect to ZnO content, while the basicity was fixed at 2 and the Al₂O₃ content was 1 wt pct: (1) 1 wt pct ZnO, (2) 2 wt pct ZnO, and (3) 3 wt pct ZnO.

Table IV. Chemical Compositions of the Samples by ICP and Wet-Chemical Analysis (Weight Percent)

| Sample | Fe₂O₃ | FeO | M-Fe | CaO | SiO₂ | Al₂O₃ | ZnO |
|--------|-------|-----|------|-----|------|-------|-----|
| 1      | 71.97 | 5.32| 0.39 | 13.00| 6.22 | 2.20  | 0.90 |
| 2      | 71.97 | 4.45| 0.47 | 12.85| 6.00 | 2.40  | 1.87 |
| 3      | 70.34 | 5.34| 0.49 | 13.08| 5.58 | 2.38  | 2.79 |
| 4      | 71.62 | 4.48| 0.40 | 13.99| 4.54 | 4.01  | 0.96 |
| 5      | 71.39 | 5.35| 0.41 | 15.59| 3.88 | 2.48  | 0.90 |
| 6      | 71.11 | 5.34| 0.43 | 12.75| 5.88 | 3.58  | 0.90 |
| 7      | 69.44 | 4.73| 0.46 | 12.83| 5.86 | 5.74  | 0.95 |

![BSE micrographs](image)

Fig. 5—BSE micrograph of the samples: (a) sample 1 (1 wt pct ZnO), (b) sample 2 (2 wt pct ZnO), and (c) sample 3 (3 wt pct ZnO). S: SFCA, H: hematite, SP: spinel group including magnetite and franklinite, C₂S: dicalcium silicate, V: void, and EDX analysis points are indicated as green dots (Color figure online).
EDX analysis, Zn was detected in SFCA. To obtain the average composition of SFCA, three points of SFCA for each sample were investigated with SEM-EDX. The analyzed points are indicated as green dots in Figure 4. The average composition of SFCA is summarized in Table V. It is found that SFCA contained 1.34 to 2.02 wt pct ZnO. Accordingly, the majority of the increased ZnO resulted in the formation of the franklinite. Since Zn has low boiling temperature and high vapor pressure, it can be easily volatilized during the sinter plant operation under a reducing atmosphere. However, from this study, it was confirmed that ZnO formed franklinite and it was partially dissolved in SFCA at least in the initial stage of the practical sinter operation. It should be noted that the reducing condition was maintained only for 60 seconds, while coke particles reacted with the 79 pct N₂ to 21 pct O₂ carrier gas mixture, and after consuming coke particles, it was switched to the oxidizing condition. In a conventional sinter plant, reduction and evaporation of Zn and reoxidation may occur simultaneously. To investigate the removal of Zn in a conventional sinter plant, kinetic studies should be carried out separately under isothermal conditions for a longer period.

### B. Effect of Basicity

To investigate the effect of basicity, the basicity was controlled from 2 to 4, while the ZnO and Al₂O₃ contents were fixed at 1 wt pct, respectively. For this purpose, samples 1, 4, and 5 are compared. Figure 6 shows the identification results of the XRD patterns of samples 4 and 5. Fe₂O₃, Fe₃O₄, FeO, SFCA, C₂S, franklinite, and Al₂O₃ were identified for these samples. The Rietveld analysis results are summarized in Table III and graphically shown in Figure 7. The amount of SFCA increased with increasing basicity, whereas those of Fe₂O₃, Fe₃O₄, and franklinite decreased.

Figure 8 shows the BSE microscope observation results of samples 4 and 5. It is found that with increasing basicity, the fraction of SFCA increased from 47.4 to 79.2 wt pct and the width of the columnar SFCA became larger. As the fraction of SFCA increased, the fraction of franklinite decreased from 1.1 to 0.2 wt pct, while ZnO content in the SFCA was almost constant in the range of 1.08 to 1.48 wt pct (Table V). Accordingly, it is considered that as the basicity increased, ZnO tended to be preferentially dissolved in SFCA rather than forming franklinite.

### C. Effect of Al₂O₃

To investigate the effect of Al₂O₃, the Al₂O₃ content in the iron ore sinter was controlled from 1 to 3 wt pct, while the basicity was fixed at 2 and the ZnO content was 1 wt pct. For this purpose, samples 1, 6, and 7 are...
compared. Figure 9 shows the identification results of the XRD patterns of samples 6 and 7, and the Rietveld analysis results are graphically shown in Figure 10. As the Al$_2$O$_3$ content increased from 1 to 3 wt pct, the amount of SFCA increased from 47.4 to 83.2 wt pct. On the other hand, the amount of Fe$_2$O$_3$, Fe$_3$O$_4$, and franklinite decreased. Al$_2$O$_3$ is known as a stabilizing element for the formation of calcium ferrite,[44–46] and Webster et al.[19] reported that with increasing Al$_2$O$_3$ content, the formation temperature of SFCA in the cooling process increased. Accordingly, increased Al$_2$O$_3$ content would accelerate the formation of SFCA in the sinter.

Figure 11 shows the BSE microscope observation results of samples 6 and 7. There is no significant morphological change investigated in these samples. The ZnO content in SFCA of each sample did not change so much with increasing the Al$_2$O$_3$ content, while the fraction of SFCA considerably increased. Accordingly, it is concluded that most of the ZnO is dissolved in the SFCA phase.

D. Crystal Structure Change of SFCA

In the crystal structure of SFCA, the substitution of atoms is performed between tetragonal sites and octahedral sites. Hamilton et al.[17] reported that substitutions are made in M6, Ca1, and M12 sites. Here, M6 and M12 are the tetrahedral sites and Ca1 is the octahedral site. Figure 12 shows the positions of the atoms in the crystal structure. They assumed that M6 is occupied by Al, while M12 is substituted between Si and Al, and Ca1 is substituted between Ca and Fe. In this study, for the first time, it can be assumed that Zn substitutes the tetragonal sites as well as the octahedral sites. The probable position of the Zn atom is then simulated by placing the Zn atom in each site using a simulation program, X’pert highs score. The simulation results are summarized in Table VI. The atomic coordinate information of the substitution site, which is used in the simulation and reported by Hamilton et al.,[17] is indicated in Table VII. The simulation results show that the most probable position of Zn is the M12 sites. Takayama et al.[24] reported that Fe can be substituted not only at Ca1 sites but also at M6 sites. In this study, to satisfy the mass balance of Fe in SFCA, it was optimized that Fe substituted at all M6, Ca1, and M12 sites.

Figure 13 shows the composition change in SFCA obtained from EDX analysis and the change of the atomic occupancy at M6, Ca1, and M12 sites as ZnO content in sinter increases. As the initial ZnO content increases, the concentrations of ZnO and Al$_2$O$_3$ in SFCA increase, whereas that of SiO$_2$ decreases. Structural analysis reveals that Fe mostly occupies M6 and Ca1 sites. On the other hand, Si, Zn, and Al occupy M12 sites, where the substitution occurs. With increasing ZnO content, Zn and Al occupancies increase, whereas Si occupancy decreases.

Figure 14 shows the composition change in SFCA and the change of the atomic occupancy at M6, Ca1, and M12 sites as basicity in sinter increases. As the basicity increases, the concentration of Fe$_2$O$_3$ increases, while that of SiO$_2$ decreases. This trend is also related to the increased occupancy of Fe at the M12 sites and the decreased occupancy of Si. However, there is no significant change in the Zn occupation at the M12 sites.
Figure 15 shows the composition change in SFCA and the change of the atomic occupancy at M6, Ca1, and M12 sites as Al₂O₃ content in sinter increases. As the initial Al₂O₃ content increases from 1 to 3 wt pct, the concentration of Al₂O₃ in the SFCA increases from 1.30 to 5.29 wt pct, whereas that of Fe₂O₃ decreases from 80.46 to 71.85 wt pct. This trend is related to the increased occupancy of Al and the decreased occupancy of Fe.
of Fe in the M6 sites. However, it is noticeable that atomic occupancy at the M12 sites does not change considerably. The atomic occupancy at the Ca1 sites is relatively complex. Nevertheless, the occupancy of Zn is investigated at the M12 sites only, and it did not change with increasing Al2O3 content.

Based on the preceding simulation results, it is concluded that when ZnO is added in the sinter, it is dissolved in the SFCA phase and Zn preferentially occupies the M12 site. It is also found that the basicity and content of Al2O3 do not affect the dissolution behavior of Zn in the SFCA phase. Since the Rietveld

| Atom Site | Ref. [17] | This Study | Probable Occupancy |
|-----------|-----------|------------|-------------------|
| M6        | Al        | Al/Fe      | 0/1               |
| Ca1       | Ca/Fe     | Ca/Fe      | 0/1               |
| M12       | Si/Al/Fe  | Si/Al/Fe   | 0.7/0.1           |
|           |           | Zn/Fe      | 0.2/0             |

Table VII. Atomic Coordinates of M6, Ca1, and M1 in SFCA[17]

| Atom Site | x     | y     | z     |
|-----------|-------|-------|-------|
| M6        | 0.8374| 0.5293| 0.2345|
| Ca1       | 0.5558| 0.404 | 0.1563|
| M12       | 0.7282| 0.7307| 0.9286|

![Fig. 13—Composition change in SFCA and atom occupancy change in SFCA with respect to the ZnO content: (a) composition of SFCA from EDX, (b) atom occupancy changes in the M6 site, (c) atom occupancy changes in the M12 site, and (d) atom occupancy changes in the Ca1 site.](image)
analysis results are comparable with the SEM-EDX analysis results, it is quite reasonable to accept the simulation results.

IV. CONCLUSIONS

As the use of by-products increases in the iron ore sinter process, it is necessary to understand the phase stability of the iron ore sinter containing ZnO. In this study, the effects of the ZnO addition, basicity, and Al₂O₃ content on the behavior of Zn in the iron ore sinter were investigated. The phase fractions, the composition of SFCA, and the substitution sites of Zn could be identified by using Rietveld analysis, wet-chemical analysis, and SEM-EDX analysis. It was confirmed that ZnO existed as franklinite or was dissolved in the SFCA phase of the iron ore sinter. As the ZnO content increased, the amount of franklinite and SFCA increased, while the amount of Fe₂O₃ decreased. It was considered that Zn occupies the M12 sites of the SFCA phase. As the basicity of the iron ore sinter increased, the amount of franklinite considerably decreased, while the amount of SFCA increased. Here, the occupancy rate of Zn in the M12 sites did not change so much in the SFCA phase. As the Al₂O₃ content increased, the amount of franklinite slightly decreased and the amount of SFCA remarkably increased. Here, the occupancy rate of Zn in the M12 sites remained almost constant. Accordingly, with increasing Al₂O₃,
the SFCA containing ZnO became more stable. From this study, it is concluded that to remove Zn from the iron ore sinter, a kinetic study on the chemical reaction between CO and Zn in the SFCA is required in the future.

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