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Melting Property of MgO Containing Sinter*

By
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Synopsis
The solution property of Mg0 containing materials into the CaO-Fe203 system and the melting property of Mg0 containing sinter during reduction were investigated. The results obtained are summarized as follows:
(1) The formation of high viscosity silicate and magnetite prevents the homogenization of slag and therefore, Cr slag and Ni slag remain unslagged. The formation of dicalcium ferrite prevents the dissolution of periclase into the CaO-Fe203 system.
(2) During reduction unslagged Mg0 makes the shell of Mg0-FeO solid solution with FeO in glassy silicate and its solution is negli-gibly low.
(3) For both normal sinter and pellet, all silicate minerals dissolve above about 1240°C.
(4) Mg0 addition in sinter is effective to decrease the slag quantity during reduction but the melting property of sinter is not so improved as that of pellet. The proper basicity for sinter and pellet is about 1.4.

I. Introduction

Sinters with a high Mg0 concentration have been made recently by adding materials such as dolomite and serpentine. This purpose is mainly to prevent the decrease in fluidity of blast furnace slag, which is caused by charge of high A1203 ores. Many studies'-4) on Mg0 containing sinters so far reported were therefore restricted to the sintering behaviors of Mg0 containing materials and the effect of Mg0 on the low temperature characteristics of sinters such as reducibility and disintegration.
On the other hand, Mg0 containing pellets have been developed and it becomes clear that Mg0 is effective not only to decrease disintegrations~ but to raise the softening-melting temperatures during reduc-tion.6''~ And good operation results of blast furnaces have been obtained by use of them.6-9i
The present report describes the effect of Mg0 with change of basicity on the softening and melting

property of sinters in comparison with that of pellets.

II. Samples

Dolomite sinters and pellets prepared by industrial apparatuses and sinters by a testing pot were used as the samples. Those chemical compositions are shown in Table 1. Chemical reagents of Mg0, A1203, Si02, CaO, Fe203, and electrolytic iron were used for the quenching experiments to study generating quantity of liquid slag and liquidus temperatures of unreduced cores of sinter and pellet.

III. Melting Behavior of Unreduced Core

1. Experimental Procedure
1. Melting Experiments of Slag Minerals
Pellets and sinters were crushed and then reduced

by pure CO at 400°C for 20 min. By this treatment, hematite was reduced to magnetite, while the other minerals unchanged. The powders thus obtained were separated by using a magnet in methanol. The residual substances, exclusive of hematite and mag-netite, were reduced to wustite by CO-CO2 gaseous mixture (CO : 60%, CO2: 40%) at 900°C. Each 0.5 g of the obtained powders enveloped in platinum foil were heated so as to reach the fixed temperature by 1 min, held them at that temperature for 10 min and then quenched in ice water. The resulting powders were identified by X-ray diffraction.
2. Melting Experiment of Unreduced Core
Sinters and pellets were reduced to wustite by CO-CO2 gaseous mixture (CO: 60%, C02: 40%) at 900°C. After crushing by a disk mill, they were

pressed to columnar briquets of 10 mm~ x 9 mm under a pressure of 650 kg/cm2. They were heated with the rate of 5°C/min in N2 using a horizontal electric furnace and the melting behaviors of the samples were photographed.
3. Quenching Experiments of the Five Components Chemical reagents of hematite and electrolytic

Table 1. Chemical composition of the sample sinters and pellet.

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iron were mixed to give the stoichiometrical com-

position of wustite. The mixture was melted and crushed, and iron existing in the sample was removed by a magnet. The obtained wustite and chemical reagents of CaO, SiO2, A12O3 and MgO were mixed to fixed ratios. Each 0.5 g of the resulting powders packed in iron envelopes were heated at 1470°C for 20 min in N2. The sample was cooled to a fixed temperature with the rate of 15°C/ mm, held at that temperature for 60 min and then quenched in ice water. Minerals were identified by the use of an optical microscope, an EPMA and an X-ray diffracto-meter to determine the ratio of wustite and liquidus temperature.

2. Experimental Results
1. Melting Behavior of Unreduced Core
Previously we measured the permeability resistance of the packed bed of pre-reduced sinters and pellets at high temperature simulating the softening-melting zone of blast furnace, which led to the finding that it depends on the melting property of unreduced cores.7~ Since the reduction of porous ores to wustite finishes in the early stage, the unreduced core is com-posed of wustite, slag minerals and silicate glass. The melting property of cores is dependent on the mineral ratios, their melting points and their dis-solving behaviors into the molten slag. This induces us to study the melting property of core exactly in order to improve the high temperature property of
ores.

First of all, the melting behaviors of slag minerals of cores were studied. Here, sample A of high basicity and sample B of low basicity were studied, since slag minerals differ by the basicity." When the untreated samples are reduced to wustite, the ratio of wustite is so large that it is difficult to identify the other minerals by X-ray diffractometry. This required the concentration of slag by magnetic sepa-ration after reduction.12~ The resulting samples were

quenched from the fixed temperature and the minerals were identified by X-ray diffractometry. The results obtained are shown in Figs. 1 and 2. The slag min-erals of sample A are ~3-dicalcium silicate (shortened as C2S) and melilite. The diffraction intensities of these slag minerals at 1 200°C are still strong, but at 1 220°C, most of melilite dissolves and C2S di-minishes in quantity. Dissolution of C2S finished at 1 240°C and it is composed of molten slag and a part of wustite reduced from calcium ferrite.

On the other hand, the slag minerals of sample B of low basicity are melilite and kirschsteinite (CaFe-SiO4). The silicate glass melts at 1 150°C which is lower than that of the sample A. Dissolution of kirschsteinite finished at 1 160°C and that of melilite at 1 170°C.

The slag minerals of pellet D are melilite and calcium magnesium silicate (Ca7Mg(SiO4)4) which is dissolved as well at about 1220°C.

Next, the melting properties of the samples A, C and D were studied by heating those briquets to examine the effect of MgO on the melting property

and to compare the property of pellets with that of sinters. The results are shown in Photo. 1. In the case of sample C of no MgO addition, the lower angles became smooth at 1 270°C and the molten slag in-creased in quantity at 1 280°C. On the contrary in the case of dolomite sinter A, the lower angles began to become smooth at 1 300°C and the increase in the

quantity of molten slag caused deformation at 1 320°C, which is about 40°C higher than that of sample C. Besides the same state is reached at higher temperature for pellet D, namely the change hardly occurred till 1320°C and it began to deform a little at 1 330°C.
2. Liquidus Temperatures and Ratios of Wustite of the Five Components System
As described above the permeability resistance of

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Fig. 1. X-ray diffraction
sinter A reduced
tion.

figures with Cu
to wustite after

Ka radiation of
magnetic separa-

Fig. 2. X-ray

sinter
tion.

diffraction
B reduced

figures with Cu Ka radiation of
to wustite after magnetic separa-

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the softening and melting zone of blast furnace depends on the melting property of unreduced cores of ores. And slag minerals dissolve in the molten slag at this temperature, which led to the fact that the change of unreduced cores is attributed to the melting property of the five components system of FeO, CaO, SiO2, A12O3 and MgO.
Thereupon liquidus temperatures and quantities of molten slag were studied from the change in quanti-ty of wustite by basicity corresponding to those of pellets and sinters under constant quantity of A12O3 and MgO. For instance, weight percent of wustite becomes 75.0 for sinter A reduced to wustite under the assumption that the composition of FeO is con-stant. In the same way it becomes 85.4% for pellet D. Experiments were made for two levels. One cor-responds to the sinter level where weight percent of FeO was 75.0, that of A12O3 3.0, that of MgO 3.0, and that of the sum of CaO and SiO2 19.0, while the basicity was changed from 0.43 to 2.33. The other is based on the pellet level where weight percent of FeO was 85.0, that of A12O3 1.8, that of MgO 1.8, and that of the sum of CaO and SiO2 1 while the

basicity was changed from 0.5 to 2.0. In these ex-periments all primary crystals were wustite. In addition X-ray diffractometry was not applied to determine liquidus temperatures since dendritic wus-tite precipitated from molten slag.
The resulting liquidus temperatures for the two levels are shown in Figs. 3 and 4. Figure 3 reveals that the liquidus temperature shows the minimum value of 1 362°C at the basicity of 0.43 and increases with increasing basicity till the basicity of 1.5. It becomes the nearly constant value of 1430° C in the basicity range of 1.5 to 1.9 but it decreases when the basicity increases more than 1.9. Figure 4 shows the same trend, namely, liquidus temperature increases with increasing basicity in the range of 0.5 to 1.4 but above this range it decreases with increasing basicity. Next the ratio of molten slag was examined. In these experiments all primary crystals were wustite and only wustite and silicate glass existed above 1300°C. In this experiment the ratio was evaluated by the following method,15~ since minerals were homo-geneous and their compositions were nearly equal to those of equilibrium state although a chemical

analytical methodx4~ can be applied as well. Minerals were evaluated by the point analysis using EPMA and their ratios were calculated from Eq. (1) by taking the meterials balance obtained by the least squares method.

Ci = aij•xj ....(1)

Ci : a weight ratio of chemical composition

Photo. 1
Melting behavior of the wustite samples.

Fig. 3. Change of liquidus temperature by basicity.

Fig. 4. Change of liquidus temperature by basicity.

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az~ : a concentration of each mineral (EPMA)
xi : a weight ratio of each mineral
i : 1, 5 MgO, A1203, Si02, CaO, T.Fe
j : 1, 2 wustite, silicate glass
Figure 5 gives the ratios between wustite and silicate glass at 1300°C for the sinter level. The maximum ratio of wustite is about 55% at the basicity of 1.0, while there is a little difference between them in the

basicity range of 0.66 to 1.9. Except this range, the ratio of wustite decreases, particularly, at low basicity. Figure 6 shows the mineral ratio at 1 300°C for the pellet level. This indicates that the ratio of wustite has a nearly constant value of 65% in the basicity range of 0.7 to 2.0.

3. Discussion

When pellets and sinters are charged into a blast furnace, they are reduced from the outside of particles. And the outer shell of the particle is composed of metallic iron, wustite and slag minerals, while the inner core is composed of wustite and slag minerals. When it is heated at high temperatures, metallic iron becomes to form large continuous crystals by sintering, which leads to prevent the diffusion of reducing gas into inner cores and to retard reduction. At the

softening and melting zone, the molten slag generatess in the unreduced cores of ores and the load on ores

causes the exudation of slag containing wustite to its surface.

Vacant spaces between ores are filled with this molten slag at the softening and melting zone. This causes the decrease of fractional void volume to pro-duce a high pressure drop.
On the contrary, it is said enough to consider hematite, magnetite, hemicalcium ferrite, C2S, meli-lite, iron gehlenite and silicate glass as the principal minerals of fluxed sinters.10> Sasaki et a1.4~ revealed that hemicalcium ferrite of commercial sinters is

reduced to a fine mixture of magnetite and slag miner-als, and is further changed to a mixture of wustite and slag minerals.
Consequently unreduced cores of sinters are com-posed of slag minerals, silicate glass and wustite reduced from hematite, magnetite and hemicalcium ferrite at the shaft of blast furnace. Molten slag begins to be generated in the cores of sinters with descending through the shaft and increasing tem-perature. And minerals dissolves in this slag to increase the quantity.
Figure 1 indicates that silicate glass melts first between 1200° and 1220°C, then, parts of melilite, C2S and wustite dissolve at 1 220°C. And at 1 240°C

the increase of solubility allows to dissolve all C2S. This generating path of molten slag should be con-sidered on the basis of the phase diagram as follows : Considering that the gangue constituents of sample A comprise only CaO, SiO2 and A1203, it may be discussed on the basis of the phase diagram of CaO Si02 2CaO •A1203. Si02Fe019> system assuming the basicity as 2.0 instead of the real value of 1.85. This does not differ greatly from the actual generating path of melt although there is a little difference in

their melting points. There is a little variation in the chemical compositions of silicate glass which are the rest of melts solidifying after the precipitation of primary crystals in the sintering process. But the point analysis by EPMA revealed that they cor-responded approximately to the compositions near the eutectic line between CaO • Si02 and melilite

of the phase diagram of CaO•Si022CaO•A1203 Si02 FeO19~ system as shown in Fig. 7. Melt gener-ates at 1 220°C in which melilite and C2S begin to dissolve, and with increasing quantity and basicity it reaches the eutectic point of three components of L in Fig. 8. With further increasing temperature melilite becomes to disappear and the melt composi-tion moves on the eutectic line of C2S-FeO to point M. Hereupon all C2S dissolves and with increasing temperature it moves again on the line MN to point N which corresponds to the average chemical com-position of the core. Sasaki et al. stated131 that C2S would be a'-type judging from the temperature range of the sintering process. The identification as 8-type may be attributed to the holding temperature of 400°C at prereduction.
Photograph 2 shows the microstructures of sample A; one of which was reduced to wustite at 900°C and the other was quenched after it was kept at 1220°C for 10 min. It reveals that wustite from hemicalcium ferrite deform and dissolve in melt much

more than those from hematite and magnetite. But

Fig. 5. Change of mineral ratio of five components system

at 1300°C.

Fig. 6. Change of mineral ratio of five components system
at 1300°C.

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the point analysis of many wustites by EPMA shows an almost uniform composition. Chemical composi-tions of hematite, magnetite and hemicalcium ferrite differ from one another and they vary even in the same minerals, which are kept to wustite. However, the melt formation makes wustites composition uni-form. We should consider the similar eutectic melt formation process for B and D. For this reason in the case of discussion of melting behavior above about 1 240°C of the softening and melting zone of blast furnace where no slag minerals of sinters and pellets exist, it is sufficient only to consider that chemical compositions of cores independent of the kind and quantity of minerals.
The curve of liquidus temperature in Fig. 3 differs from that of the ratio of wustite in Fig. 5. For in-stance the value of the ratio of wustite reaches a maximum at the basicity of 1.0, while that of liquidus temperature at the basicity of 1.5. Consequently molten slag increases more rapidly above 1 300°C at the basicity of 1.0 than at that of 1.5. It is proper basicity where ores have high values of both the ratio of wustite during increasing temperature and liquidus temperature. In this sense industrial sinters and pellets are found to be produced at almost optimum basicity.

Value of the ratio of wustite at 1 300°C for a sample of Fe0 75.0, A1203 3.0, Ca0 13.2, and Si02 8.8wt% was about 19%, which led to the finding that Mg0 decreased the solubility of Fe0 in the melt.
For the above reasons the result of Photo. 1 will be considered as follows :

The melting property of sinters is improved greatly but the effect of Mg0 becomes smaller on sinters than on pellets since larger amount of Fe0 dissolves in slag because of greater quantity of gangue, and minerals containing Mg0 remains unslagged, which will be discussed in the next.

Iv. Behavior of Mg0 during Sintering and Re-duction

1. Experimental Procedure
1. Solution Experiments of MgO Containing Materials It has been examined the solution property of Mg0 containing materials in the Ca0-Fe203 system which should exist to a great extent in sinters. First of all, chemical reagents of Fe203 and Ca0 were mixed to be fused in a ratio of 85wt% to 15wt%. The slag obtained was crushed and packed around Mg0 containing materials in Pt envelopes. They were heated at 1350°C for 10 min with an electric furnace in air and were quenched in ice water.
2. Behavior of Unslagged MgO during Reduction Effect of unslagged Mg0 on the melting behavior of a core was studied. First wustite sinter A was

heated at 1220°C and then quenched. Its silicate glass was analyzed by EPMA. To obtain slag of the same chemical compositions, wustite was mixed with chemical reagents in the ratio of Fe0 40wt%, Ca0 30wt%, Si02 23wt%, and A1203 7wt% and heated it at 1 470°C in N2. This was crushed and packed around reagent grade Mg0 which was pressed to a mass under a hydrostatic pressure of 3 000 kg/cm2. They were wrapped in an iron envelope and heated at 1 220°C for 20 min in N2 with an electric furnace, then, quenched in ice water.

2. Results and Discussion

1. Solution Property of MgO Containing Materials It is well known that the sintering process depends on both the generating order and property of melt. And two paths have been considered for the melt

Fig. 7.
System CaO. Si02 2CaO. .A120,. 5i02-FeO.

Fig. 8.
System 2CaO. Si0ti 2CaO.A1203. Si0ti FeO.

Photo. 2. Change of microstructures of wustite sinter.

formation, namely, the one is Olivine while the other calcium ferrite.16~ Besides, the experiment of sim-

plified composition system has revealed that the reac-tion between Fe203 and CaO is the most liable to

occur and gangue minerals dissolve in it.17) On the other hand, MgO containing materials are difficult to react and apt to remain as intermediate products.4) In the present paper the solution property of MgO containing materials such as Ni slag, Cr slag and dolomite into the CaO-Fe203 system was studied. Photograph 3(1) shows X-ray images of the re-

presentative boundary texture of Cr slag in the slag obtained by EPMA. Cr slag is composed of forsterite

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(2MgO • Si02) and spinel (MgO .A1203) which remain unslagged as large crystals as shown by No. 1 and No. 3 in Table 2. Besides, a heavy element is con-sidered to be present at the black periphery of spinel, which would be made by the exchange of A13+ ions of spinel with Fe3+ ions of the slag as indicated by the analytical value of No. 2. For this reason the solu-tion property of spinel is bad and forsterite dissolves mainly as is indicated by the analytical value of No.
4. And magnetite, concentrating MgO and A1203,

precipitates from the CaO-Fe203 slag.
Ni slag also remains unslagged. It is composed of forsterite and enstatite (MgO • Si02) and enstatite

Photo. 3. X-ray images by EPMA of the
of Mg0 containing materials
slag.

mixture
and the

Table 2. Analytical results of the quenched mixture o f Mg0-containing material and calcium ferrite by X-ray microanalyzer.

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dissolves selectively at the interface and so only forsterite remains. In this case also magnetite, highly concentrating MgO, precipitates from the melt. When Cr and Ni slag dissolve in the CaO-Fe2O3 system, the formation of high viscosity slag containing much SiO2 and precipitated magnetite prevent the homogenization in this manner. Hence, the solubility of slag reaches saturation for forsterite and it would be left unslagged.
In the case of dolomite sinter it is known that MgO remain unslagged as periclase.4~ On the other hand, it is known that CaO dissolves at 1 320°C in the

CaO-Fe2O3slag composed in the ratio of 20 to 80wt%, while MgO hardly does so since it reacts with Fe2O3 in the slag to make the shell of MgO • Fe2O3 around itself.18~ The scanning image of Photo. 3(3) shows that two other phases are present except the white spherical periclase. The analytical value of No. 9 indicates light gray phases as dicalcium ferrite (2 (Ca, Mg, Fe)O •Fe2O3), while that of No. 10 denotes dark gray phases as calcium ferrite melt. At first dolomite was heated to decompose into MgO and CaO. CaO dissolves selectively in the slag, while MgO did not so. CaO dissolved decreased the activity of Fe2O3 in the CaO-Fe2O3 melt to prevent the formation of MgO •Fe2O3, and MgO began to dissolve. MgO and CaO continued dissolving to make dicalcium ferrite precipitate, then, the solution of MgO would scarcely occur. As mentioned above MgO containing materials remain unslagged since the generating melt is difficult to become homogene-

ous during the solution process. This requires that the grain sizes of MgO containing materials should be as small as possible and they should be mixed uniformly.
2. Behavior of Unslagged Mg0 during Reduction
As mentioned above parts of MgO containing materials are inclined to remain unslagged. The resulting periclase in the matrix is said to decrease the low temperature strength.4~ The present report de-scribes the effect of unslagged MgO on the softening and melting property of ores during reduction. Photograph 4 shows the images by the current and charactertistic X-rays of EPMA of the quenched samples. Table 3 shows the results by point analysis of the present phases. The solid solution of MgO-FeO varying continuously in composition are formed around the MgO grain as shown by the images of the characteristic X-ray of Fe and Mg of Photo. 4. And at the outside of this phase a solidified slag layer containing CaO and SiO2 for 11 % shown by No. 2 is present. Besides they are surrounded by a layer of C2S among which little gehlenite (2 CaO • A12O3 • SiO2) of the composition of No. 4 was located. Wustite dissolving MgO for 3 % precipitates from the original slag in which MgO scarcely dissolves as shown by the value of No. 6.

Consequently at first MgO makes MgO-FeO solid solution of high melting point at the slag JMgO inter-face through which MgO diffuses to dissolve in the slag. CaO, SiO2 and A12O3 move to the slag bulk due to the combination of MgO and FeO after MgO

Photo. 4. Scanning images of characteristic X-ray
of quenched mixture of Mg0 and silicate
glass.

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dissolving in the slag. The rest of melts is, then, considered to solidify after the precipitation of C2S and gehlenite. For this reason MgO scarcely dis-solves.

Like this MgO containing materials are inclined to remain unslagged in sinters which scarcely dis-solve at high temperatures in a blast furnace which results in little effect to increase the softening and melting temperature for the amount of MgO.

V. Conclusion

The solution property of MgO containing materials into the CaO-Fe203 system and the effect of un-slagged MgO at the high temperature zone of a blast furnace were studied. And this report describes the effect of basicity on liquidus temperatures and the ratio of melt at 1 300°C for the two levels of FeO

of FeO-CaO-SiO2 A1203MgO system.

(1) Spinel of Cr slag does not proceed to dissolve since it reacts with Fe3+ ion in the slag to form the shell of MgO. (Al, Fe)203. When Cr and Ni slag dissolve in the CaO-Fe203 system, the formation of high viscosity slag and precipitated magnetite prevent the homogenization. Periclase remain in the slag due to the formation of dicalcium ferrite after de-

composition of dolomite. For this reason low tem-

perature strength of sinters decreases with MgO addition.

(2) Unslagged MgO does not improve the high temperature property of ores since it makes the shell of solid solution of MgO-FeO at the boundary with slag and scarcely dissolves.

(3) MgO addition is less effective for sinters than for pellets while it is valid to decrease the quantity of generating melt. And low temperature dis-integration is improved since MgO promotes the dis-sociation of hematite to magnetite. For these reasons

MgO addition is
sinters.

effective to improve the quality of

Table 3. Analytical results of the quenched mixture of MgO and silicate glass by X-ray microanalyzer.

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