Dephosphorization Equilibrium between Liquid Iron Containing Cr and BaO-Cr₂O₃-FeO Slags*

By Ryo INOUE,** Hong LI*** and Hideaki SUITO**

Synopsis

Phosphorus distribution between MgOₙ sat.-BaO-Cr₂O₃-FeO(-BaF₂) slags and liquid iron containing Cr ([%Cr] = 0–16) has been measured in the temperature range from 1 550 to 1 650°C. The refining limit of dephosphorization in steel containing Cr and hot metal was discussed by using the values for phosphate capacity \( CP = (\%P)/[a_{O_2} \cdot d_{Cr}^2] \). Thermodynamic analysis indicates that the phosphorus partition ratio increases with \( C \) up to a critical value and decreases beyond that.

Key words: dephosphorization; iron; chromium; BaO; thermodynamics; equilibrium.

I. Introduction

Recently, the dephosphorization of steel as well as hot metal containing Cr has been studied by many researchers both on laboratory and industrial scale. The dephosphorization of Cr steels can be carried out either under oxidizing or reducing conditions. In the former case, the CaO-based,1-4 Na₂O-based5 and BaO-based6,7 fluxes containing Cr₂O₃ as an oxidizer have been used. The present work was undertaken to obtain the phosphorus distribution ratio in the liquid iron containing ~16 % Cr and BaO-Cr₂O₃-FeO(-BaF₂) system in the temperature range of 1 550–1 650°C using MgO crucible and discuss the refining limit of dephosphorization of stainless steel from thermodynamic viewpoint under oxidizing conditions.

II. Experimental

A vertical resistance furnace with heating bars of LaCrO₃ was used in the deoxidized Ar (100 ml/min) atmosphere in the temperature range of 1 550–1 650 °C. The slags were prepared by mixing appropriate amounts (8–10 g) of powdered BaO, BaF₂, Cr₂O₃, FeO and FeO-Cr₂O₃ and pelletized by pressing. FeO-Cr₂O₃ sample was made by sintering of FeO and Cr₂O₃ mixture at 1 350°C for 10 h. All these samples were dried at 800–900°C before an equilibration run. Commercial grade pure iron, Fe–26%P alloy, Fe–62%Cr alloy and reagent grade FeS were used for the preparing the alloy (25 g) whose initial content of P, Cr, and S were 0.3 %, 0–18 %, and 0.5 %, respectively.

The equilibrium time was 3 to 4 h depending on the slag composition and temperature. After equilibration the MgO crucible containing the experimental sample was withdrawn quickly using a Mo wire and rapidly quenched in water. But in the case of experiments BaF₂-containing slags, the bottom of the crucible was cooled by water and the slag surface by He gas.

Dephosphorization of liquid iron containing Cr using Na₂CrO₄ was carried out in an induction furnace. The alloy of 500 g ([%Cr] = 16–17, [%C] = 0, 1) was first melted in Al₂O₃ crucible under Ar atmosphere (200 ml/min) and 15 g of pelletized flux dried at 150°C for 2 h was added in batches of 3 g at a time of 30 s interval. Silica samplers were used to collect metal samples.

The chemical analysis of the elements in metal and slag except for O, S, and Ba was carried out by the JIS standard: O and S contents in metal were determined by the thermal conductivity measurements (HORIBA EMGA-1130) and by coulometry after combustion, respectively. The contents of BaO and F in slag were analyzed by the BaSO₄ gravimetric method8,9 and by the La-alizarin complexone absorptionmetry (JIS-K-0101), respectively.

III. Thermodynamic Consideration on Dephosphorization Equilibrium of Liquid Iron Containing Cr under Oxidizing Conditions

During dephosphorization, the oxidation potential should be carefully controlled in order that Cr in the metal is not oxidized. Under these oxidation potential the slag basicity must be as high as possible. In order to study the dephosphorization using fluxes with different basic oxides, the following three dephosphorization reactions are considered.

\[
3\text{Na}_2\text{O} (l) + 2\text{P} + 5\text{O} = 3\text{Na}_2\text{O} \cdot \text{P}_2\text{O}_5 (l) \quad (1)
\]
\[
\Delta G^\circ = -1 825.4 + 0.605 T \text{ (kJ)}^{10,11}
\]

\[
3\text{BaO} (s) + 2\text{P} + 5\text{O} = 3\text{BaO} \cdot \text{P}_2\text{O}_5 (s) \quad (2)
\]
\[
\Delta G^\circ = -1 613.5 + 0.6096 T \text{ (kJ)}^{11-13}\text{ [Appendix I]}
\]

\[
4\text{CaO} (s) + 2\text{P} + 5\text{O} = 4\text{CaO} \cdot \text{P}_2\text{O}_5 (s) \quad (3)
\]
\[
\Delta G^\circ = -1 336.6 + 0.5468 T \text{ (kJ)}^{14}
\]

The relations between log \( a_{O_2} \) and log [%P] at 1 600°C were calculated from corresponding values of \( \Delta G^\circ \) given by Eqs. (1)–(3) and the results are indicated in the right hand side of Fig. 1.

The oxygen potentials are assumed to be fixed by the following reactions:

\[
2\text{Cr} + 3\text{O} = \text{Cr}_2\text{O}_3 (s) \quad (4)
\]
\[
\Delta G^\circ = -707.8 + 0.3037 T \text{ (kJ)}^{15}
\]

* Based on the paper presented to the 113th ISIJ Meeting, April 1987, S234, at The University of Tokyo in Tokyo. Manuscript received on June 9, 1987; accepted in the final form on September 11, 1987. © 1988 ISIJ

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The log $a_0$ values calculated using $\Delta G^\circ$ values for the above two equations and the relevant interaction parameters$^{11,15}$ listed in Appendix II plotted against [%Cr] are shown in the left hand side of Fig. 1. Considering pure solid $\text{Cr}_2\text{O}_3$ equilibrated with the melt containing 18 \% Cr, the corresponding oxygen activity, $a_0$, can be calculated as 0.007 at 1600°C, using Eq. (4). It is obvious from Fig. 1 that under this oxygen potential phosphorus can be lowered to $5 \times 10^{-5}$ and 0.056 wt\% by the pure $\text{Na}_2\text{O}$ (l) and $\text{BaO}$ (s), respectively, whereas the dephosphorization by $\text{CaO}$ (s) can not take place.

Preliminary experiments using $\text{Na}_2\text{CrO}_4$ were carried out at 1600°C. The variations of Cr, P, and O contents with time shown in Fig. 2 indicates that no dephosphorization takes place, but the increase of O content is significant. The reason for this can be explained by the lowering of dephosphorization ability as a result of $\text{Na}_2\text{O}$ in the flux reacting with liquid iron as per Eq. (6) and formation of the high melting compound ($\text{Fe}_2\text{O} \cdot \text{Cr}_2\text{O}_3$) in the slag.

\[ \text{Na}_2\text{O} (l) + \text{Fe} (l) = 2\text{Na} (g) + \text{Fe}_2\text{O} (l) \quad \Delta G^\circ = 285.6 - 0.1888T \text{ (kJ)} \]

Therefore, it follows that the dephosphorization using $\text{Na}_2\text{O}$-based flux is possible if only the above reaction can be suppressed. Although the flux of $\text{Na}_2\text{O}$-$\text{SiO}_2$-$\text{Cr}_2\text{O}_3$ system is considered to prevent the occurrence of reaction (6) due to the lowering activity of $\text{Na}_2\text{O}$, the dephosphorization becomes unfavorable with increasing Cr content.

It can be seen from Fig. 1 that next to $\text{Na}_2\text{O}$, $\text{BaO}$ could be a good dephosphorizing flux. Moreover, the $\text{BaO}$-$\text{Cr}_2\text{O}_3$ system$^{17}$ has a wide liquid composition range of 8 to 63 wt\% $\text{Cr}_2\text{O}_3$ above 1440°C. In the present work, the $\text{BaO}$-$\text{Cr}_2\text{O}_3$-$\text{Fe}_2\text{O}_3$-$\text{BaF}_2$ system was used as a $\text{BaO}$-based flux and the P and S distribution ratio has been determined.

**IV. Results**

The slag and metal composition after experiments are listed in Table 1. The initial Cr contents are 0, 2, 10, and 18 \% and the initial slag compositions are as follows;

<table>
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<tr>
<th>Cr Content</th>
<th>Slag Composition</th>
</tr>
</thead>
<tbody>
<tr>
<td>0 %</td>
<td>$87% \text{BaO}$-$13% \text{Cr}_2\text{O}_3$ (Nos. 1-5)</td>
</tr>
<tr>
<td>2 %</td>
<td>$87% \text{BaO}$-$13% \text{Fe}_2\text{O} \cdot \text{Cr}_2\text{O}_3$ (Nos. 6-10)</td>
</tr>
<tr>
<td>10 %</td>
<td>$65% \text{BaO}$-$35% \text{Cr}_2\text{O}_3$ (Nos. 11-25)</td>
</tr>
<tr>
<td>18 %</td>
<td>$65% \text{BaO}$-$35% \text{Fe}_2\text{O} \cdot \text{Cr}_2\text{O}_3$ (Nos. 31-33)</td>
</tr>
<tr>
<td>0 %</td>
<td>$48% \text{BaO}$-$48% \text{BaF}_2$-$4% \text{Cr}_2\text{O}_3$ (Nos. 41-43)</td>
</tr>
<tr>
<td>0 %</td>
<td>$65% \text{BaO}$-$35% \text{Fe}_2\text{O}_3$ (Nos. 51-53)</td>
</tr>
</tbody>
</table>

Figure 3 shows the relation between T.Fe content and the ($\% \text{BaO}$)/($\% \text{Cr}_2\text{O}_3$) ratio at 1600°C. The T.Fe content increases with decreasing Cr content at the constant ($\% \text{BaO}$)/($\% \text{Cr}_2\text{O}_3$) ratio.

**V. Discussion**

1. **Phosphorus Distribution Ratio**

The values of $\log L_P = log (\%P / [\%P])$ are plotted against the Cr content as a function of the ($\% \text{BaO}$)/($\% \text{Cr}_2\text{O}_3$) + ($\% \text{Fe}_2\text{O}$) ratio in Fig. 4. It can be seen that $L_P$ decreases with increasing the Cr content at the constant $B$ ratio and it increases with the $B$ ratio at the constant Cr content. The $L_P$ values for the slags containing $\text{BaF}_2$ are lower than those in the slags without $\text{BaF}_2$ when comparison is made at a given Cr content and $B$ ratio. This indicates that the addition of $\text{BaF}_2$ lowers the dephosphorization ability despite that it increases the activity of
Cr$_2$O$_3$ (rcr2o$_3$) = 5.7*, resulting in the increasing oxygen potential determined by the Cr/Cr$_2$O$_3$ equilibrium. The present results suggest that the dephosphorization of liquid iron containing Cr by the BaO-based flux can be effective only at lower Cr content.

Table 1. Results of chemical analysis of slag and metal (wt%)

| No. | Temp (°C) | O  | P  | S  | Cr | P$_2$O$_5$ | S  | Cr$_2$O$_3$ | BaO | Fe$_2$O | MgO | BaF$_2$
<table>
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<td>0.015</td>
<td>0.146</td>
<td>0.0606</td>
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<td>0.261</td>
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<td>72.0</td>
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<td>1600</td>
<td>0.020</td>
<td>0.0962</td>
<td>0.0176</td>
<td>14.6</td>
<td>0.723</td>
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<td>25.6</td>
<td>69.2</td>
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</tr>
<tr>
<td>3</td>
<td>1650</td>
<td>0.027</td>
<td>0.0872</td>
<td>0.0120</td>
<td>14.7</td>
<td>0.590</td>
<td>0.954</td>
<td>26.8</td>
<td>67.2</td>
<td>1.72</td>
<td>2.67</td>
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<tr>
<td>4</td>
<td>1600</td>
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<td>0.0200</td>
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<td>14.1</td>
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<td>68.4</td>
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<td>2.95</td>
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</tr>
<tr>
<td>5</td>
<td>1600</td>
<td>0.015</td>
<td>0.0339</td>
<td>0.0162</td>
<td>6.61</td>
<td>1.05</td>
<td>0.994</td>
<td>23.7</td>
<td>70.7</td>
<td>2.10</td>
<td>1.31</td>
<td></td>
</tr>
</tbody>
</table>

* By use of the date (Nos. 41.-43 in Table 1), the value of $\gamma_{\text{Cr}_2\text{O}_3}$ was calculated from the values of $\Delta G$ in Eq. (4) and the corresponding interaction parameters (Appendix II).

Fig. 3. Relation between T,Fe content and (wt%) of BaO)/(wt%) of Cr$_2$O$_3$ ratio as a function of Cr content in metal.

Fig. 4. Logarithms of $L_P$ plotted against [%Cr] as a function of (wt%) of BaO)/(wt%) of Fe$_2$O + (wt%) of Cr$_2$O$_3$ ratio.

Cr$_2$O$_3$ ($\gamma_{\text{Cr}_2\text{O}_3}$ = 5.7*), resulting in the increasing oxygen potential determined by the Cr/Cr$_2$O$_3$ equilibrium. The present results suggest that the dephosphorization of liquid iron containing Cr by the BaO-based flux can be effective only at lower Cr content.
2. Phosphate Capacity

Phosphate capacity is defined as

\[ CP = \frac{\%P}{(a_P \cdot a_{PO_2}^2)} = \frac{L_P}{f_{\text{P}} \cdot a_{PO_2}^2} \]  

...(7)

where, \( f_{\text{P}} \) : the activity coefficient of P in metal.
Since in the present experiment, P, S, and MgO contents in slags are low (<5 %), the sum of the (%BaO) + (%Cr2O3) + (%FeO) is recalculated as 100 % and the log CP values at 1600°C are plotted in the BaO'-Cr2O3-FeO' ternary diagram as shown in Fig. 5. The corresponding interaction parameters listed in Appendix II were used for the calculation of log CP. It is seen from the iso-log CP lines (5.0, 6.5) that the log CP value tends to increase with increase of BaO content.

The dependence of log CP on slag compositions was examined by means of multiple linear regression method and the following good linear relationship was observed.

\[ \log CP = 0.085[(%\text{BaO}) + 0.05(%\text{Cr}_2\text{O}_3) + 0.12(%\text{FeO})] + 0.36 \quad (r = 0.97) \]  

...(8)

Figure 6 shows the relationship between log CP and \( 1/T \) obtained in the temperature range of 1550 to 1650°C for the BaO-Cr2O3-FeO systems.

3. Sulfide Capacity

Sulfide capacity is defined as

\[ CS = \frac{(%S) \cdot a_0}{a_S} = \frac{L_S \cdot a_0}{f_S} \]  

...(9)

where, \( f_S \) : the activity coefficient of S in metal.

The log CS values at 1600°C are plotted in the BaO'-Cr2O3-FeO' ternary diagram as shown in Fig. 7. The log CS value decreases with increase in Cr2O3 content and the maximum value for log CS was found in the BaO'-FeO' binary slags. The temperature dependence of log CS is shown in Fig. 8, indicating that log CS increases with temperature, which is the opposite trend compared with that observed in log CP as shown in Fig. 6.

4. Refining Limit of Dephosphorization of Stainless Steel under Oxidizing Conditions

The present results indicate that the dephosphorization of liquid iron containing Cr by BaO-based flux seems to be considerably difficult due to the lowering of oxygen potential with increasing Cr content; using the maximum value of log CP (≈ 7) at 1600°C obtained in the present work along with the oxygen potential determined by the 18%Cr/Cr2O3 (\( a_{\text{Cr}_2\text{O}_3(s)} = 1 \)) equilibrium, the degree of dephosphorization \( Y_p \) was calculated to be 23 % in the case of 100 kg/t flux consumption. The effect of C on \( L_p \) has been studied based on the data of log CP (≈ 7) as well as the corresponding interaction parameters given in Appendix II. The calculated results are shown in Fig. 9. The lines correspond to the results calculated based on the \( a_0 \) values by substituting \( a_{\text{Cr}_2\text{O}_3(s)} = 1 \) and 0.2 into Eq. (4). The line based on the calculation from \( \Delta G^0 \) for the \( \text{C}_2\text{O}_4 = \text{CO} + \text{CO}_2 \) reaction is also shown.

Fig. 5. Logarithms of \( C_P \) in BaO'-Cr2O3-FeO' system at 1600°C.

Fig. 6. Temperature dependence of log \( C_P \).
log \( L_P = \log C_P - \log f_P \cdot a_0^{0.5} \)

As is clear from Fig. 9, the value of \( L_P \) increases with increase of C content. This is due to the interaction of C with Cr and P; that is, the decrease of \( a_0 \) resulting in the increase of \( a_0 \) as well as the increase of \( f_P \) value. The value of \( L_P \), however, begins to decrease above the C content of about 0.5% governed by the oxygen potential fixed by the CO/CO₂ equilibrium. The values of \( L_P \) determined under the \( a_0 \) values of CrO₂/Cr₂O₃ and Cr₂O₃/CrO equilibria are also plotted against the C content in Fig. 9.

The \( C_P \) value was extrapolated to 1400°C from the results of the temperature dependence of \( C_P \). The effect of C content upon the dephosphorization of hot metal containing Cr has been studied for the slag with \( C_P=8 \) at 1400°C. The results are shown in Fig. 10, indicating that \( L_P \) increases with increase in C content according to the oxygen potential fixed by the Cr/Cr₂O₃ equilibrium, but it begins to decrease from about 2% C governed by the oxygen potential of the CO/CO₂ equilibrium.

To make comparison with the previous results obtained in the dephosphorization of hot metal containing Cr by using different type of fluxes, the relation between \( L_P \) and C content has been calculated based on the values of \( L_P (=7.5) \) at 1470°C and
flux consumption of 100 kg/t and the results are shown in Fig. 11. Two solid lines (Nos. 5 and 6) correspond to the oxygen potentials fixed by the Cr/Cr₂O₃ and G/CO equilibrium, respectively. All data except No. 2 line show a maximum degree of dephosphorization, the maximum value being dependent on the slag composition. The reason for this maximum is due to the change of oxygen potentials from the Cr/Cr₂O₃ equilibrium to the C/CO equilibrium with increasing C content. It may be noted that C content corresponding to the maximum is at a higher percentage than the value of C=1.3% determined from thermodynamic considerations.

**VI. Summary**

Phosphorus and sulfur distribution ratios between liquid iron containing Cr and MgOₐs₂=BaO-Cr₂O₃-FeO(-BaF₂) slags have been determined in the temperature range of 1550°C to 1650°C. The results obtained are summarized below.

1. Phosphorus distribution ratio increases with BaO content or with decreasing Cr content.
2. Phosphate capacity increases with BaO content or with decreasing temperature. Sulfide capacity increases with decreasing Cr₂O₃ content or with increasing temperature.
3. Thermodynamic analysis of dephosphorization of steel as well as hot metal containing Cr indicates that the degree of dephosphorization increases with C content up to a critical value and decreases beyond that.

**REFERENCES**

Appendix I

\[ \Delta G^\circ \] for \( 3\text{BaO}(s) + 2P + 5/2O_2 = 3\text{BaO}_2\text{P}_2 \text{O}_5(s) \)

From the values of \( \Delta H_{298}^\circ = -409.2 \text{kJ/mol} \) for \( \text{Ba}_3(\text{PO}_4)_2(s) \) and \( \Delta G^\circ \) for the reaction: \( 2\text{BaO}(s) = 2\text{Ba}(s) + 0_2(\Delta G^\circ = 1098.9 - 0.190T \text{ (kJ)}) \), the \( \Delta G^\circ_i \) value for Eq. (A-1) is estimated as

\[ 3\text{BaO}(s) + P_2 + 5/2O_2 = \text{Ba}_3(\text{PO}_4)_2(s) \] \( \Delta G^\circ_i = -2443.6 - T(\Delta S_f + 0.2848) \text{ (kJ)} \)

where, \( \Delta S_f \): the entropy of formation of \( \text{Ba}_3(\text{PO}_4)_2(s) \).

\[ 3\text{CaO}(s) + P_2 + 5/2O_2 = \text{Ca}_3(\text{PO}_4)_2(s) \] \( \Delta G^\circ_2 = -2313.8 + 0.5565T \text{ (kJ)} \)

From the values of \( \Delta G^\circ \) for \( 1/2O_2 = O \) and \( 1/2P_2 = P^\circ \) and the assumption that the entropy change for the Eq. (A-1) is equal to that for Eq. (A-2), the \( \Delta G^\circ \) for Eq. (A-3) is given by

\[ 3\text{BaO}(s) + 2P + 5O = 3\text{BaO}_2\text{P}_2 \text{O}_5(s) \] \( \Delta G^\circ = -1613.5 + 0.6096T \text{ (kJ)} \).

Appendix II

The interaction parameters used in the present work are listed in the following Table A-1.

<table>
<thead>
<tr>
<th>( i )</th>
<th>( j )</th>
<th>( e_i^j )</th>
<th>( r_i^j )</th>
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