

Research Article

Deng Nanyang, Wang Haijun*, Ling Haitao, and Wang Jianjun

The investigation on the middle period dephosphorization in 70t converter

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Abstract: An experimental test is carried out on the 70t converter in Masteel to study the law of middle period dephosphorization in the smelting period. It is found that the dephosphorization rate is slow in the early and middle period of smelting and the content of the final P in steel cannot reach the standard due to the low oxidation of slag. The content of the final P in steel can be controlled within 0.02% by adding ore to the molten bath in the middle period and raising the lance position. The experimental test shows that the dephosphorization can still be achieved by a large margin if appropriate slag conditions are maintained in the middle period when the C and O reactions are strong. By theoretical calculation, it is concluded that the slag content should be controlled between 15 and 23%, and the basicity of slag should be between 2.5 and 2.8 in the middle period of production. Through the observation of experimental slag by SEM, it is found that the limiting factor of dephosphorization in the middle period under the test conditions is that the dephosphorization speed is too slow due to the low oxidation, rather than the solidification of phosphorus.

Keywords: converter, dephosphorization, middle period slag, slag phase

1 Introduction

Phosphorus is a harmful element for most types of steel. When the phosphorus content in steel is high, the elasticity

and toughness of the steel are significantly reduced, resulting in brittleness. As the requirements on the quality of steel in the market become stricter, the composition control requirements for steel grades become more stringent [1–5]. The double-slag and slag-remaining method that has the advantages of dephosphorization and reducing slag consumption has become a widely used smelting process, and made certain metallurgical achievements [6–12]. However, it requires the converter to be tilted at the end of the early dephosphorization phase to tap the dephosphorized slag, which will increase the smelting cycle of each heat by 3–5 min.

At present, the steel industry is on the rise. Many steel enterprises begin to pursue high-yield smelting. And compress the smelting time for each heat of steel to achieve single tapping, therefore the slag tapping time in the early period of smelting is eliminated. On this basis, to achieve large-scale dephosphorization, it is necessary to achieve dephosphorization throughout the smelting process; especially further dephosphorization is required in the middle of the smelting, when the reaction of C and O is strong.

Some scholars have studied the effect of dephosphorization in the middle period. Yu [13] believed that the middle period of the oxygen converter process is the optimal period for dephosphorization, in which the basicity of the slag is relatively high and the fluidity of the slag is better; therefore, the dephosphorization efficiency is higher. Yang [14] found that the dephosphorization rate is the slowest in the middle period of smelting, with the dephosphorization rate in the early period 15.3 times of that in the middle period, and the dephosphorization rate in the later period of smelting is 6.7 times of that in the middle period of smelting. Zhuo [15] discovered through experiments that the phosphorus distribution ratio and dephosphorization ability in the early and middle periods of smelting are relatively low; however, the rate of dephosphorization reached the highest in the later period.

Although many scholars have conducted in-depth researches, they have never achieved a unified and systematic conclusion. Therefore, we conducted the middle period dephosphorization tests in a 70t converter to

* Corresponding author: Wang Haijun, School of Metallurgical Engineering, Anhui University of Technology, Ma'anshan, 243000, China, e-mail: whjchina666@163.com

Deng Nanyang: School of Metallurgical Engineering, Anhui University of Technology, Ma'anshan, 243000, China; Ma'anshan Iron & Steel Co., Ltd., Ma'anshan, 243000, China

Ling Haitao, Wang Jianjun: School of Metallurgical Engineering, Anhui University of Technology, Ma'anshan, 243000, China

further study the law of dephosphorization in the middle period of smelting, to provide a better guide on the dephosphorization process in the production process, and to ensure that the phosphorus content of the steel obtained from single tapping will meet the requirements.

2 Industrial experiments

The experiment was mainly carried out in a converter with a nominal volume of 70t. The blown oxygen lance is a 4-hole nozzle with an oxygen supply of $3.3\text{--}3.6\text{ m}^3\text{ t}^{-1}\text{ min}^{-1}$. The composition and average temperature of the molten iron are given in Table 1. The processing route of the single tapping experiment is as follows:

- 1) Leave the slag in the converter after tapping the furnace;
- 2) Solidify the slag by using the slag splashing protection technology, and then confirm the result;
- 3) Load scrap steel and molten iron, conduct oxygen smelting; about 1.5 min later add 2/3 of the lime and magnesium, and then add the remaining slag for the rest of the time. In the middle of the steelmaking process, add an amount of ore;
- 4) Slag liquid samples and the steel samples are taken and the temperature is measured at 3, 6, 9, 12 min, and the endpoint of the oxygen steelmaking process.

The dephosphorization rate X had been defined:

$$X = \frac{[\text{P}]\%_0 - [\text{P}]\%_i}{[\text{P}]\%_0} \times 100\%$$

where $[\text{P}]\%_0$ is the original phosphorus content in molten iron, $[\text{P}]\%_i$ is the phosphorus content after dephosphorization.

3 Results and discussion

The experiment was carried out for more than 20 heats in the 70t converter, and the feeding and blowing modes

were adjusted according to the dephosphorization result of the early experimental heats. Excellent dephosphorization effect was achieved, and the process of production for fast and large-scale production was developed.

3.1 Analysis of the smelting process of the experimental heats before adjustment

According to the analysis of the data regarding the smelting process of the heats before the adjustment, the element contents in the steel and the change of the slag composition of a typical heat at individual time points were obtained and listed in Tables 2 and 3, respectively.

The feeding procedure of this heat was as follows: blow oxygen for 1.5 min; add 1.5t of lime; add 374 kg of magnesium balls; add 660 kg of lime and 150 kg of iron ore at 3 min 20 s; add iron 180 kg of ore at 7 min. The blowing lance position and oxygen supply flow rate are shown in Figure 1.

The oxygen lance position adopted the “high-low-high-low” control principle. When the ignition was correctly carried out, the oxygen lance position was lowered gradually for rapid desiliconization and demanganization. At the end of the desiliconization and demanganization period, raised the lance position to ensure an appropriate decarburization rate in the middle of the smelting process. In the later period of the smelting, due to the weak carbon–oxygen reaction, the lance position could be raised to promote dissolution and prevent rephosphorization due to slag drying. At the end period, large oxygen flow and a low lance position were used to enhance the agitation of the molten bath to equalize the composition and temperature of the molten steel.

It could be seen from the molten steel component that the dephosphorization effects in the early and middle periods of the smelting process were very poor. The dephosphorization rates were only 10.3, 19.8, and 34.5% at 3, 6, and 9 min of the smelting process, respectively. Taking into consideration the dilution effect of molten steel scrap on the concentration of P, the dephosphorization rate at early period might be even lower. The dephosphorization

Table 1: Composition and temperature of molten iron

$w(\text{C})$ (%)	$w(\text{Si})$ (%)	$w(\text{Mn})$ (%)	$w(\text{P})$ (%)	$w(\text{S})$ (%)	Temperature of molten iron (°C)
4.13–5.02	0.15–1.27	0.14–0.72	0.12–0.23	0.006–0.050	1,264–1,360
4.55	0.50	0.23	0.180	0.022	1,316

Table 2: Change of element content in steel at different time

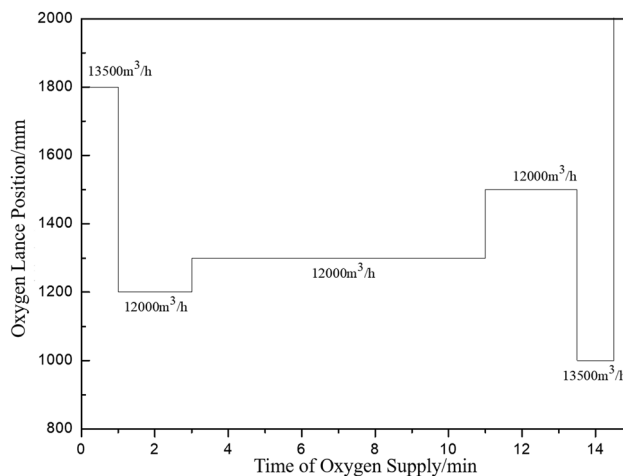
Time	Temperature (°C)	Oxygen flow (ppm)	C (%)	Si (%)	Mn (%)	P (%)	S (%)	Dephosphorization rate (%)
Molten iron	1,380		4.625	0.415	0.165	0.116	0.013	
3 min	1,396	10	2.91	0.06	0.06	0.104	0.021	10.3
6 min	1,465	11.7	2.25	0.01	0.08	0.093	0.022	19.8
9 min	1,533	21.4	1.39	0.01	0.12	0.076	0.023	34.5
12 min	1,621	71.6	0.47	—	0.13	0.051	0.020	56.0
14 min 29 s	1,675	384.5	0.11	0.01	0.087	0.025	0.019	78.4

rate at the end of the smelting was 78%, which meant that most of the phosphorus was removed as smelting proceeded from 9 to 14.5 min at which the lance is raised. The dephosphorization of the experimental heat was concentrated in the middle and late period of the smelting.

The analysis of the slag sample composition showed that the TFe contents in the slag in the early and middle periods were very low, equal to 7.5, 6.03 and 6.8% at 3, 6, and 9 min, respectively. It could be seen from the samples that the TFe contents in the slag were 11 and 14.7% at 12 and 14.5 min. Therefore, the FeO contents in the slag in the early and middle periods of the smelting were low. TFe content in the slag was barely increased even when 150 and 180 kg of iron ore were added to the furnace at 5 and 8 min. This should be the main reason why the dephosphorization rate was very low in the early and middle periods. Figure 2 showed the change of the basicity and the TFe content in the slag and the feeding details at different time points. It could be seen from the figure that although iron ore was added to the furnace twice in the middle period, the TFe content in the slag was still very low. Therefore, the addition of iron ore did not promote the dephosphorization rate in the middle period.

It could be seen from Figure 2 that the basicity of the slag reached 1.87 at 3 min, indicating that the dissolution of the lime in the early period is good, but the basicity of the slag was only 2.1 at 9 min, indicating that due to low FeO content in the slag in the middle of the smelting

process, the further melting of the lime was not significantly promoted. Therefore, the slag showed slight drying phenomenon, which has a great influence on the dephosphorization reaction. From 9 min to the terminal point of the smelting, the slag basicity increased rapidly, and the final slag basicity reached 3.1, indicating that the C and O reaction was not strong during this period, and the TFe content in the slag has increased, and the lime melted faster with increasing temperature. During this

**Figure 1:** The lance position and oxygen flow control parameters in the experiment heat.**Table 3:** Change of slag composition at different time

Time	CaO (%)	SiO ₂ (%)	Tfe (%)	MgO (%)	P ₂ O ₅ (%)	Basicity
3 min	26.56	14.21	7.46	4.06	1.42	1.9
6 min	49.12	24.4	6.03	5.30	1.89	2.0
9 min	34.61	16.55	6.8	5.34	1.92	2.1
12 min	42.93	16.61	11.14	6.74	3.03	2.6
14 min 29 s	43.38	13.85	14.73	6.24	2.93	3.1

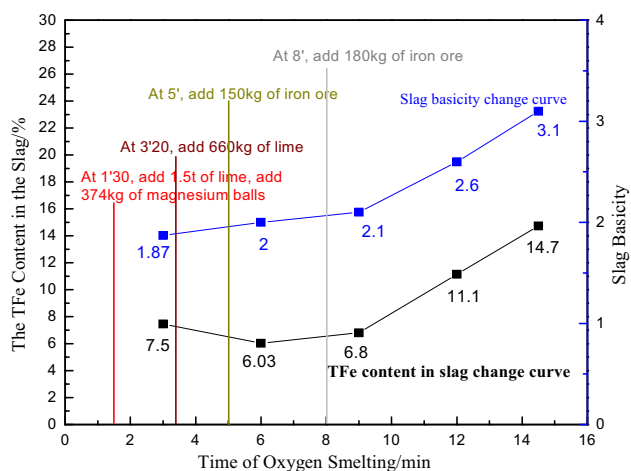


Figure 2: The change of the basicity and the TFe content in the slag and the feeding details at different periods.

time period, the dephosphorization reaction at the steel slag interface began to proceed on a large scale.

The experimental results showed that the FeO contents in the slag in the early and middle period of the smelting process were very low, and the slag in the middle period exhibited signs of drying, which made the dephosphorization rate in the early and middle period pretty low, and the dephosphorization in the whole smelting process was concentrated in the later period. The molten steel at the endpoint had a phosphorus content of 0.025% and did not meet the requirement of 0.02% of special steel. Therefore, in the subsequent experiments, measures such as adding more ore in the middle period, and appropriately raising the oxygen lance position were taken to ensure the TFe content in the middle period slag and to further explore the dephosphorization effect in the middle period.

3.2 Analysis of the smelting process of the experimental heats after adjustment

Because the TFe content in the middle period was low and there existed the “drying” phenomenon in the experiment

before the adjustment, the adjusted experimental heat changed the feeding mode and added more iron ore in the middle period. The specific feeding details were as follows: blow oxygen for 1.5 min, after that add 1,534 kg of lime and 333 kg of magnesium balls; add 443 kg of lime and 243 kg of iron ore at 4 min; add 240 kg of iron ore at 7 min; add 240 kg of iron ore at 10 min, and in the middle of the oxygen smelting, change the lance position from the previous 1,300 to 1,350 mm. The typical heat information after adjustment including molten steel and slag, respectively, at each time point of the smelting process is given in Tables 4 and 5.

It could be seen from the feeding mode and the change of the lance position that more iron ore was added in the middle period to the heat after adjustment compared with the heat before the adjustment. The analysis of the molten steel composition indicated that the dephosphorization rate in the early and middle period of the adjusted heat were significantly improved, reaching 39.3% at 3 min, 66.4% at 6 min, 79.3% at 9 min, and over 90% at the endpoint. After adjustment, the dephosphorization of the heat was concentrated in the early and middle periods. Table 4 showed that from 3 to 9 min of the process, in which large-scale C and O reaction occurred, the phosphorus in the steel was still removed by 40%, indicating that dephosphorization reaction occurred as long as the slag conditions were suitable. Even during intense decarburization reaction in the middle period, phosphorus in the molten steel could still be significantly reduced during this period.

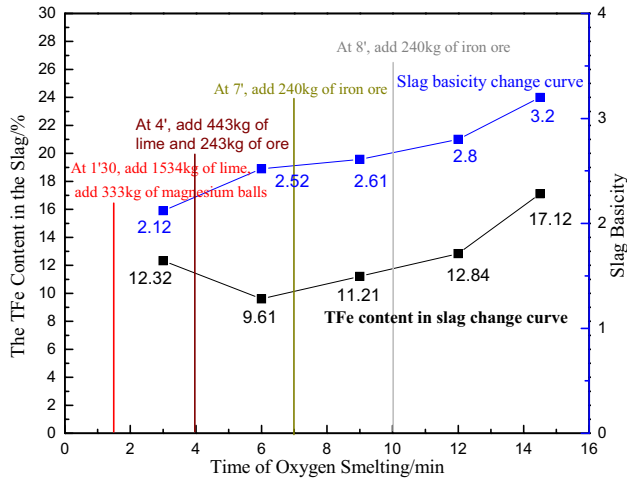
Analysis of slag compositions revealed that the TFe in the slag in the early and middle period of the heat was greatly improved compared with that before the adjustment, and the converted FeO was less than 15% only at 6 min, above 15% for the rest of the time and exceeding 20% in the end. Figure 3 showed the basicity of the slag, change of the TFe content in the slag, and the feeding details at each time point. It displayed that the slugging of the slag in the early and middle period of the smelting process of this heat was good. The slag basicity reached 2.12 at 3 min, and gradually increased in the middle and

Table 4: Change of element content in steel at different time

Time	Temperature (°C)	Oxygen flow (ppm)	C (%)	Si (%)	Mn (%)	P (%)	S (%)	Dephosphorization rate
Molten iron	1,312		4.22	0.38	0.14	0.155	0.03	
3 min	1,347		3.27	0.06	0.088	0.094	0.035	39.3
6 min	1,447		2.51	—	0.1	0.052	0.036	66.4
9 min	1,536	28.2	1.24	—	0.16	0.032	0.035	79.3
12 min	1,628	149.4	0.218	—	0.11	0.016	0.026	89.7
13 min	1,645	549.2	0.07	—	0.09	0.015	0.025	90.3

Table 5: Change of slag composition at different time

Time (min)	CaO (%)	SiO ₂ (%)	Tfe (%)	MgO (%)	P ₂ O ₅ (%)	Basicity (%)
3	43.19	20.37	12.32	5.21	2.32	2.12
6	45.56	18.08	9.61	5.36	2.94	2.52
9	46.73	17.90	11.21	5.16	3.40	2.61
12	47.89	16.95	12.84	4.92	3.61	2.80
13	45.15	14.08	17.12	5.27	3.11	3.20

**Figure 3:** The change of basicity and TFe in slag and the feeding details at different time points.

later period, which played a very important role in promoting dephosphorization of the molten steel. The end-point phosphorus content of the molten steel after adjustment was 0.015%, which was within the limit of 0.02% for special steel.

3.3 Analysis of middle period dephosphorization

The experiment after adjustment indicated that phosphorus could still be removed even when the decarburization reaction was strong in the middle period. Therefore, the slag conditions were further studied for the middle period dephosphorization. In the middle period of smelting, due to the intense reaction of C and O, a large number of CO bubbles were generated in the furnace, which would strongly stir the steel and slag., thus good kinetic conditions for the dephosphorization reaction were created as long as the slag drying phenomenon did not occur. Also due to the decarburization reaction, the temperature of the molten steel at this time has been raised by a certain extent compared with that of the early period.

Many scholars have studied the effect of temperature on dephosphorization, such as the common C–P selective oxidation transition temperature [16]. It is generally accepted that above a certain temperature, the decarburization reaction rather than dephosphorization in the molten bath is more likely to happen. Zhou et al. [17] calculated the equilibrium temperature of dephosphorization reaction, and believed that when the temperature is higher than the dynamic equilibrium temperature of dephosphorization reaction and phosphorus reversion reaction, the dephosphorization reaction no longer happens in the molten bath; Wang et al. [18] studied the dephosphorization temperature and found that the temperature suitable for dephosphorization should be as low as possible. He considered that a temperature higher than the melting point of dephosphorization slag superheat is more favorable for dephosphorization. Although temperature has great influence on the dephosphorization reaction, we believe that as long as the proper slag conditions (such as basicity, oxidizability, viscosity, etc.) are ensured, the process of the converter smelting is accompanied by the dephosphorization reaction, despite different slag conditions required by the dephosphorization reaction and different efficiencies of dephosphorization in different periods. Since the temperature of the molten bath in the early period is low, the slag may have relatively low basicity and oxidizability for rapid dephosphorization; when the temperature is higher in the later stage, higher basicity and oxidizability are required to balance the adverse effect of increased temperature on dephosphorization. If the oxidizability is low and the slag is dried, the phosphorus reversion reaction may happen.

The dephosphorization reaction requires the coupling of various conditions. Industrial experiments have shown that phosphorus can still be greatly removed even when the decarburization reaction in the middle period is strong. Therefore, when the middle period temperature is high, by calculating the appropriate slag conditions at this time, the dephosphorization of the molten bath can be further promoted to ensure proper phosphorus content at the tapping point. When the reaction proceeds to

the middle period, the thermodynamics of the dephosphorization reaction can be better analyzed by calculating its phosphorus distribution ratio. To find better slag dephosphorization conditions, we calculated the middle period slag using the Healy [19] and Suito *et al.* [20] equations of phosphorus distribution ratio, as shown in equations (1) and (2).

$$\log L_p = 0.08(\text{CaO}\%) + 2.5 \log(\text{TFe}) + \frac{22,350}{T} - 16 \quad (1)$$

± 0.4

$$\log L_p = \frac{11,570}{T} + 0.072 [(\text{CaO}\%) + 0.6(\%\text{P}_2\text{O}_5) + 0.3(\%\text{MgO})] + 2.5 \log(\text{TFe}) - 10.52 \quad (2)$$

To make the calculation results more representative, the authors analyzed a large number of experimental data to obtain the representative slag conditions in the middle period of smelting: temperature $T = 1,540^\circ\text{C}$, and in the slag $\omega(\text{MgO}) = 6\%$ and $\omega(\text{P}_2\text{O}_5) = 2.5\%$. The middle period $\log L_p$ values can be calculated by combining the typical middle period slag conditions with equations (1) and (2), as shown in Figure 4(a) and (b), respectively.

Figure 4 shows the relationship between $\log L_p$ values and the slag basicity obtained by Healy equation as well as the relationship between $\log L_p$ and FeO content by Suito equation. Although the calculated $\log L_p$ values are not the same, the variation trends are the same. With the increase of basicity of the slag, the value of phosphorus distribution ratio increases continuously. With the increase of TFe content in the slag, the value of phosphorus distribution ratio first increases and then decreases, with

the peak value appearing at the TFe content of 13–18%. This implies that in the middle period, the reaction of C and O is fierce due to the increase of temperature, and at this point, the slag needs more FeO content to satisfy simultaneous decarburization and dephosphorization. The converted FeO content is 16.7–23%. Figure 4 also exhibits that when the basicity of the slag is low, the calculated $\log L_p$ value increases greatly with the increase of basicity, and when the basicity of the slag is high, the $\log L_p$ value increases to a lesser extent as the basicity increases. This implies that under the middle period smelting conditions, the low slag basicity cannot meet the dephosphorization requirements of the slag, and the slag basicity becomes the limiting condition of dephosphorization reaction at this time, so the effect of increasing the slag basicity on the increase of phosphorus distribution ratio is more significant. However, when the basicity of the slag is increased to a certain range, where the basicity meets the dephosphorization requirement of the slag, it is no longer a limiting factor for the dephosphorization of the slag. Therefore, the effect of increasing the basicity on increasing the phosphorus distribution ratio is no longer significant, and subsequently, the $\log L_p$ value increases to a lower degree.

In summary, in the middle period of smelting, it is necessary to keep the FeO content in the range of 16.7–23% and the basicity of the slag in the range of 2.5–2.8, to ensure dephosphorization while decarburization happening. According to the on-site test, the FeO content in the slag is difficult to control in the middle period because the C and O reaction is too fierce. Therefore, the FeO content in the middle period can be relaxed to a range of 15 to 23%

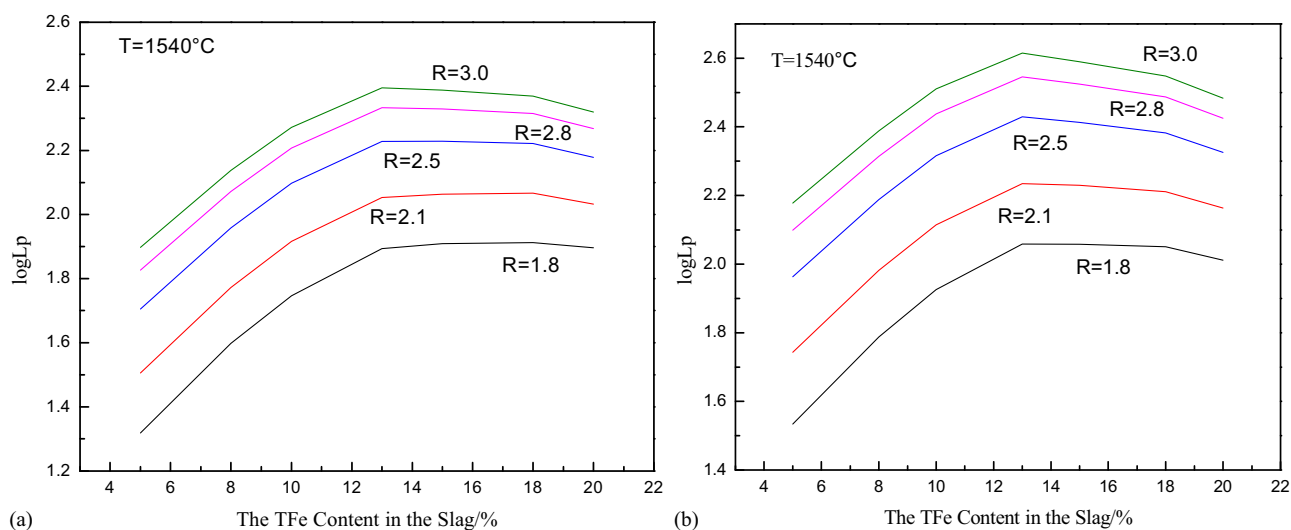


Figure 4: The middle period $\log L_p$ values by different calculation equations. (a) The $\log L_p$ value calculated using Healy equation. (b) The $\log L_p$ value calculated using Suito equation.

and the slag basicity in the range of 2.5–2.8. This condition would promote the dephosphorization in the middle period.

3.4 Middle period slag-phase analysis

To investigate the difference of dephosphorization before and after adjustment, the slag taken at 6 min was inlaid, ground, and polished, and the phase was observed and analyzed using scanning electron microscopy. The slag phase before adjustment was shown in Figure 5(a), and the slag phase after adjustment was shown in Figure 5(b). The compositions of each point in the electron micrographs in a and b were obtained by energy spectrum analysis as given in Table 6.

The slag sample electron micrograph in Figure 5 showed roughly two phases: the a1 phase, which was dark gray and close to an ellipse shape, and the a2 phase, which was light gray and amorphous. Combined with the point composition in Table 6, it indicated that there were very few metal elements in the a1 phase, which consisted of basically Ca, Si, and O elements and was enriched with certain amount of P element. It could be inferred that this phase should be the $2\text{CaO}\cdot\text{SiO}_2\text{--}3\text{CaO}\cdot\text{P}_2\text{O}_5$ solid solution phase. The content of each element in the a2 phase was relatively higher, and there was a small amount of P element, which could be inferred that this was a composite phase formed by various silicates. The slag sample electron micrograph in Figure 5(b) exhibited that there were three phases: a dark gray b1 phase, a light gray b2 phase, and a small amount of whitened b3 phase. Together with the point composition in Table 6, the data showed that the b1 phase was a $\text{C}_2\text{S}\text{--}\text{C}_3\text{P}$ solid solution phase

relatively richer in P element, the b2 phase was a silicate composite phase enriched with a certain amount of P element, and the b3 phase contained more metal elements such as Fe and Mg, which should be a composite RO phase. The electron micrographs of a and b indicated that there are a lot of solid solutions of dicalcium silicate and tricalcium phosphate in the slag samples before and after the adjustment; however, the content of P in a solid solution of phase a was much lower than that of the phase b.

The process of dephosphorization of the converter included dephosphorization and P-containing solid phases in the slag, that is, the phosphorus in the molten steel first entered into the slag through the oxidation reaction, further combined with the dicalcium silicate formed or being formed in the slag, to create solid solution and finally precipitation of P-containing solid phases in the slag, as shown in Figure 6. There was a good amount of dicalcium silicate in the middle period slag before and after adjustment, providing enough phosphorus-fixing carrier; however, the difference of phosphorus content in the slag was large, indicating the difference from the first step of the dephosphorization of the two conditions. Before the adjustment, the oxidizability of the slag was very low, resulting in a very slow dephosphorization reaction rate, and the amount of the phosphorus content moving from the molten steel to the slag was too small; after the adjustment, the oxidizability of the slag was increased, so the dephosphorization reaction was accelerated. Meanwhile, the phosphorus that entered the slag could be solidified by dicalcium silicate in time, so the phosphorus content in the slag was higher, and the dephosphorization effect was also greatly improved.

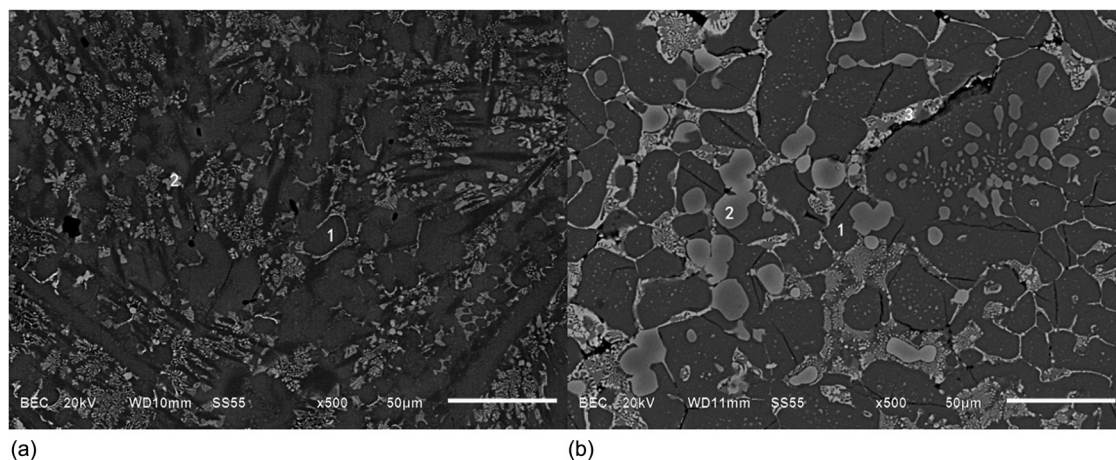
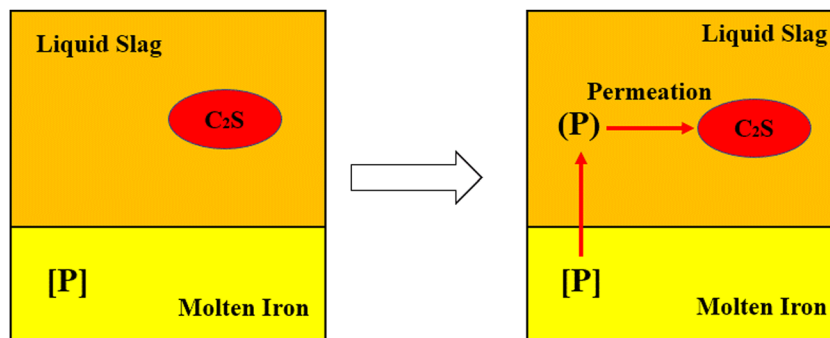


Figure 5: The SEM of Slag at 6 min before and after adjustment. (a) Before adjustment. (b) After adjustment.

Table 6: Mass percent of elements in each annotation position in Figure 5

Position	Ca (%)	Si (%)	O (%)	Fe (%)	P (%)	Mg (%)	Mn (%)
a1	35.31	14.68	24.49	2.33	1.23	0.84	1.60
a2	27.12	14.75	22.99	8.83	0.41	6.13	5.44
b1	43.81	13.15	29.83	1.38	2.94	0.64	1.03
b2	24.29	6.45	23.39	18.91	0.98	1.23	3.80
b3	0.83	—	19.63	45.21	—	13.79	11.89

**Figure 6:** Diagram of dephosphorization process of converter.

4 Conclusion

- (1) Through industrial experiments, it is concluded that phosphorus can still be greatly removed even when the C and O reaction in the middle period is strong. The main reason for the low dephosphorization rate before adjustment is the low oxidizability of the slag. By adding ore to the molten bath in the middle period, and appropriately raising the lance position, the dephosphorization rate in the middle period of the smelting can be greatly improved.
- (2) Dephosphorization happens in the whole process of smelting, except that the dephosphorization rate in each stage is different depending on the slag conditions. And the middle period slag conditions should be controlled as follows: FeO content should be between 15 and 23%, and the basicity of the slag should be between 2.5 and 2.8.
- (3) By observing the phase of the middle period slag of the test, we found that the dephosphorization process includes the oxidative dephosphorization process and the precipitation of P-containing solid phases in the slag, and concluded that the limiting factor of the dephosphorization in the middle period dephosphorization is not the precipitation of P-containing solid phases in the slag.

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