Influence of Basic Fluxes on Slag Formation in A Blast Furnace Using LKAB Pellets

Lena Sundqvist Ökvist, Guangqing Zuo and Jitang Ma

Lena Sundqvist Ökvist, M. Sc., Ph.D. Student, Research engineer, SSAB, Tunnplåt AB, Luleå, S-971 88, Sweden; Tel: 46-(0)920-92219, Fax: 46-(0)920-92061.
Guangqing Zuo, M. Sc., Ph.D. Student, Division of Process Metallurgy, Luleå University of Technology, S-97187, Sweden; Tel: 46-(0)920-91930, Fax: 46-(0)920-91199.
Jitang Ma, Retired professor, formerly worked at Division of Process Metallurgy, Luleå University of Technology, Sweden.

Abstract

Based on a proposal of injecting flux into the blast furnace via tuyeres, laboratory tests of softening and melting properties of four types of LKAB pellets with and without additional flux, corresponding to the bosh slag formation, have been carried out. The melting point measurements of coke and coal ashes with and without addition of basic flux, corresponding to the tuyere slag, have also been performed.

The test results showed that the additional basic fluxes could improve softening and melting properties of olivine pellets to a certain extent, but significantly deteriorate melting properties of self-fluxed pellets. Adding fluxes into coke and coal ashes could improve melting properties considerably.

These results indicate that injection of flux via tuyere will considerably improve the slag formation process in the blast furnace using self-fluxed pellets, both at cohesive zone and the raceway, as expected.
Introduction

The use of the olivine pellets produced by LKAB, Sweden has contributed considerably to improvement of the blast furnace performances at SSAB, Luleå Works, Sweden [1]. To further improve the quality of pellets, new types of self-fluxed-pellets with high-iron content are being developed.

The laboratory test has shown that the new types of self-fluxed pellets have quite good melting properties, in terms of melting temperature and narrow interval between softening and melting temperature [2]. However, an industrial trial using a new type of self-flux pellet did not show apparent improvement of the blast furnace operation [3]. Contrarily, there were some slag formation problems during test, e.g. high and variable resistance to gas flow in the blast furnace, very high viscous slag in the slag runner etc.

Jitang Ma [4] has pointed out that the slag formation problem was caused by formation of an excessive basic slag at the cohesive zone, where the top-charged fluxes dissolved into the self-fluxed gangue content of the pellets. Accompanied with further reduction of iron oxides the melting point as well as viscosity of the high basicity bosh slag increases considerably. Inevitable segregation of the top-charged fluxes would make it even much worse. Therefore, a proposal of injecting flux into the blast furnace via tuyeres, for replacing the conventional top-charging flux, has been presented. The use of injection of flux via tuyeres will greatly optimise the slag formation process, the basicity of the slags formed along all the height of the blast furnace will be quite even and within a desired range, besides decreasing the volume of bosh slag significantly.

In the past decades, great efforts have been given to studies of slag formation. A large number of laboratory tests of softening and melting measurements of iron ores have been carried out [5-11], but few tests included the slag formers. Therefore, these results cannot provide enough information of slag formation and the melting properties of the iron ores with fluxes.

In this study, laboratory tests of softening and melting properties of four types of LKAB pellets with and without additional flux, corresponding to the formation of the bosh slag, have been carried out. The melting point measurements of coke and coal ashes with and without addition of basic flux, corresponding to the tuyere slag, have also been performed. Three types of slags, primary slag, bosh slag and tuyere slag, have been mainly concerned. The primary slag was assumed to be formed by the gangue content of pellets; The bosh slag formed by the primary slag with dissolution of additional flux; The tuyere slag formed by ashes contained in top-charged coke and tuyere-injected coal. When injection of flux used, the injected flux is also included.

Experimental Technique

Experimental Apparatus for Softening and Melting Test

The experimental apparatus is shown in Figure 1. The furnace is heated electrically by U-shape Super-Kanthal with a heating zone of about 800 mm in height. The highest working
temperature can reach 1600°C. The crucible (40 mm i.d. x 85 mm height) made of graphite or alumina, as shown in Figure 2, has 6 holes of 6 mm in diameter on the bottom, allowing dripping of the molten materials and gas through the sample bed. A balance mounted with a container is installed beneath the reaction tube for weighing the dripped materials.

Samples of the test were sandwiched by two-layer of coke. Throughout the test, a constant load of 0.9 kg/cm² was applied to the sample bed by means of a pneumatic piston rod. The pressure given by the piston and a nitrogen gas flow through sample bed were regulated via a computer – PC 1. The bed contraction, the pressure drop across the sample bed, and the weight of dripped materials as well as the temperature in the course of the experiment were measured with corresponding devices and recorded by the second computer – PC 2. After reaching the temperature expected, the sample was cooled down to ambient temperature. The whole process was under the protection of nitrogen gas with a flow rate of 7 l/min. The experimental conditions used for softening and melting tests are summarised in Table1.

For characterising the softening and melting behaviours of the samples, the following two indices have been used:

• $T_1$: a temperature, at which the pressure drop and the shrinkage of the sample bed start to increase sharply; it serves as the softening temperature in this paper. Figure 3 shows one example of this temperature for Pellet MPBO;
• $T_{sd}$: start-of-dripping temperature, at which the first droplet of the melting material dropped to the collector, it also stands for the melting temperature in the paper.

**Experimental Apparatus for Measuring Melting Point**

Figure 4 shows a schematic view of the high temperature microscope for measuring the melting point. The furnace is also heated electrically. The highest working temperature can reach 1640°C. When measuring the melting point of slag, a specimen of slag sample of 3 mm in height and 2mm×2 mm in cross section area was placed on an alumina sample holder in the furnace and heated up gradually in a nitrogen gas atmosphere. A magnified contour of the specimen can be observed via a magnifier, as shown in Figure 5. A camera could be used to take photos of the contour of the specimen. Changes of the shape of the specimen during the test could be either drawn manually, according to optical observation, or recorded by the camera for determining the following characteristic temperatures:

• Softening: At which the specimen started to deform, the edges of specimen disappeared, Figure 5-b;
• Flow point: The materials flows and the height of the specimen decreases to about 1/3 of its original one, as shown in Figure 5-c. This temperature is defined as the melting temperature of the sample in this study.
Preparation of Samples

Pellets and fluxes
Four types of pellets made at LKAB were tested. The chemical analyses of these pellets as well as three types of fluxes, taken from SSAB, used in the study are shown in Table 3. Pellet A and B are self-fluxed, being developed, the former one has slightly higher CaO content, but lower MgO content than the latter one. MPBO and KPBO are olivine pellets, differ from each other in CaO content slightly.

Samples of the self-fluxed pellets were pre-reduced to reduction degrees 60, 75 and 90 in percentage under conditions of 850°C and 55%\(N_2\)-40%\(CO\)-5%\(H_2\) reducing gas (cracks of pellets during the reduction could be avoided to a large extent). Olivine pellets were pre-reduced at a temperature of 950°C with the same reducing gases. Accordingly, BOF-slag used was also pre-reduced under the same conditions.

Considering the amount used and segregation of the BOF-slag in the blast furnace, 5% or 10% of BOF-slag in weight of pellet were added into the samples. Three different placements of BOF-slag in the pellet bed were used, as shown in Figure 6.

Coke and coal ashes
As mentioned early, the tuyere slag in this study was considered to be formed by the ashes of coke and coal burnt in front of tuyere. When injection of flux is assumed, the injected flux should be included. To make test specimens similar to the actual tuyere slag, the specimens were made of mixtures of coke and coal ashes, with or without additions of fluxes. The ratios of these materials used were different, according to different injection levels of coal and flux. Coke and coal ashes used were obtained by burning coke and coal, taken from SSAB, slowly up to 800°C in a furnace in the coal and coke laboratory of SSAB.

Three levels of coal injection and five levels of flux injection, no injection, low (L), medium (M), high (H), very high (VH), were considered in the tests as shown in Table 4. The amounts of different types of fluxes used were different, determined on the basis of obtaining five levels of same basicity of \(B_\frac{2}{2}\) (CaO/SiO2) of the tuyere slag, as that of BOF-slag used. The compositions of the specimens used are listed in Table 5.

Experimental Results

Softening and Melting Properties of Pellets
Softening and melting tests of pellets were carried out mainly in graphite crucible. Pellet samples were sandwiched by two layers of coke.

As shown in Figure 7, these two new types of self-fluxed pellets have lower softening and melting temperatures than that of olivine pellets. However, the temperature intervals between the softening and melting of the self-fluxed pellets are narrower than that of the olivine pellets tested.
The test results also show that the softening and melting temperatures increase with the increase of the pre-reduction degree, as shown in Figure 8.

**Melting Property of BOF-slag**

Softening and melting tests of BOF-slag were carried out in two types of crucibles, graphite and alumina. Table 6 shows the test conditions and results.

The results indicate that the melting property of BOF-slag is strongly dependent on the presence of carbon. When BOF-slag is sandwiched by two-layer of coke in a graphite crucible, it does not melt down at all up to a temperature 1500°C. Contrary, when using alumina crucible, the measured softening temperature BOF-slag is only about 1343 °C, and it starts dripping at a temperature 1400°C.

These results indicate that the carbon has an important role in the melting process of BOF-slag. Therefore, in order to obtain the results in relevant to the conditions in the blast furnace, graphite crucible and two-layer of coke, putting at the up and bottom of the samples, were used in the all latter softening and melting tests.

**Influence of Fluxes on the Melting Property of Pellets**

The test results showed that the effects of fluxes on the softening and melting properties of pellets were different, depending on the type of pellets and of the fluxes used.

**Influence of BOF-slag on self-fluxed pellets**

Generally, BOF-slag could increase the melting temperature of the self-fluxed pellet considerably and softening temperature slightly. The changes of the melting temperature were related to the distribution of BOF-slag in the pellet bed.

As shown in Figure 9, when BOF-slag is evenly distributed in the pellet bed, the melting temperature increases dramatically by more than 80°C. Not any dripping material is collected at temperature up to 1500 °C. It seems that the BOF-slag can block the dripping process entirely during the tests. Therefore, melting point is not obtained. When the BOF-slag was unevenly placed in the sample bed, the melting temperature increases by about 50°C, and the amount of the dripped metallic iron collected is decreased. When it was uniformly placed beneath the pellet layer, no change of melting temperature of the pellets can be seen. However, some white-gray powders could be found in the crucible after the tests for the latter two cases. A preliminary X-ray diffraction analyses showed that the white-gray powder contained di-calcium silicate (Ca2SiO4), and other complex compounds, e.g. 3CaO•Al2O3, 3CaO•SiO2 etc. TGA test showed that the melting temperature of the powder was higher than 1600°C, much higher than that of pellets and BOF-slag. These results indicate that some compounds with very high melting points, e.g. di-calcium silicate- (melting point ~2100 C), were formed during the test. The mechanism of generation of these powders and their influences on the slag properties remain to be further studied.

**Influence of BOF-slag on olivine pellets**

As shown in Figure 10, in general, additional BOF-slag decreases the melting temperature of samples for pellets of 75 and 90% pre-reduction degree, but has little effect on the softening
temperature. Adding 5% BOF-slag to pellet bed give more influence on the decrease of the melting temperature than adding 10% BOF-slag. It is also found that the flux gives more influence on the melting temperature than on the softening temperature.

**Influences of some other basic additions**

Tests of adding limestone and burnt dolomite to sample bed of pellet A were also conducted. The basicity $B_2$ -CaO/SiO$_2$ or $B_3$ – (CaO+MgO)/SiO$_2$ of the samples were adjusted to be the same as that of adding of 5% BOF-slag, respectively. The pellets used were pre-reduced to a degree of 75%. Slag formers were evenly distributed in the pellet bed.

The results showed that limestone or burnt dolomite could increase melting temperature of pellet A by about 30°C and 25°C, respectively, much less influence than BOF-slag. The reasons will be discussed later.

Some white-grey powders could also be found in the sample bed after the experiment. However, the amount is too little to make further examination.

Tests of the olivine pellets with the addition of lime were carried out too. The basicity of $B_2$ of the slag was the same as that of adding 10% of BOF-slag. The results showed that the influence of the lime on softening and melting temperature was similar to that of adding BOF-slag.

**Melting Points of Coke, Coal ashes and BOF-slag**

As shown in Figure 11, the coke ash has the highest softening and melting temperatures among those materials tested. The coal ash has much lower ones, only 1331 and 1430°C, respectively. The BOF-slag has the medium values, 1305 and 1364°C. A tuyere slag formed by a mixture of coke and coal ashes, corresponding to the condition without injecting flux, has higher softening and melting points than that of coal ash and BOF-slag alone, but lower than that of coke ash.

**Effect of Fluxes on Melting Points of Coke and Coal Ashes**

Addition of fluxes to coke and coal ashes, corresponding to the tuyere slags with injection of flux, can considerably lower the melting temperature and the interval between the softening and melting temperatures, as shown in Figure 11 and Figure 12. However, the changes of the melting temperature varied with the types and the amounts of the fluxes used.

Generally, the melting temperature of the specimens decreases significantly with the increase of the addition of BOF-slag or lime, until up to the “high” level. Further increasing the addition of BOF slag and lime to the “very high” level will increase the melting point of the specimen slightly.

Adding dolomite to a “low” injection level decreases its melting point considerably. However, further increasing the additions will increase its melting point, as shown in Figure 12.
Discussion

Melting Properties of Pellets and BOF-slag

The test results showed that these two types of self-fluxes pellets both had higher softening temperature, lower melting temperature and therefore narrower intervals between these two temperatures than that of olivine pellets.

According to its chemical composition as shown in Table 7, the primary slag, formed by gangue content of pellet A, is located at a low melting temperature region in a ternary system of CaO-SiO$_2$-MgO with 10% Al$_2$O$_3$, as point A shown in Figure 13 (a). Its melting point is estimated to be about ~1300 °C. Since its basicity B2 is about 1, its viscosity is also supposed to be quite low. A good dripping property of the sample obtained could be one of the evidences. Therefore, the pellet A itself should not be the cause of slag formation problem.

Contrary, the melting properties of BOF-slag were changeable, in presence of carbon, as it is in the blast furnace. Its melting point could be increased dramatically, also with a very bad dripping property. These phenomena can be considered as a result of reduction of the FeO in the slag by carbon. As the basicity B2 of the BOF slag is about 4, decrease of FeO content would significantly increase its melting point. Chemical analysis of the BOF-slag after the tests up to 1500°C showed that the FeO and Fe$_2$O$_3$ in the slag were only 5.3% and 1.5%, respectively, while the original ones were 16.4% and 8.08%, respectively. The melting temperature of the BOF-slag after the tests, according to our measurement result, was higher than 1520°C, much higher than that of the original BOF-slag, about 1400°C.

Causes of Troublesome Melting Process when Adding Fluxes to Self-fluxed Pellet Bed

With dissolution of flux into the self-fluxed primary slag of pellets, the basicity of slag formed increases accordingly. If the inevitable segregation of top-charged flux is considered, the slag formed could have very high basicity locally. Table 7 lists some calculated composition of slags, based on the samples used. Bosh slag 1-2 are two slags formed by the gangue content of pellet A and 5% BOF-slag added, with no FeO content and about 10% FeO content, respectively. Bosh slag 4 formed by 10% BOF-slag and pellet A without FeO content, representing the case with segregation of BOF-slag. The basicities of B2 of the bosh slags formed can reach up to 1.87 and 2.36, the same as or very close to the basicity 2 of di-calcium silicate (2CaO·SiO$_2$, B2= 1.87).

As shown in Figure 13 (a) both bosh slag 1 (A1) and 3 (A2) have much higher melting point than that of A.

In addition, reduction of FeO can significantly change its melting point at this range of slag basicity. As shown in Figure 13(b), when basicity B2 is greater than 1, decrease of FeO can increase melting point considerably. With decrease of FeO content, the high melting point of the bosh slag 2 will further increase its melting point dramatically. Therefore, adding BOF-slag into the samples will bring twofold effects on melting properties: 1) Increase melting point by an increased basicity; 2) Further increase melting point by reduction of FeO in this region. In this case, formation of a very high melting point of di-calcium silicate is quite possible.
Improvement of the Melting Process when Adding Flux to the Olivine Pellets

Both MPBO and KPBO pellets contain high MgO, but with a low ratio of CaO/SiO₂, quite different with that of self-fluxed pellets. Table 8 shows the chemical composition of some slags supposed to be formed during melting process when using pellet MPBO. Bosh slag 1 and 2 represent the slags adding 5 and 10% BOF-slag, respectively. The primary slag has an estimated melting point about 1600°C, point M in Figure 13-a. When adding 5 or 10% of BOF-slag, the basicity of slags formed increases as shown in Table 8. Accordingly, the melting point of slag decreased to be about 1350°C and 1550°C, as points M1 and M2 of Figure 13-a. Therefore, adding 5% BOF-slag can considerably improve the melting property. However, adding 10% gives less effect. Further increase CaO content may increase its melting point dramatically, due to the increase of the slag basicity.

Enhancing the Melting of Coke and Coal Ashes when Adding Flux

The coke and coal ashes contain mainly the acid components, Al₂O₃ and SiO₂. Their melting points are generally high, also with a very high viscosity. For instance, the melting point of a specimen, corresponding to a tuyere slag formed by a mixture of coke and coal ashes was about 1600°C. When adding flux to the specimen, the acid components would be neutralised. The basicity of the slags formed would be increased with increase of the additions. When using “high” or “very high” level addition, the basicity B₂ would be around 1.0, much close to a normal final slag in the blast furnace. Its estimated melting point is only about 1300°C, much lower than the original one. In additions, we can expect that its viscosity would be also quite low.

The BOF-slag has a different effect compared to lime and dolomite on lowering the melting point of the coke and coal ashes. This may be due to the differences mainly in MgO and Al₂O₃ content of the specimens and contents of some other oxides, as for example iron oxides and manganese oxides, in the BOF-slag.

Influence of Fluxes on Slag Formation Process

The slag formation in the blast furnace is a complex process, involving the softening and melting of burden, dissolution of slag formers and additives in the primary slag, absorption of ashes of fuels by bosh slag, and various chemical reactions, etc.

The major objective of using fluxes in the blast furnace process is to neutralise the gangue of iron ores and ashes of fuels. However, most of ash of fuels releases only at the time when fuels are burnt in front of tuyeres. Therefore, the bosh slag has a higher basicity than that of the primary and final slags. When the basicity of bosh slag is high, considerable changes of the slag property during the transformations from primary slag to bosh slag and from bosh slag to final slag occur. Obviously, the influence of flux on the slag formation process is dependent on iron-bearing burden used and the way of supplying flux into the blast furnace process.

When acid pellets are used, the top-charged flux would neutralise the acid components in the burden, generally improving its melting properties until reaching a certain limit, above this limit the slag property may still become worse. Contrary, when self-fluxed pellets are used, the top-charged flux would generally make the bosh slag an excessive basicity, giving a quite
negative effect on melting properties. In order to avoid an excessive basic slag formed at the cohesive zone and a very acid slag formed in front of tuyeres, injection of certain amount of flux into the blast furnace can be a good solution.

Conclusions
The influences of the fluxes on the softening and melting properties of LKAB self-fluxed and olivine pellets and the melting point of coke and coal ashes have been studied experimentally in the laboratory. The test results showed that adding flux to the self-fluxed pellets could worsen the softening and melting property of the pellet; while adding flux to the olivine pellets could improve softening and melting property until to a certain limit. Adding flux to the specimen of the coke and coal ashes could lower its melting points significantly.

Therefore, when self-fluxed iron-bearing burden e.g. self-fluxed pellets are used the top-charged fluxes will give very negative influence on the slag formation process in the blast furnace. On the contrary, when olivine pellets are used, the top-charged flux will generally improve the slag formation process.

Irrespective of the types of iron-bearing burden used, injection of flux will improve the tuyere slag property, thus improving the raceway conditions.

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References


Figures

Figure 1. Schematic view of experimental apparatus

Figure 2. Placement of samples in the furnace tube

Figure 3. Definition of the softening temperature
Figure 4. Schematic view of the high temperature microscope for measuring the melting point

Figure 5. Changes of contour of the specimen during a test

Figure 6. Three different placements of BOF-slag in pellets bed, a) layered pellets and BOF-slag; b) even mixture; c) uneven mixture.
Figure 7. Softening and melting temperature of different pellets tested (pre-reduction degree of pellet = 75%).

Figure 8. Variations of softening and melting temperature with the changes of pre-reduction degree of pellets.

Figure 9. Melting properties of Pellet A with three different placements of BOF-slag (5%).
Figure 10. Variation of the Softening and melting temperatures of MPBO and KPBO with the addition of BOF-slag (Pre-reduction degree of pellets = 75%).

Figure 11. Variations of the softening and melting temperatures of slag with the increase of BOF-slag addition; C: Coke ash; C': Coal ash; L, M, H, VH represent four levels of flux injection; BOF: BOF-slag.

Figure 12. Melting temperature of specimens with changes of flux added
Figure 13. Phase diagrams

a) System of CaO-SiO2–MgO–Al2O3 system [13]

b) System of CaO-SiO2-FeO [14]
### Tables

**Table 1. Summary of test conditions**

<table>
<thead>
<tr>
<th>Sample bed diameter</th>
<th>40 mm</th>
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<tr>
<td>Sample bed height</td>
<td>55 ~ 60 mm</td>
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**Particle size**

- Pre-reduced pellets: 9 ~ 13.5 mm
- BOF-Slag, burnt dolomite and limestone: 3 ~ 6 mm
- Coke breeze: 6 ~ 9 mm

**Pellets weight of each test**

- ~100 g

**Nitrogen gas flow**

- 7 l/min

**Heating rate**

- 200 °C/h

**Pre-reduction degree of pellets**

- 60, 75, 90, %

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**Table 2. Pre-reduction degree of BOF-slag**

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<tr>
<td>Reduction degree of pellets, %</td>
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<td>21</td>
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<tr>
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<td>75</td>
<td>23</td>
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**Table 3. Chemical analysis of materials used**

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<th>MgO</th>
<th>Al₂O₃</th>
<th>Fe₂O₃</th>
<th>FeO</th>
<th>TFe</th>
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**Table 4. Injection rate of flux and coal, kg/THM**

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* THM: Tonne of hot metal
Table 5. Composition of the specimens used (wt%)

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<td>Medium</td>
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<td>Medium</td>
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<td>4.29</td>
<td>6.07</td>
<td>8.08</td>
<td>9.16</td>
<td>1.36</td>
<td>1.41</td>
<td>1.46</td>
<td>1.49</td>
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<td>18.84</td>
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<td>34.67</td>
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<td>0.51</td>
<td>0.89</td>
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</table>

No, Low, High, Medium, Very high: represent the injection levels of flux.

B₂=CaO/SiO₂; B₄=(CaO+MgO)/(SiO₂+Al₂O₃) on molar basis.

Table 6. Softening and melting test results of BOF-slag

<table>
<thead>
<tr>
<th>No.</th>
<th>Sample</th>
<th>Crucible</th>
<th>Coke</th>
<th>$T_1$, °C</th>
<th>$T_{sd}$, °C</th>
<th>$T_{max}$, °C</th>
<th>Dripping</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>BOF-slag</td>
<td>Alumina</td>
<td>No</td>
<td>1343</td>
<td>1400</td>
<td>1520</td>
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<td>2</td>
<td>BOF-slag</td>
<td>Graphite</td>
<td>Yes</td>
<td>-</td>
<td>-</td>
<td>1500</td>
<td>No</td>
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</tbody>
</table>

*: The maximum temperature reached during the test.

Table 7. Chemical composition of slag with pellet A

<table>
<thead>
<tr>
<th>Slag</th>
<th>Composition, wt%</th>
<th>CaO/SiO₂</th>
<th>(CaO+MgO)/SiO₂</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>CaO</td>
<td>SiO₂</td>
<td>MgO</td>
</tr>
<tr>
<td>Primary</td>
<td>30.37</td>
<td>30.37</td>
<td>7.99</td>
</tr>
<tr>
<td>BOF</td>
<td>41.09</td>
<td>9.84</td>
<td>12.97</td>
</tr>
<tr>
<td>Bosh 1</td>
<td>43.92</td>
<td>23.51</td>
<td>12.96</td>
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<tr>
<td>Bosh 2</td>
<td>41.83</td>
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<td>12.35</td>
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<tr>
<td>Bosh 3</td>
<td>39.67</td>
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<td>Bosh 4</td>
<td>47.36</td>
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<td>14.36</td>
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</table>

Table 8. Chemical composition of slag with pellet MPBO

<table>
<thead>
<tr>
<th>Slag</th>
<th>Composition, wt%</th>
<th>CaO/SiO₂</th>
<th>(CaO+MgO)/SiO₂</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>CaO</td>
<td>MgO</td>
<td>SiO₂</td>
</tr>
<tr>
<td>Primary</td>
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<tr>
<td>BOF</td>
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<td>9.84</td>
<td>12.97</td>
</tr>
<tr>
<td>Bosh slag 1</td>
<td>27.13</td>
<td>26.49</td>
<td>34.76</td>
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<tr>
<td>Bosh slag 2</td>
<td>36.63</td>
<td>23.25</td>
<td>30.53</td>
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