Lime Dissolution in Foaming BOF Slag

JOHAN MARTINSSON, BJÖRN GLASER, and DU SICHEN

The paper describes the dissolution mechanisms of lime into liquid and foaming slags relevant to the BOF process. Two different master slags are employed, representing two different periods of the converter process: an early stage where the FeO content is fixed to 45 wt pct, and a later stage where the FeO content is fixed to 25 wt pct. For these master slags, the ratio between CaO/SiO₂ is varied to examine the effect of basicity on lime dissolution. Calcium silicates are formed and peeled off, or partially peeled off, from the interface between the lime cube and the slag in all cases. The main difference for the dissolutions in pure liquid slag and foaming slag is the controlling step for dissolution. In liquid slag, the controlling mechanism is the removal of the calcium silicate layers, while in foaming slag, the controlling mechanism is the contact area between the lime and the liquid slag phase of the foam. The strong convection in the foam enhance the dissolution process, in some cases, the lime even dissociates into small pieces.

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I. INTRODUCTION

THE dissolution of lime into BOF slags has been the topic for several researchers and is of great interest for the steel industry.^[1–14] The dissolution of lime and consequently the increase of slag basicity enhances the dephosphorization process. An increase in basicity is also important to obtain a lower viscosity of the slag. A very acidic slag would increase the size of the silica ions and increase the viscosity, which in turn has shown to decrease the foaming ability of the slag.^[15,16]

It is well documented that the CaO content increases rapidly in the beginning of the BOF process and increases slowly after approximately 5 minutes of the blowing time.^[17] It is also described that foam starts to form profoundly after approximately 3 to 5 minutes of the process. The industry is interested in good data of the lime dissolution to develop/improve their process models for better process control.

Evans *et al.* and Deng *et al.* studied the dissolution mechanisms of lime in different liquid BOF slags containing CaO-FeO-SiO₂.^[1,7–10] It was found that the lime reacts with SiO₂ in the liquid slag, forming solid calcium silicate layers, 2CaO·SiO₂, in the interface between lime and slag. The controlling dissolution mechanism is the removal of these interfacial layers by shear stress, irrespective of the initial CaO content in the

slag. It was also found that slag with higher FeO content dissolves the CaO faster. The FeO penetrates into the lime and helps the detachment of the calcium silicate layers. On the other hand, when the FeO content is low, the calcium silicate layer is dense. The dense layer protects the lime from slag penetration and therefore decreases the dissolution rate.^[7] Maruoka *et al.* studied the possibility of using quicklime where a core of CaCO₃ remained. It was suggested that the decomposition would enhance the dissolution process since the generated CO₂ gas would help to carry away the calcium silicate layer.^[14]

To the best knowledge of the present authors, no laboratory study on lime dissolution in foaming slag has been reported. By addition of carbon saturated iron into a FeO containing slag, a massive gas evolution is generated. The aim of this work is to find out if there is a difference between lime dissolution in foaming slag compared to its dissolution in pure liquid slag.

II. EXPERIMENTS

The slag evolution in the BOF process can be divided into two main periods, *viz.* an early stage when the FeO content is above 40 wt pct, and a later stage when the FeO content has decreased to 20 to 25 wt pct due to the decarburization.^[17] In view of this change in slag composition, two different master slags were studied in this paper, one representing the early period where the FeO content was fixed at 45 wt pct, and a later stage where the FeO content was fixed at 25 wt pct. The CaO content was chosen to vary from 0 to 30 wt pct in both master slags to evaluate how the lime dissolution rate varied at different initial CaO content. The slag

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compositions are listed in Table I. All slags were utilized in the liquid slag experiments, while only slags A, B, C, and D were chosen for the foaming slag experiments, since the foaming in the BOF process starts appreciably after lime has started to dissolve into the slag. Also the theoretical density and dynamic viscosity of the slags are included to the table.^[18] The density, $\rho_{\text{theoretic}}$, is given in (kg/m^3) and viscosity, μ_{dynamic} , in (Pa s). The viscosity data are based on iso-lines from the reference and are therefore given in ranges.^[18]

A. Material Preparations

The FeO was produced by mixing Fe_2O_3 and iron powder with totally 51 at. pct oxygen. The mixture was kept in a closed iron crucible at 1143 K for 60 hours in argon atmosphere. The sintered body was then crushed into small pieces. The formation of FeO was confirmed by XRD analysis.

The CaO powder was calcined at 1173 K for 10 hours. SiO_2 and MnO were dried at 383 K for approximately 24 hours.

Burnt lime was provided by a supplier to TATA Steel, and no further calcination was carried out before the experiments. The lime was cut and grinded into $1.0 \times 1.0 \times 1.0$ cm cubes. The quality of the lime varied and impurities were detected. The density of the cubes was found to vary between approximately 1700 and 2500 kg/m^3 . Cubes with the density of approximately 2000 kg/m^3 were chosen for the experiments to minimize the quality variation.

B. Dissolution of Lime in Liquid Slag

A vertical tube furnace was employed for the experiments of lime dissolution in liquid slag. The setup is schematically described in Figure 1. An alumina tube was used as reaction chamber. The furnace was equipped with super Kanthal heating elements. A type B thermocouple was used to control the furnace temperature. Another thermocouple of type B was inserted from the bottom of the reaction tube and kept just beneath the sample to record the sample temperature.

The slag components were mixed thoroughly into selected composition. In the case of slags 0, A, B, and C, an amount of 30 g mixed powder was put into a molybdenum crucible with an inner height of 45 mm and inner diameter of 30 mm. In the case of slag D, only 20 g of slag could be put in the Mo crucible (due to its low density). The small molybdenum crucibles were then placed in a molybdenum holder which was connected to a steel tube and a lifting system. A molybdenum impeller was inserted inside the steel tube and connected to a stirring motor placed on top of the lifting system. With this arrangement, the slag bath along with the CaO sample could be stirred during the experiments.

The slags were pre-melted for one hour and then quenched to assure that the slags were homogeneous before the lime cubes were introduced into the slag. A lime cube was put on top of the solidified slag and was

then lowered down to a resting position of the reaction tube and kept there for 15 minutes. The temperature of the resting position was just above the melting temperature of the slag. The sample was then lowered down fast to the heating zone at which point the entire slag melted at the same time. The purpose of using the resting position was to ensure a homogeneous temperature in the slag which allows a better control of the melting moment of the slag. This moment could easily be controlled by lowering down the stirring impeller and touch the surface of the slag. The stirring started immediately when the slag was molten and was ongoing for 30, 60, or 180 seconds. The stirrer was then raised up from the slag after which the sample was quenched in the cooling chamber.

C. Dissolution of Lime in Foaming Slag

An induction furnace with a water-cooled copper coil was employed to generate the foams, see the experimental setup in Figure 2. The furnace was set to 270 V, 25 A, and 26 kHz during the experiments.

1 g of graphite powder and 7 g of pig iron containing 3.9 wt pct carbon were put in the bottom of a graphite crucible with inner height of 140 mm, inner diameter of 30 mm, and a wall thickness of 10 mm. 67 g of a selected slag was then added on the top of the hot metal. The crucible was painted with Yttrium Oxide paint to avoid heavy oxidation of the graphite during the experiment. The temperature was measured using a calibrated infrared temperature sensor of model thermoMETER CTM-1SF75-C3, on consideration that regular thermocouples would be affected by the magnetic field and could therefore not be used.^[19] The slag was molten approximately 3 minutes after the furnace was turned on and the foaming started a couple of seconds later due the reduction of FeO by the carbon in the hot metal. When the foam was well developed and an appropriate temperature was reached at around 1873 K, a lime cube was dropped into the foam from the top, and a stop watch was started. The furnace was turned off after 30, 60, or 120 seconds and the slag was solidified spontaneously within seconds.

D. Sample Preparation and Examination

Both the samples along with the crucibles from liquid slag and foaming slag experiments were cut horizontally after the experiments. The size of the remaining lime cubes were measured with a pair of calipers. The samples were then mounted to pellets in epoxy. The pellets were grinded and polished in ethanol before examination in a scanning electron microscope (SEM).

SEM was employed for the examination of the interface between lime cube and slag. The pictures were taken with backscattered electrons (BSE). Energy-dispersive X-ray spectroscopy (EDS) was employed for point analysis and map analysis. This information was essential for an understanding of the dissolution mechanisms. Many pores and cracks were detected inside the lime cube. They appear during sample preparation due

Table I. Slag Compositions in Weight Percent

Slag	A	B	0	C	D
FeO	45	45	45	25	25
CaO	30	15	0	30	15
SiO ₂	25	40	40	25	40
MnO	0	0	15	20	20
$\rho_{\text{theoretic}}$	3500	3300	3500	3400	3100
μ_{dynamic}	0.05 to 0.1	0.15 to 0.2	0.16 to 0.24	0.05 to 0.1	0.15 to 0.2

Fig. 1—Experimental setup for liquid slag.

to the poor strength of the lime, and they are easily detected as black areas in the SEM pictures (marked in the figures discussed later).

III. RESULTS

A. Lime Dissolution in Liquid Slag

Figure 3 presents the remaining cube sizes of the lime from the dissolution experiments in liquid slag. As expected, the lime dissolution is fastest in slag 0, where the initial CaO content is zero, and therefore has the strongest driving force for dissolution due to the concentration gradient. The cube is dissolved into the slag already after 60 seconds. The figure also shows that slags A, B, and 0, where the initial FeO content is 45 wt pct, have faster lime dissolution than slags C and D, where the initial FeO content is only 25 wt pct. This result is in good agreement with the literature.^[7] The fast dissolution of lime is due to that FeO facilitates the removal of calcium silicates.

Figures 4(a) through (c) illustrate the dissolution process of a lime cube into slag B. The penetration of slag into the cube is also well brought out by Figure 4(a).

The SEM microphotograph of the interface between lime cube and liquid slag B after 60 seconds stirring is presented in Figure 5. Figures 4 and 5 are in very good accordance with the results of previous works by Evans *et al.* and Deng *et al.*^[1,7,9] As shown in Figure 5, a layer of 2CaO·SiO₂ about 20 to 30 μm in thickness has been formed in the interface between slag B and lime cube after 60 seconds. It should be mentioned that a layer of 2CaO·SiO₂ is detected in all samples after the stirring. On the other hand, the thickness depends on the composition of the slag and the reaction time. Figure 5 also shows how islands of calcium silicates are torn off from the cube surface and brought into the slag phase. The fact that the main population of the small calcium silicate islands are very close to the surface of the cube implies that they are flushed off from the surface of the cube due to stirring. The distance between the small islands and the layer of calcium silicates reveals that

Fig. 2—Experimental setup for foaming slag.

Fig. 3—Dissolution of lime in liquid slag.

these islands have already detached from the layer. One can also see liquid slag containing mostly FeO inside the lime cube. The depth of pure liquid slag could also be seen varying in the samples. Slags A and B had deeper liquid slag penetration than slags C and D, which is also in agreement with the results of the literature.

As seen in Figure 5, some small amounts of $3\text{CaO}\cdot\text{SiO}_2$ are formed in the CaO matrix near the interface. The formation of $3\text{CaO}\cdot\text{SiO}_2$ instead of $2\text{CaO}\cdot\text{SiO}_2$ in this region can be well explained by the high CaO activity ($a_{\text{CaO}}=1$) in the region. The formation of $3\text{CaO}\cdot\text{SiO}_2$ would also consume SiO_2 in the liquid, leading to high FeO content in the penetrating liquid phase as mentioned above. The black areas are (as mentioned earlier) pores of missing lime pieces that was torn off during the sample preparation.

B. Lime Dissolution in Foaming Slag

Figure 6 presents the results of the dissolution of lime cubes in foaming slag. The results show that the dissolution rates in foaming slags A and C are similar to the lime dissolution in the corresponding liquid slags, while the dissolution of lime in foaming slags B and D are faster than in the corresponding liquid slags. The

Fig. 5—Interface between lime cube and liquid slag B after 60 s.

Fig. 4—Dissolution of lime in slag B after (a) 30 s, (b) 60 s, and (c) 120 s.

Fig. 6—Dissolution of lime in foaming slag.

Fig. 7—Dissociated lime cube in foaming slag D after 120 s.

Fig. 8—Upper right corner of lime cube in foaming slag C after 120 s.

dissolution in foaming slag D differs greatly from the trend; the lime cube was found dissociated to small pieces after 120 seconds, see Figure 7. This experiment was repeated several times and the outcome was always the same, which will be discussed later.

The SEM picture shown in Figure 8 reveals a discontinuous layer of gas phase separating big parts of the lime cube from the liquid phase of the slag. This result was obtained with slag C after 120 seconds of reaction time. The presence of the gas phase layer at the interface would reduce the contact area between the slag and the cube.

Fig. 9—Interface between cube and foaming slag B after 60 s.

As the lime dissolves into the slag, an interfacial layer of calcium silicates is found between the cube and the foaming slag, although the layer is thinner compared to the case of pure liquid slag. Figure 9 shows that the interfacial layer of calcium silicate is only approximately $5\ \mu\text{m}$ after reaction with the foaming slag B for 60 seconds. Similar to the case of pure liquid slag, the liquid slag that has penetrated into the CaO matrix contains mostly FeO. The thickness of the penetrated layer is usually less than $100\ \mu\text{m}$.

IV. DISCUSSION

The dissolution mechanisms of lime into liquid BOF slags have been well documented by Evans *et al.* and Deng *et al.*^[1,7–10] These mechanisms have further been confirmed by the present results. An example is shown in Figure 5. As mentioned in the result part, calcium silicates have been formed at the interface between cube and slag, and islands of calcium silicates can be seen leaving the cube into the slag phase. Liquid slag containing FeO, CaO, and small amounts of SiO_2 (and some MnO for slag C and D) can also be seen inside the cube. The formation of $3\text{CaO}\cdot\text{SiO}_2$ has resulted in very low SiO_2 content in the liquid phase among the CaO matrix.

Some studies has shown that these solid calcium silicates are important for the dephosphorization reaction.^[20–24] Since the main focus of this work is the lime dissolution, no phosphorous was added to the system. However, it would be an interesting and valuable continuation to study the mechanisms of dephosphorization in foaming slag and to examine whether the generated gas would also be able to remove the calcium silicate layer with the presence of phosphorous.

The dissolution of lime into liquid slags as functions of slag composition is presented in Figure 3. The rapid dissolution rate observed in Slag 0 is not surprising since the initial CaO content is 0 and thus has the strongest driving force to dissolve the lime cube. It is also shown that the dissolution rates are faster in slags A and B compared to slags C and D, which confirms the theory that a high FeO content dissolves the lime faster.^[7] Li *et al.* showed that both Fe^{2+} and Mn^{2+} ions facilitate the inward diffusion of slag into lime, which increases

the dissolution rate.^[12] The present experiments show a faster dissolution rate when using 45 wt pct FeO instead of 25 wt pct FeO and 20 wt pct MnO (comparing slag A and C). The Fe^{2+} ion is smaller than the Mn^{2+} , and could (along with a less complex slag) perhaps facilitate the inward diffusion, and hence increase the dissolution rate. However, more detailed studies are needed before a reliable conclusion can be made. Industrial experiences using lime particles coated with FeO and/or MnO has also shown that the coating substantially helps the lime dissolution. However, the high cost of the coated lime has hindered the development of this application. In fact, the fast dissolution of FeO-coated lime is in accordance with earlier laboratory observations obtained in the present lab (Prof. Du Sichen, Department of Materials Science and Engineering, Royal Institute of Technology, Stockholm, Sweden, 2018, Private communication). Nevertheless, further studies are strongly recommended to find clarity in the matter.

Comparing the results to the work by Maruoka *et al.*, one major difference can be seen. Maruoka *et al.* found two periods of gas generation from the quicklime during the initial stage after exposure to the slag.^[14] CO_2 was generated by thermal decomposition of CaCO_3 , and continued for totally 150 seconds. Yet in this work, Figure 4 evidently shows that no gas was generated during the experiments in pure liquid slag. The absence of gas in these experiments could be due to the type of lime which might be different compared to the work used by Maruoka *et al.* The decomposition of CaCO_3 was also studied by Deng *et al.* It is worthwhile to mention that the inner part of the sample did not reach the decomposition temperature, even after 180 seconds, when the lime piece was put into a slag at 1823 K.^[10]

Note that the main focus of the present work is to study the difference in lime dissolution mechanisms in foaming slag in comparison with the same in pure liquid slag.

Three main differences have been observed between the dissolution of lime in foaming and liquid slag. In the foaming slag, it is evident that the gas phase interrupts the dissolution reaction of lime as indicated by the following observation:

1. In Figure 8, gas is present at the interface between the cube and the slag. The gas diminishes the contact area between the lime and the liquid phase of the slag.
2. The thickness of the calcium silicate layers at the cube interface are shown to be different between liquid and foaming slag. In liquid slag, the layer thickness in slag B is approximately 30 μm after 60 seconds, while it is only approximately 5 μm in corresponding foaming slag. This aspect is clearly brought out by a comparison of Figures 5 and 9.
3. Figures 5 and 9 also show that the depth of slag penetration is smaller in the foaming slag compared to liquid slag, only 100 μm instead of 200 μm .

The presence of a discontinuous layer of gas between the cube and slag in the case of foaming experiment could be due to the gas accumulation. The higher friction to the gas movement at the solid surface could be the

reason for the formation of this layer. However, this aspect would need a careful and systematic study in the future. Nevertheless, the presence of gas phase between the sample and foaming slag reduces the area of reaction.

The thinner layer of calcium silicate in the case of foaming slag could be explained by the chaotic environment that occurs in the foaming slags. The violent movement of the foam would push the lime cube up and down, which would enhance the detachment of the calcium silicate pieces. In addition, the busting of gas from the cube surface would further help the flushing off of the calcium silicate pieces. It is reasonable to expect that the thinner calcium layer will enhance the dissolution process.

The smaller depth of slag penetration in the foam in comparison with the case of liquid slag would be a direct result of the presence of gas layer and less contact area between the slag and the solid sample. In fact, the less slag penetration is expected to result in lower dissolution of lime. A combination of the above-mentioned three differences would lead to different situations for the different slag compositions and their behavior.

For slag compositions A and C, the dissolution rate is very similar in the foaming slag and liquid slag. Although the reaction might be slightly faster in liquid slag, the removal of calcium silicate layers is faster in the foaming slag. All in all, the dissolution mechanisms seem to even out and in total, the dissolution rates turn out to be the same for both systems. Slag A and slag C both have high basicity and relatively low viscosities. It was found that these slags generate uniform foams in an earlier work.^[15] Because of the violent movement of the foam, as discussed earlier, (1) the faster removal of calcium silicate pieces and (2) the reduce area of the interface seem to compensate each other. The joint effect of the two factors leads to a dissolution rate very similar to the rate in pure liquid slag.

Slag B has high viscosity. It was found that slags with too high viscosity does not generate uniform foams.^[15] The gas phase does not exist as small gas bubbles, but as long gas channels. The cross section of a typical sample containing a gas channel is shown in Figure 10.

Fig. 10—Cross section of sample containing gas channel.

Although some smaller bubbles can also be observed in the figure, the majority of the gas joins the channel with a diameter of approximately 10 mm. The gas channels create a chaotic system where the slag surface oscillates rapidly up and down as the big gas channels rise toward the surface and punctuate the surface allowing the gas to escape. This violent movement of the slag phase would greatly enhance the removal of the calcium silicate layer at the surface of the lime surface. This would explain well the faster dissolution of lime in this “foaming” slag compared to the dissolution in the pure liquid slag.

The chaotic behavior that was generated in slag B was also seen in the case of slag D. However, in “foaming” slag D, the lime dissociated into small pieces after 60 seconds into the experiments. The experiment was repeated several times and the outcome was always the same. The violent movement of the slag due to the formation of gas channels would be responsible for the lime dissociation. The dissociation of CaO generates larger reaction area between liquid slag and the lime. The bigger contact area enhances greatly the dissolution process. This would explain the profound increase of the dissolution of slag D in “foam” slag in comparison with the case of pure slag.

Hence, while the controlling dissolution mechanism in liquid slag is the removal of interfacial layers, the controlling dissolution mechanism in foaming slag is the contact area between the lime and liquid phase of the foaming slag.

The dissolution of lime in the later stage of the BOF process is best represented by the experiments conducted in foaming slag C. During this period, as revealed by the present experimental result, the lime dissolution is very slow, almost no dissolution during two minutes. Note that the CaO content shows a certain increase during this period according to the literature. However, this increase could be mostly due to the decrease of FeO by decarburization. If the amount of FeO decreases, the content of CaO will consequently increase.

The experimental results that might be the most interesting for the lime dissolution process in the BOF furnace is the dissolution of lime cubes in the liquid slag 0 and in the foaming slag B. While the dissolution of lime in pure Slag 0 is relevant to the initial stage of the BOF process (little foaming of slag is present), the dissolution of lime in both liquid slag B and foaming slag B would through some lights on the behavior of lime in the slag a few minutes after the start of the blowing.

V. SUMMARY

Lime cubes were dropped into liquid and foaming slags relevant to BOF process. It was found that the dissolution rate was very similar in slags (A and C) having lower dynamic viscosities. The dissolution in foaming slags B and D, both of which had higher dynamic viscosities, was faster than in the same liquid slags. Lime in foaming slag D dissociated to small pieces after 60 seconds of experiment, which led to even faster

dissolution. While the controlling dissolution mechanism in liquid slag was the removal of interfacial layer, the controlling dissolution mechanism in foaming slag was the contact area between the lime and liquid phase of the foaming slag.

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