Nucleation and Growth of Alumina Inclusion in Early Stages of Deoxidation: Numerical Modeling

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In order to gain a better understanding of deoxidation phenomena, there is a need to develop a model that could involve mass transfer, nucleating and growth kinetics of inclusion to simulate unhomogeneous state at the initial stage of Al deoxidation process. Based on the computation of the model for steel droplet with aluminum at center, it is found that the reaction zone between aluminum and oxygen in liquid steel is located in a limited zone at a certain fixed time and it is moved from center to outmost with the time going. For the zone besides the deoxidizer, i.e. aluminum, the supersaturation degree $S_o$ is very high at first, and then is decreased quickly, and the main part of oxygen in steel is absorbed by nucleation. In the other side, for the outmost zone, the main part of oxygen in steel is absorbed by growth of inclusion and the growth of inclusion lasts for longer time than that of inner zones.

KEY WORDS: alumina inclusion; unhomogeneous state; early stages of deoxidation; model; mass transfer; reaction; nucleation; growth of inclusion.

1. Introduction

Deoxidation of steel using aluminum wire injection and further removal of inclusions plays a crucial role in ladle metallurgy. Oxide inclusions, especially hard aluminates, are known to be harmful to the mechanical properties of the steel products. There is ongoing interest in understanding the forming, growth, removing mechanism of oxides inclusions in liquid steel.

It is important to clarify the absorption mechanism of oxygen into Al$_2$O$_3$ inclusion in early stage of deoxidation, which has a great influence to the morphologies of Al$_2$O$_3$ inclusion in steel.

E. T. Turkdogan$^{1,2}$ used the classical nucleation theories to analyze deoxidation of molten steel and homogeneous nucleation of inclusions. Based on his works,$^{1,2}$ the deoxidation of steel involves the formation of critical nuclei of the deoxidation product, a progress of deoxidation product, and their separation from the molten steel.

Recently, a lot of researchers, such as W.-C. Doo et al.$^3$ L. Zhang et al.$^4$ T. Nakaoka et al.$^5$ and J. Zhang et al.$^6$ have tried to develop a general nucleation-growth model, in which inclusions nucleate from homogeneous molten steel and growth due to the diffusion of oxygen and deoxidizer elements, Ostwald ripening and some collision mechanisms. In their models the population balance equations (PBE) of inclusions are solved, and the particle size distribution (PSD) of inclusions in molten steel at early stage are investigated.

Meanwhile, for the unhomogeneous state at the initial stage of Al deoxidation process, K. Beskow et al.$^7$ develop comprehensive model involving fluid flow, deoxidation kinetics, and removal of deoxidation products to simulate the concentration profiles of oxygen and aluminum during deoxidation by aluminum wire injection. For the deoxidation reaction, the source terms of the concentration controlling equations have been computed assuming that local thermodynamic equilibrium is achieved.

In order to gain a better understanding of deoxidation phenomena, there is need to develop a model that could involve mass transfer, nucleating and growth kinetics of inclusion to simulate unhomogeneous state at the initial stage of Al deoxidation process.

In the present work, observation of Al oxide inclusion in the un-uniform concentration fields at the initial stage of Al deoxidation process in steel droplet has been conducted. A one-dimension spherical coordinate mass transfer and reaction model has been developed to simulate of the concentration profiles of oxygen, aluminum and alumina in steel droplet, and the calculation results are used to analyse the morphology of the Al$_2$O$_3$ inclusions in steel droplet.

2. Model Formulation

The steel droplet with certain initial oxygen and aluminum in the center represents a unsteady state diffusion pattern in hemispherical and semisolidified steel phase, as shown in Fig. 1. Based on one dimension spherical coordinate mass transfer and reaction model the concentration field of the oxygen, aluminum and alumina in the liquid
2.1. Assumptions

The principal assumptions of the model:

(1) The shape of the liquid steel droplet is hemispherical, and the surface and bottom of the droplet are boundary for the calculation.

(2) For convective mass transferring caused by the convecting flow in the liquid droplet, an effective diffusion coefficient is introduced to stand for overall mass transport.

(3) For Al\textsubscript{2}O\textsubscript{3} inclusion, the concentration field of it is calculated with consideration of reaction only. After the supersaturation degree \(S_0\) (\(S_0 = K_{AlO}/K_{eq(O)}\), \(K_{AlO}\) is the product content of oxygen in the liquid steel when \(S_0\) starts to be above the criteria level) is higher than the critical degree, Al\textsubscript{2}O\textsubscript{3} inclusions are nucleated. After the nucleation period, the free oxygen atom in the liquid steel is transferred to the surface of aluminum and then reacted with the aluminum and the reacted alumina is aggregated onto the surface of Al\textsubscript{2}O\textsubscript{3} inclusion and, the control step is the convective mass transfer of reactants.

(4) Aluminium is in the center of steel droplet, and is also hemispheric. In the experiment it is observed that after the aluminium wire is inserted into liquid steel, the Al–Fe alloy with Al content of 30% is soon formed outside of the aluminium wire, but the boundary of liquid steel and Fe–Al alloy is clear in image, the Al content at the boundary is almost fixed, and the shape of the residual Al wire with outside Al–Fe alloy is close to sphere. For simplifying the model, the shape aluminium is assumed as sphere. At the interface of aluminium and liquid steel, [%Al] is taken a fix value according to experiment results.

2.2. Transport Equations

The concentration profiles of O and Al are computed in transient mode. These equations have the following forms:

For AI:
\[
\frac{\partial C_{Al}}{\partial t} = \frac{1}{r^2} \frac{\partial}{\partial r} \left( r^2 D_{eqAl} \frac{\partial C_{Al}}{\partial r} \right) + Sc_{Al} \tag{1}
\]

For O:
\[
\frac{\partial C_{O}}{\partial t} = \frac{1}{r^2} \frac{\partial}{\partial r} \left( r^2 D_{eqO} \frac{\partial C_{O}}{\partial r} \right) + Sc_{O} \tag{2}
\]

For Al\textsubscript{2}O\textsubscript{3}:
\[
\frac{\partial C_{AlO}}{\partial t} = Sc_{AlO} \tag{3}
\]

where, 
\(D_{eq}\) = equivalent diffusion coefficient, m\(^2\)/s

\(Sc\) = the source term
\(r\) = radius, m
\(t\) = time, s

2.3. Equivalent Diffusion Coefficient

\(D_{eq}\) can be calculated with the following relationship:
\[
D_{eq(i)} = m_i D_{(s)} \quad i = Al, O \tag{4}
\]

where, \(m_i\) = equivalent coefficient for convective mass transfer.

According definition of sherwood number, \(m_i = sh + 1\), and sherwood number can be calculated with following equation:
\[
Sh = 0.664 Re^{0.5} Sc^{1/3} \tag{5}
\]

For the liquid steel, Sc number is close to 1 000. As the liquid droplet is soon frozen on the water cooling copper plate within 5 s based on the heat transfer computation, Re number of the semi-solidified liquid steel is very low, in this study its value is taken to one. So the value of \(m_i\) is close to 8.

2.4. Source Term

For the source term, the chemical reaction is considered:
\[
2Al + 3O = Al_2O_3(solid) \tag{6}
\]

The reaction will take place in the area where the supersaturation degree \(S_0\) should be above the critical level (= 14.7, measured by H. Suito\textsuperscript{9}) and the theoretic value is equal to 529), only in 6.58\times10\textsuperscript{-6} s\textsuperscript{-1} the nucleation period is ended, and after nucleation stage, no more nuclei of Al\textsubscript{2}O\textsubscript{3} are formed. For the addition of Al, the following equation defines how fast the Al\textsubscript{2}O\textsubscript{3} inclusion appear and disperse in the liquid steel after the supersaturation degree above the criteria level:

For \(S_0 > 14.7, t - t_c < 6.58\times10\textsuperscript{-6} s\)
\[
Q_O = (C_O - C_{Oe})[1 - \exp(-2.45\times10^5 \times (t - t_c))] \tag{7}
\]

where
\(Q_O\) = the amount of oxygen content removed from liquid steel by nucleation of alumina inclusion, mol/m\(^3\)
\(C_{Oe}\) = equilibrium content of oxygen in liquid steel, mol/m\(^3\)
\(t_c\) = time when \(S_0\) starts to be above the criteria level at that position, s

\[
Sc_{Oe} = \frac{\partial Q_O}{\partial t} \tag{8}
\]

After the period of nucleation, the reactants are absorbed to the inclusion to form Al\textsubscript{2}O\textsubscript{3} through convective mass transfer.
For $S_{0} > 1$, $t - t_{0} > 6.58 \times 10^{-6}$ s

$$S_{c_{A}} = -4\pi \bar{r}_{p}^{2}N_{p} \left( \frac{D_{(0)}}{\bar{r}_{p}} \right) \left( C_{O} - C_{Oe} \right) \cdots (11)$$

$\bar{r}_{p}$ is mean radius of the inclusion, $\bar{r}_{p}$ is taken as 0.4 $\mu$m in the early stage of deoxidation. $N_{p}$ is the number of inclusion per unit volume, in the early stage of deoxidation $N_{p}$ is equal to $5 \times 10^{14}$ m$^{-3}$.\(^{9}\)

The calculation of the aboving equations needs the equilibrium value of [%O] and [%Al]. The equilibrium constant ($k_{eq}$) for reaction (6) can be described as:

$$k_{eq} = \frac{1}{f_{2}^{3}([%Al] - \Delta [%Al])^{2} f_{3}^{3}([%O] - 0.889 \cdot \Delta [%Al])^{3}}$$

.........................................(12)

where $f_{2}$ and $f_{3}$ are the activity coefficients of Al and O, respectively. In this study $f_{2}^{3} f_{3}$ is taken as 1.

2.5. **Condition of Definite Solution**

1. Initial condition

$$C_{O(r,0)} = C_{in}$$
$$C_{Al(r,0)} = 0$$
$$C_{Al,O_{2}(r,0)} = 0$$

.........................................(13)

where $C_{in}$ is the oxygen concentration at initial time, mol/m$^3$.

2. Boundary condition:

At the interface between aluminium and steel, the aluminium content equal to a fix value, based on the experimental data.

$$r = r_{Al}; C_{Al(r,0)} = C_{b}$$

.........................................(14)

where $r_{Al}$ is the radius of aluminium in the center of droplet, m

$C_{b}$ is the fixed aluminium concentration at the interface, mol/m$^3$

2.6. **Numerical Procedure**

The transport equations can be solved using a fixed grid numerical solution procedure. For the results presented in this article, an implicit finite difference scheme has been used to discrete the conservation equations. The implicit equations are handled using inverse matrix algorithm.

3. **Experimental Procedure**

The steel sample used in this experiment is prepared in an induction heating furnace with electrolytic iron.

**Figure 2** shows the experimental setup. The tungsten coil around the silica nozzle is used to heat up the steel sample in the nozzle up to 1 873 K. The interior of the total setup is purged with argon to keep at an inert atmosphere. After the steel sample is melted, the pressure in the silica nozzle is suddenly surged by the hypodermic syringe, and the molten steel is dropped onto a water-cooled copper plate, on which a small aluminum wire is standing to dissolve into the steel.

In order to regulate cooling intensity, the copper plate is covered with MgO plate in some heats of the experiment.

The experimental conditions are listed in **Table 1.** The inclusions in the samples are observed with scanning electron microscopy (SEM).

4. **Results and Discussions**

4.1. **Inclusion in Droplet**

For the dropping experiment, the thin aluminum wire is only partly soluted into the steel, and there is aluminum in the liquid state in the center of droplet. The line EDS analysis results around the interface between the remaining aluminium and steel is shown in **Fig. 3.** From the figure, it is found that the [%Al] is around 5–7 at the liquid steel side.

With digital simulation the solidification process of the steel droplet, which is verified by the location of shrinkage cavity observed by laser microscope. It is concluded that the steel droplet is solidified quickly in 4.3 s on water cooling copper plate, and in 3 s there is half of the droplet is frozen to solid. It takes 9.4 s for the complete solidifying of steel droplet on MgO plate that placed on copper plate, and in 8 s there is half of the droplet is in mushy zone. According to the result of the simulation, quenching solidifying only influences the zone with the distance from the bottom plate less than 1 mm, and for the nucleation and growth time of inclusion is far less than 1 s, the influence of solidification of droplet is small to that of inclusion.

For the observation of inclusion, the droplet is classified...
into 3 locations, i.e. location (a), (b), and (c), Fig. 4. The morphologies of inclusions at each location are listed in Table 2, and the inclusions are classified into 4 types of morphologies, that is, sphere, aggregation, early stage of inclusion growth, and dendrite, as shown in Fig. 5. For the sphere type inclusion, the shape of this type is sphere and the size of this type is often less than 1 μm; for the aggregation type inclusion, the inclusions are consisted of several sphere alumina with the size of 0.1–1 μm, and the tiny alumina are aggregated in the zone with the size more than 1 μm; for the early stage type inclusion, this type of inclusion is in the early stage of growth, the primary dendrite of the inclusion is in the early stage of growth; for dendrite type inclusion, the dendrite of inclusions is fully developed, and the inclusion can be grown into equiaxial alumina or alumina with one-direction dendrite. Based on the observation, from location (c) to (b), the number of early stage inclusion is decreased, and from location (c) to (a), the number of sphere inclusion is increased. At location (b) and (a), there are dendrite inclusions appearing.

The number distribution of inclusions of each type in one viewing field of SEM at all the above three location is also plotted in the Fig. 5(f).

### 4.2. Concentration Profile

To model the concentration profile of the droplet with certain initial oxygen and aluminum in the center, some important parameters are necessary and listed in Table 3 and Table 4.

Figure 6 are the concentration field and supersaturation profile in the liquid steel droplet on the water cooling copper plate with aluminum in the center and initial oxygen content of 200 ppm. In the Fig. 6(e), it is found that as the time moves from 1 to 2 s, the Al–O reaction zone moves from less than 1 to 1.5 mm off the aluminium wire, and the peak value of $SO$ keeps at 12.5, and the sum number of alumina inclusion in one vision field also keeps at 55–60. As the supersaturation degree determines the nucleation rate of inclusions, the area with higher $SO$ value may have more number of inclusions. So the calculation results of the model agrees with phenomenon observed in the actual sample.

From above Fig. 6, it is shown that high aluminium zone is gradually diffused from the center of the droplet to the outsider, while high oxygen zone in the droplet is withdrawn with almost constant speed in the same direction in the same time. At the position where is overlapped by high aluminium and oxygen zone, there is high supersaturation degree, and the high supersaturation zone is moving from center to outsider with time going, while the peak supersaturation degree is decreased with time going. This means that the reaction zone between aluminum and oxygen in liquid steel is located in a limited zone at a certain fixed time and it is moved from center to outmost with the time going.

For the $Al_2O_3$ distribution in the steel droplet, there is a homogeneously distribution zone near the center aluminum zone, then the $Al_2O_3$ content is increased sharply and then there is a peak appearing, and outside of the peak zone the $Al_2O_3$ content is increased slowly with the increasing of the distance from the center, at the edge of the $Al_2O_3$ area its content is decreased sharply.

<table>
<thead>
<tr>
<th>Point</th>
<th>Exp. (90 ppm O)</th>
<th>Exp. (210 ppm O)</th>
<th>Exp. (585 ppm O)</th>
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<tbody>
<tr>
<td>(a)</td>
<td>sp</td>
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</table>

sp : sphere  
ag : aggregation  
esig : early stage of inclusion growth  
de : dendrite
4.3. Nucleation and Growth of Inclusion at Different Location in Liquid Steel

Based on the distribution of Al₂O₃, the droplet can be divided to four regions, as shown in Fig. 7: the zone besides aluminum (I zone), the peak zone (II zone), the middle zone (III zone) and the outmost zone (IV zone). The trend curve of precipitating rate of Al₂O₃ at the typical point (i.e., point A, B, C, D and E) in the each regions have different pattern, as shown in Fig. 8. In Figs. 7 and 8, %Al₂O₃ stands for the weight percent ratio of precipitated Al₂O₃ versus liquid steel.

4.3.1. The Zone Besides Aluminum (I Zone)

The I zone is just beside the center, where there is aluminum, and in this region the alumina content is not very high but is even distributed, as shown in I zone of Fig. 7. The precipitating rate of Al₂O₃ in this region is very high at the start time, but is quickly decreased to zero, as shown in the curve of A point in Fig. 8. For A point in I zone, the ratio of oxygen in steel absorbed by nucleation can reach 74% based on the computation of the model, that means the main part of Al₂O₃ precipitated by nucleation. The curve of the supersaturation (S₀), [%Al] and [%O] versus time at point A (the position of point A is shown in Fig. 7) in I region is shown in Fig. 9. It is found that the [%Al] is increased to 1% in less than 0.5 s, and in the same time the [%O] is decreased to less than 10⁻⁴H₂O. When [%O] is less than 10⁻⁴, the rate of deoxidation reaction is very low. From the Fig. 9, the supersaturation degree S₀ is very high at first, and then is decreased from nearly 1000 to less than 100 in less than 0.3 s.

As the above mention, the nucleation rate of inclusion in this zone is very high, the growth of inclusion is limited in a very short time. The characteristics of inclusion in this region is that the number is high and the mean size is low, main part of inclusion is the sphere type inclusion with tiny size, as shown in Fig. 5(a).
Fig. 6. Simulating the droplet contain 200 ppm oxygen on copper plate.

Fig. 7. The four regions inside the liquid steel droplet.

Fig. 8. The trend of precipitate rate of Al₂O₃ at the typical point.
4.3.2. The Peak Zone (II Zone)

The II zone has the peak of the Al$_2$O$_3$ content (point B in Fig. 7). The reason is that it takes a little time for the aluminum is diffused to this location, in the same time [%O] is not decreased, the supersaturation is also very high at first and the high value of $S_0$ lasts for the time ($>0.8$ s) far more than that of I zone ($<0.3$ s), Fig. 10. All this causes the precipitate rate of Al$_2$O$_3$ is very high and the high value lasts for a while, as shown in Fig. 8 (point B). So the Al$_2$O$_3$ content forms a peak in this region.

In the observation of inclusion morphologies for the droplet in the dropping experiment, it is often found straight-line band of inclusion just beside the aluminium zone, Fig. 11. As the [%O] is decreased to very low level at this region long before solidification, and can not cause Al$_2$O$_3$ precipitation during solidification, this part of inclusion is considered to be formed in liquid steel. This straight-line bands of inclusions, which are often located in the I and II zone based on the SEM images, may be the sign of the reaction zone with high nucleation in I and II zone, as the peak content of Al$_2$O$_3$ makes distribution of inclusion in this zone more visible.

4.3.3. The Middle Zone (III Zone)

The III zone is the one in which Al$_2$O$_3$ level is not very high and is even distributed. From the Fig. 7, the precipitate rate of Al$_2$O$_3$ in this region (point C and D) is not as high as region I and II, but lasting time is obviously longer than that in the former two regions. The supersaturation $S_0$ is not so high, and [%Al] is not very high, Figs. 12 and 13. In this region, after the nucleation of inclusion, there is enough [%O] for continuing growth of the inclusion, and the growth of inclusion plays an important role in this region.

Based on the calculation of the model, the ratio of oxygen absorbed by growth of inclusion is increased to 88% at point C. And from Fig. 6, it is found that the isoconcentration line of [%Al] in this region is very dense, so the inclusion in this region may grow in radial direction, and be-
come a orientated growth dendrite type inclusion just as observation of inclusion morphologies in actual droplet, Fig. 5(e).

4.3.4. The Outmost Zone (IV Zone)

The IV zone is the area in which inclusion is not distributed evenly. From the Fig. 7, the precipitate rate of Al2O3 in this region (point E) is not as high as the inner regions, but the duration time of precipitation is the longest. The supersaturation So is the lowest, [%Al] is not high, and [%O] is high even at the solidification end of the droplet, Fig. 14. According to the model, the ratio of oxygen absorbed by growth of inclusion is increased to 98% at point E. So in this region, after the nucleation of inclusion, there is less nuclei of the inclusion than the inner regions, the growth of inclusion plays an main role in this region, and as low number of nuclei the growth of the inclusion is quit free. And from Fig. 6, it is found that [%Al] in this region is the very low compared with the inner side, so the reaction can not take place in each location, and the inclusion in this region may growth into equiaxed dendritic alumina, Fig. 5(d).

4.4. The Influence of Solidification

As the model is simulating the steel droplet which quickly solidified in copper plates in 4.3 s. The influence of solidification should be considered, as the oxygen can be segregated before the solidification front.

Based on the model proposed by H. D. Brody and M. C. Fleming,10) the segregation ratio of Oxygen (\( R^* = C^*_L / C_0 \), where \( C^*_L \) is the equilibrium content of O in the liquid steel, and \( C_0 \) is the average content of O in the total steel include liquid and solid phases) is increased from 1 to 6, with the solid fraction increased from 0 to 0.9.\(^{11)} \) So the average oxygen content in the liquid phase is about 3 times of the average content in the total steel. In this case, the model is calculated with the initial oxygen content of 600 ppm, which is 3 times of the 200 ppm oxygen content in steel. The calculating results are shown in Fig. 15.

From Fig. 15(a), in the area nearing the aluminium wire and the peak zone the curve is similar to the results of initial oxygen content equal to 200 ppm, but in the middle zone and outmost zone the curve become more flatness, as there is enough high content of oxygen in these two area the nucleation and the \( S_o \) values in all four zones are far above the critical value for nucleation of new inclusions, Fig. 15(b). So the growth of alumina inclusions is not as difficult as that results with the initial oxygen content of 200 ppm. And the peak value of \( S_o \) is decreased with the increase of distance from the aluminium wire, which leads to the decreasion of the number of inclusions with the farther distance from the aluminium. This trend is similar to that of initial oxygen content of 200 ppm, but more obvious.

4.5. The Simulation of Solidification on the MgO Coating Copper Cooling Plate

With the the MgO coating copper cooling plate, the time duration of solidificating process of steel droplet is increased to 9 s, for the MgO plate decreasing the heat transfer flux. The simulation results of the droplet cooled on the MgO plate is shown in Fig. 16. Although the liquid phase lasts more time, the trend of the curve of the simulation results is similar to that case on the copper plate.

5. Conclusion

Alumina inclusion nucleated and growthed has been investigated during rapid solidification process of liquid steel droplet with certain initial oxygen content and aluminum in the center, using the mass transfer and reaction model. The findings are summarized as followings:

1. The reaction zone between aluminum and oxygen in liquid steel is located in a limited zone at a certain fixed time and it is moved from center to outmost with the time going.
2. High aluminium zone is gradually diffused from the center of the droplet to the outsider, while high oxygen zone in the droplet is withdrawn with even pace in the same direction in the same time.
Based on the distribution of Al$_2$O$_3$, the droplet can be divided into four regions: the zone nearing aluminum, the peak zone, the middle zone and the outmost zone.

For the zone besides aluminum, the supersaturation degree $S_O$ is very high at first, and then is decreased quickly, and the main part of oxygen in steel is absorbed by nucleation based on the computation of the model. The nucleation rate of inclusion in this zone is very high, the growth of inclusion is limited in a very short time.

For the peak zone, there is a peak content of precipitated Al$_2$O$_3$. The observed straight-line bands of inclusions may be the sign of the thin located reaction zone, as the peak content of Al$_2$O$_3$ makes distribution of inclusion in this zone more visible.

For the middle zone and the outmost zone, the main part of oxygen in steel is absorbed by growth of inclusion, the supersaturation degree $S_O$ is not very high at first, the growth of inclusion is lasting for longer time. For outmost zone the aluminum content is low, so the reaction can not take place in each location.

Fig. 16. Simulating the droplet cooled on the MgO plate with initial oxygen content of 200 ppm.

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