

INVESTIGATION OF OPTIMUM SLAG FOR REMOVAL OF  $Al_2O_3$  INCLUSION IN STEEL<sup>1)</sup>

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Synopsis: The investigations of molten slags were carried out for making clean steel. To determine an optimum slag system and composition for removal of fine  $Al_2O_3$  inclusion in Al-killed steel, a wettability of molten slag with  $Al_2O_3$  and dissolution rate of  $Al_2O_3$  to slag were measured experimentally. From those results, CaO-CaF<sub>2</sub>- $Al_2O_3$  system slag could be estimated to eliminate  $Al_2O_3$  inclusion effectively. To confirm this estimation, Al-killed steel were made with the flux by a 2.5t vacuum furnace experimentally. This slag could decrease total oxygen content to about 6 ppm in Al-killed steel. Furthermore it was found that basicity of the slag and composition of CaF<sub>2</sub> were also affected to absorption of  $Al_2O_3$  inclusion in an application process.

Key words : Inclusion, Clean steel, Wetting angle, Dissolution rate, Slag, Refining,  $Al_2O_3$  inclusion, Basicity, Secondary Refining,

1. Introduction

In recent years there has been an increased demand for steels containing extremely low levels of nonmetallic inclusion. By reducing the number and decreasing the size of the non-metallic inclusion to extremely low levels, the characteristics of steels, such as toughness, fatigue-resistance and flexible bending, were improved. The bearing industry and its steel suppliers have given a lot of attention to lowering the non-metallic inclusion content of bearing steels.

The purpose of this study is to find the optimum slag composition, in order to remove the inclusion containing  $Al_2O_3$  by absorbing the inclusions into the molten slag. It is considered that the optimum slag has to possess properties, as follows; shown in Fig.1. 1) Small wetting angle between molten slag and solid  $Al_2O_3$ , so that it is easy for the non-metallic inclusion such as  $Al_2O_3$  particles, to adhere to molten slag. 2) High dissolution rate of adhered  $Al_2O_3$  particles in the molten slag.

In this report, the slag system and its composition met above-mentioned demands have been decided by using CaO-CaF<sub>2</sub>- $Al_2O_3$  and CaO-CaF<sub>2</sub>-SiO<sub>2</sub> slag systems.

2. Wettability between molten slag and solid  $Al_2O_3$

In order to clarify the adhesion level of  $Al_2O_3$  particle to the molten slag, the wetting angle between the molten slag and the solid  $Al_2O_3$  has been measured by using a high temperature microscope.

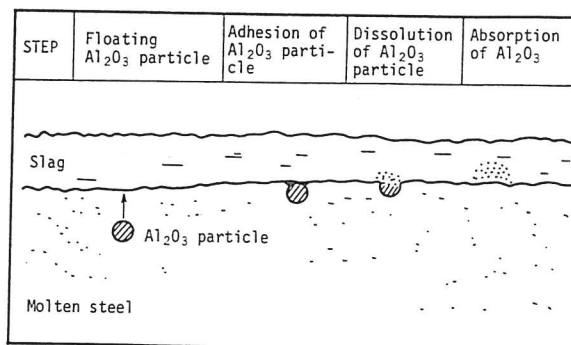


Fig.1 Schematic profile of absorption mechanism of  $Al_2O_3$  particle into molten slag

2-1 Experimental method

A schematic profile of the high temperature microscope is shown in Fig.2. The graphite plate and  $Al_2O_3$  plate were set side by side, as shown in Fig. 3, on the support table which was made of graphite. After melting the mixed flux on the graphite plate, the molten slag was moved to the  $Al_2O_3$  plate. The wetting angle between the molten slag and  $Al_2O_3$  plate were measured by recording photographically the change of molten slag image. Temperatures of slag sample and  $Al_2O_3$  plate were kept at  $1600^\circ C$ . The mixed flux systems were 1)  $CaO-CaF_2-Al_2O_3$  and 2)  $CaO-CaF_2-SiO_2$ .

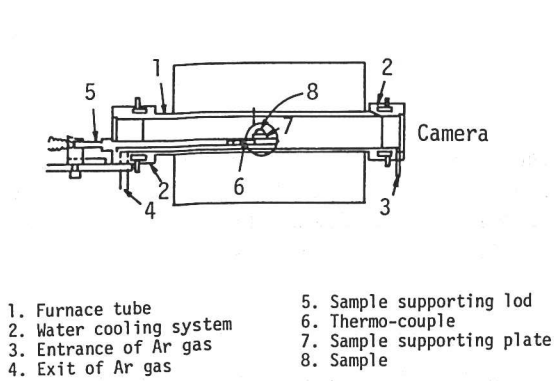


Fig.2 Schematic profile of high temperature microscope

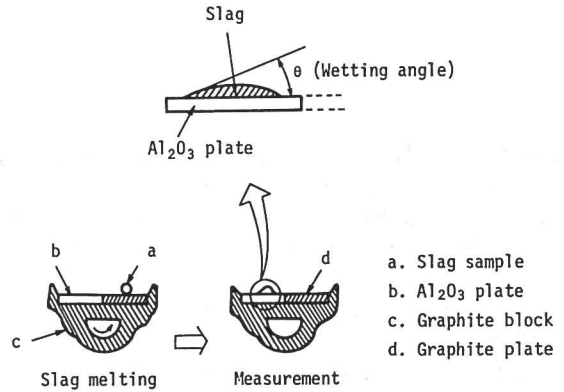


Fig.3 Experimental method of measuring a wetting angle between molten slag and solid  $Al_2O_3$  at  $1600^\circ C$

2-2 Results and discussion

1)  $CaO-CaF_2-Al_2O_3$  system

a) Influence of  $CaF_2$  content in molten slag on the wetting angle( $\theta$ )

When the basicity of the mixed flux was kept at constant, ( $CaO/Al_2O_3=1.0$ ), the behaviours of wetting angle was shown in Fig.4. The wetting angles decreased with time, and the final values reached 15 degree in  $CaF_2=5\%$ , and 10 degree in  $CaF_2=20\%$ . The wetting angle  $\theta$  decreased with the increase of the  $CaF_2$  composition in the molten slag.

b) Influence of the basicity of the molten slag on the wetting angle( $\theta$ )

When the  $CaF_2$  content in molten slag was kept at constant, ( $CaF_2=20\%$ ), the final value of wetting angle reached 10 degree, independent of the slag basicity.

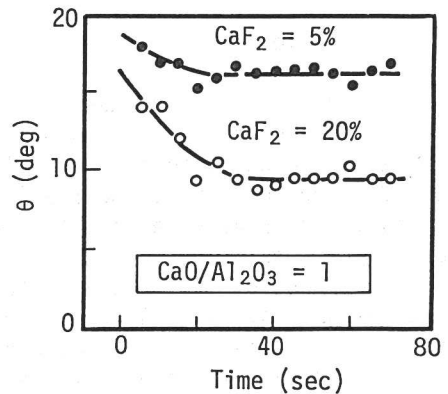


Fig.4 Behaviours of wetting angle ( $\theta$ ) between molten slag and solid  $Al_2O_3$  (Influence of  $CaF_2$  content in  $CaO-CaF_2-Al_2O_3$  slag system on  $\theta$ )

2)  $CaO-CaF_2-SiO_2$  system

a) Influence of  $CaF_2$  content in molten slag on the wetting angle( $\theta$ )

When the basicity of the mixed flux was kept at constant, ( $CaO/SiO_2=1.0$ ), the behaviours of wetting angle was shown in Fig.5. The wetting angles decreased with time, and the final values reached 10 degree, independent of the  $CaF_2$  content in molten slag.

b) Influence of the basicity of the molten slag on the wetting angle( $\theta$ )

When the  $CaF_2$  content in molten slag was kept at constant, ( $CaF_2=20\%$ ), the final value of wetting angle reached 10 degree in  $CaO/SiO_2=1.0$ , and 13 degree in  $CaO/SiO_2=2.0$ . The wetting angle increased with the increase of the basicity of the molten slag. And it was found out that the wetting angle of high basicity slag was quite low level at the very beginning of the contact between the molten slag and  $Al_2O_3$  plate.

- 3) Comparison of two slag systems on the wetting angle between the molten slag and Al<sub>2</sub>O<sub>3</sub> plate.

When the basicity of the slag and the CaF<sub>2</sub> content were the same conditions, 2.0 and 20% respectively, the final value of the wetting angle in the CaO-CaF<sub>2</sub>-Al<sub>2</sub>O<sub>3</sub> system was lower than that of the CaO-CaF<sub>2</sub>-SiO<sub>2</sub> system in Fig. 6.

Table 1 shows the experimental results of measuring the wetting angle between molten slag and solid Al<sub>2</sub>O<sub>3</sub>. As for the adhesion of Al<sub>2</sub>O<sub>3</sub> particle to the molten slag, CaO-CaF<sub>2</sub>-Al<sub>2</sub>O<sub>3</sub> system was selected at the conditions of high basicity and high CaF<sub>2</sub> content.

Table 1 Experimental results of measuring wetting angle ( $\theta$ ) between molten slag and solid Al<sub>2</sub>O<sub>3</sub>

Slag system Condition	CaO-CaF <sub>2</sub> -Al <sub>2</sub> O <sub>3</sub>	CaO-CaF <sub>2</sub> -SiO <sub>2</sub>
Increase of basicity	No significant effect on decrease of $\theta$	Not so good for decrease of $\theta$
Increase of CaF <sub>2</sub> content	Good for decrease of $\theta$	No significant effect on decrease of $\theta$

3. Dissolution rate of Al<sub>2</sub>O<sub>3</sub> to molten slag

3-1 Experimental method

A schematic profile of Tammann furnace is shown in Fig. 7. Al<sub>2</sub>O<sub>3</sub> disk was rotated at a desired speed, 100 rpm in the molten slag which was mixed to 200g and was melted at 1600°C. the dissolution rate was calculated by the change of the Al<sub>2</sub>O<sub>3</sub> disk weight.

1) Al<sub>2</sub>O<sub>3</sub> disk

The shape of the Al<sub>2</sub>O<sub>3</sub> disk was 50.85  $\phi$   $\times$  5<sup>t</sup>, the density was 3.94g/cm<sup>3</sup>, the weight was 38.77g and the porosity was 0.0. Photograph 1 shows the cross section of the Al<sub>2</sub>O<sub>3</sub> disk after an experiment.

2) Calculation of dissolution rate

The dissolution rate was calculated by the following equation which was contained with the weight of Al<sub>2</sub>O<sub>3</sub> disk, the thickness of the disk and the dissolution time.

$$j = (1/2) \{ (W_0 - W_1) / \pi r^2 \rho t \}$$

$$= (1/2) (h/t) (1 - W_1/W_0)$$

- j : Dissolution rate (cm/sec),
- W<sub>0</sub> : Initial weight of disk (g),
- W<sub>1</sub> : Final weight of disk (g),
- r : Radius of disk (cm),
- $\rho$  : Density of disk (g/cm<sub>3</sub>),
- t : Dissolution time (sec)
- h : Initial thickness of disk (cm)

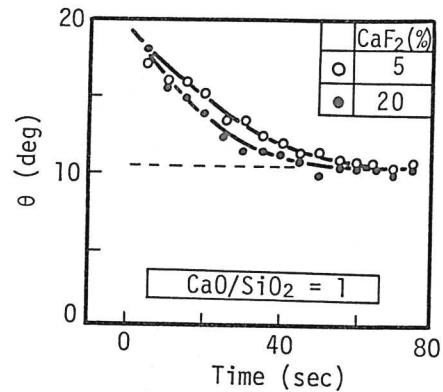


Fig. 5 Behaviours of wetting angle ( $\theta$ ) between molten slag and solid Al<sub>2</sub>O<sub>3</sub> (Influence of CaF<sub>2</sub> content in CaO-CaF<sub>2</sub>-SiO<sub>2</sub> slag system on  $\theta$ )

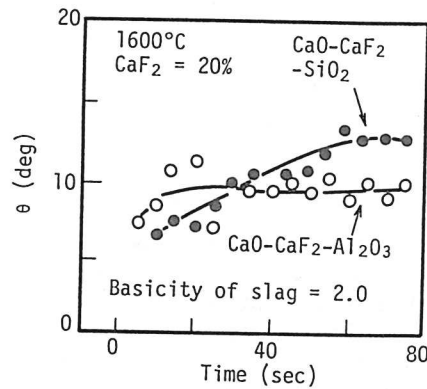


Fig. 6 Influence of slag system on wetting angle ( $\theta$ ) between molten slag and solid Al<sub>2</sub>O<sub>3</sub>

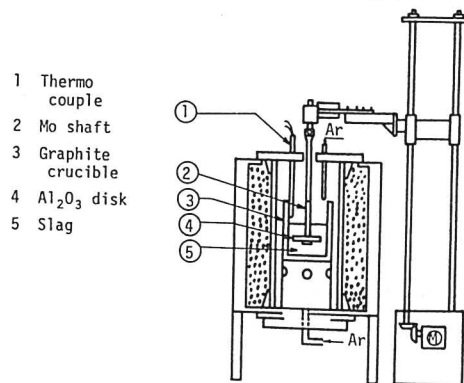


Fig. 7 Schematic profile of tammann furnace for measuring a dissolution rate of Al<sub>2</sub>O<sub>3</sub> into molten slag at 1600°C

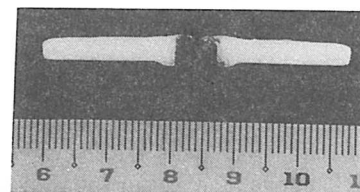


Photo. 1 Cross section of Al<sub>2</sub>O<sub>3</sub> disk after experiment

## 3-2 Results and discussion

The increased value of  $\text{Al}_2\text{O}_3$  content in the molten slag during rotating the  $\text{Al}_2\text{O}_3$  disk was just equal to the calculated value by the amount of  $\text{Al}_2\text{O}_3$  disk dissolution.

1) Influence of rotating speed on the  $\text{Al}_2\text{O}_3$  disk dissolution

It was found out that the dissolution rate of  $\text{Al}_2\text{O}_3$  disk increased in proportion to the square root of rotation speed of the disk<sup>2)-4)</sup> when the rotation speed was less than 100 rpm. And the shape of this disk was not maintained uniformly after experiment at over 120 rpm rotating speed. The experiments were proceeded at the rotation speed of 100 rpm.

2) Influence of slag basicity on the  $\text{Al}_2\text{O}_3$  dissolution rate(j)

Figure 8 shows the influence of slag basicity on the dissolution rate of the  $\text{Al}_2\text{O}_3$  disk into the molten slag,  $\text{CaO-CaF}_2\text{-Al}_2\text{O}_3$  system and  $\text{CaO-CaF}_2\text{-SiO}_2$  system respectively.

In each slag system, the dissolution rate of  $\text{Al}_2\text{O}_3$  disk increased with the increase of slag basicity. And that value in the  $\text{CaO-CaF}_2\text{-Al}_2\text{O}_3$  system was about twice as much as that in  $\text{CaO-CaF}_2\text{-SiO}_2$  system.

3) Influence of the  $\text{CaF}_2$  content in the slag on the  $\text{Al}_2\text{O}_3$  dissolution rate(j)

Figure 9 shows the influence of the  $\text{CaF}_2$  content in the slag on the dissolution rate of the  $\text{Al}_2\text{O}_3$  disk into the molten slag,  $\text{CaO-CaF}_2\text{-Al}_2\text{O}_3$  system and  $\text{CaO-CaF}_2\text{-SiO}_2$  system respectively.

In each slag system, the dissolution rate of  $\text{Al}_2\text{O}_3$  disk increased with the increase of the  $\text{CaF}_2$  content. And that value in the  $\text{CaO-CaF}_2\text{-Al}_2\text{O}_3$  system was about twice as much as that in  $\text{CaO-CaF}_2\text{-SiO}_2$  system.

4) Comparison of two slag systems on the dissolution rate of solid  $\text{Al}_2\text{O}_3$  into the molten slag

When the basicity of the slag and the  $\text{CaF}_2$  content were the same conditions, the dissolution rate of solid  $\text{Al}_2\text{O}_3$  in the  $\text{CaO-CaF}_2\text{-Al}_2\text{O}_3$  system was much higher than that in  $\text{CaO-CaF}_2\text{-SiO}_2$  system.

Table 2 shows the experimental results of measuring the dissolution rate of  $\text{Al}_2\text{O}_3$  disk into the molten slag. As for the dissolution of  $\text{Al}_2\text{O}_3$  particle into the molten slag,  $\text{CaO-CaF}_2\text{-Al}_2\text{O}_3$  system was selected at the conditions of high basicity and high  $\text{CaF}_2$  content.

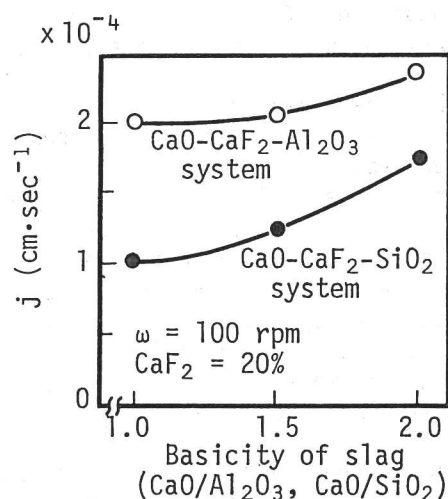


Fig.8 Influence of slag basicity on the dissolution rate (j) of  $\text{Al}_2\text{O}_3$  disk into the molten slag at 1600°C

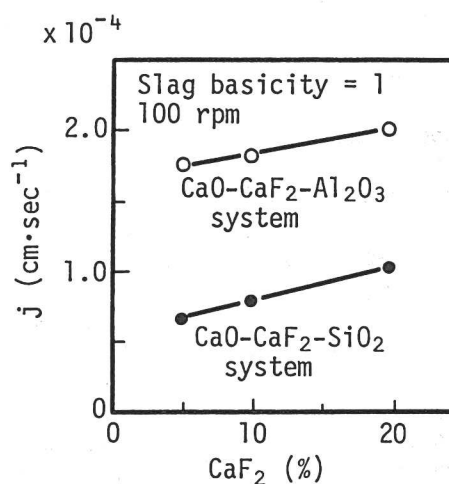


Fig.9 Influence of  $\text{CaF}_2$  content on the dissolution rate (j) of  $\text{Al}_2\text{O}_3$  disk into the molten slag at 1600°C

Table 2 Experimental results of measuring the dissolution rate (j) of  $\text{Al}_2\text{O}_3$  disk into the molten slag

Slag system Condition	$\text{CaO-CaF}_2\text{-Al}_2\text{O}_3$	$\text{CaO-CaF}_2\text{-SiO}_2$
Increase of basicity	Good for increase of j	Good for increase of j
Increase of $\text{CaF}_2$ content	Good for increase of j	Good for increase of j

4. Experiment of removal of  $\text{Al}_2\text{O}_3$  inclusion from Al-killed steel containing 1%-Carbon

In order to confirm the above estimation, the experiment was proceeded by use of a 2.5t induction furnace.

4-1 Experimental method

After the decarburization by use of oxygen gas, deoxidation of molten steel was proceeded by metallic Al, and the mixed flux was put on the molten steel surface.

The flow rate of Argon gas was about  $1 \text{ Nm}^3/\text{min.t}$  and the temperature of the molten steel was controlled at  $1600^\circ\text{C}$ . The lining of this furnace was  $\text{Al}_2\text{O}_3$  brick and  $\text{MgO-Cr}_2\text{O}_3$  brick.

The amount of flux used was  $40 \text{ kg/t}$ . The basicity of the flux was 2.0 and the  $\text{CaF}_2$  content was 20%.

4-2 Results and discussion

Figure 10 shows the behaviours of T.[O] content and  $\text{Al}_2\text{O}_3$  inclusion number in 1%C steel which was covered by each slag system.

In the case of using the  $\text{CaO-CaF}_2\text{-Al}_2\text{O}_3$  system, T.[O] and  $\text{Al}_2\text{O}_3$  inclusion number were reduced to half of these in the case of the  $\text{CaO-CaF}_2\text{-SiO}_2$  system.

5. Conclusion

Optimum slag system and composition was investigated in order to remove the  $\text{Al}_2\text{O}_3$  inclusions by absorbing the inclusions into the molten slag. The best slag system was  $\text{CaO-CaF}_2\text{-Al}_2\text{O}_3$  system at the conditions of high basicity and high  $\text{CaF}_2$  content. The recommended flux-composition was  $\text{CaO}=50\sim 60\%$ ,  $\text{CaF}_2=15\sim 20\%$  and  $\text{Al}_2\text{O}_3=20\sim 25\%$ .

6. References

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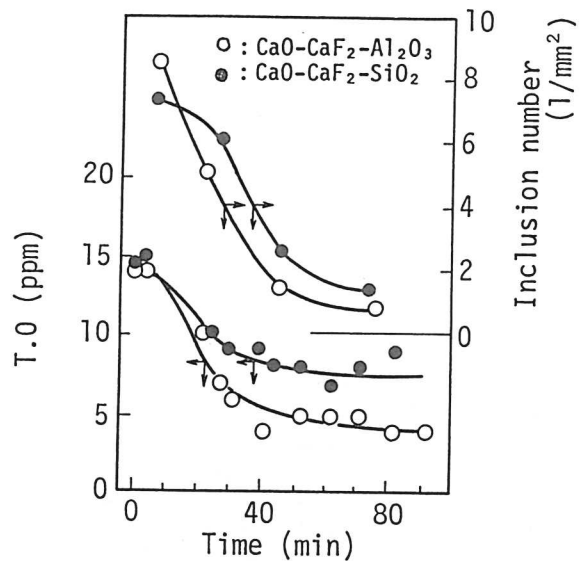


Fig.10 Influence of slag system on the behaviours of T.[O] and of inclusion number in 1%C-steel (2.5t induction furnace lined with  $\text{Al}_2\text{O}_3$  brick)