

# INVESTIGATION OF THE WETTING CHARACTERISTICS OF LIQUID Fe-19%Cr-10%Ni ALLOYS ON THE ALUMINA AND DOLOMITE SUBSTRATES AT 1873 K

**Joonho Lee & Minsoo Shin**

Korea University, Korea

**Joo-Hyun Park**

University of Ulsan, Korea

## ABSTRACT

*The use of dolomite refractory has increased during the recent several years for clean steel processing in the stainless steelmaking. However, the use of dolomite refractory caused the skull formation. In the present work, in order to understand the skull formation, the wetting characteristics of liquid Fe-19%Cr-10%Ni alloys on alumina and dolomite substrates in various oxygen partial pressures were investigated at 1873 K using the sessile drop technique. It was found that the wetting index,  $(1+\cos\theta)$ , of dolomite is approximately 40% higher than those of alumina. In addition, the oxygen partial pressure to generate surface oxide, which may capture the liquid metal on the refractory surface, for dolomite is much lower than that for alumina. From the present study, it was concluded that dolomite is much easier to yield skull formation than alumina due to stronger wettability as well as surface oxide formation at a lower oxygen partial pressure.*

## INTRODUCTION

Clean steel technology requires precise adjustment of metal composition. Particularly, concentration of critical elements such as carbon and nitrogen can be changed by a small amount of skull generated on the refractory surface in the previous steelmaking process. Therefore, it is essential to understand the formation mechanism of skull on the refractory surface.

The wetting characteristic of liquid alloy on the refractory substrate can be identified by the work of adhesion ( $W_a$ ): the work to separate liquid metal from a refractory.

$$W_a = \sigma_{LS} - (\sigma_L + \sigma_S) = \sigma_L (1 + \cos\theta) \quad (1)$$

where  $\sigma_L$ ,  $\sigma_S$ ,  $\sigma_{LS}$ , and  $\theta$  denote the surface energy of liquid, the surface energy of solid, the interface energy between liquid and solid, and the contact angle. According to Equation 1, it is understood that at a fixed surface energy of liquid metal the work of adhesion would increase by decreasing the contact angle. Therefore, as the contact angle decreases, it becomes more difficult to separate liquid metal from the refractory surface, and easily forms skull. On the other hand, when the liquid metal is exposed to air atmosphere, it is subsequently oxidized, and the contact angle drastically changes [1]. Therefore, the surface of liquid metal attached on the refractory surface will be oxidized during solidification. As the authors concern, there has been no report on the in-situ observation of the oxide formation on a stainless steel surface, causing skull formation on the refractory surface.

In this study, the wetting characteristics of liquid Fe-19mass%Cr-10mass%Ni alloys on alumina and dolomite substrates were examined by using the sessile drop technique under different oxygen partial pressure.

## METHODOLOGY

The apparatus used for the sessile drop measurements consisted of an image analyzing system and a MoSi<sub>2</sub> resistance furnace. A schematic diagram of the experimental apparatus is presented in Figure 1. A high resolution CCD camera (1600 x 1200 pixels) and a halogen lamp were used to capture a clear image of the metal sample on ceramic substrates. The MoSi<sub>2</sub> heating elements were capable of heating the furnace up to 1973 K. The furnace temperature was controlled by a B-type thermocouple. Two quartz windows of 25 mm in diameter were provided on the opposite sides of the furnace for observation of the sample assembly inside the furnace. An alumina reaction tube with an inner diameter of 60 mm and a length of 900 mm was placed horizontally in the furnace. The entire system was capable of operating under vacuum, inert gas, or with gas mixtures.

In the contact angle measurements, a piece of cylinder-type metal sample (approximately 0.3 gram) was placed on an alumina substrate. The sample assembly was placed in the center of the furnace and the system was evacuated to 10<sup>3</sup> Pa. The reaction tube was then filled with purified argon gas. This procedure was repeated for three times, and then H<sub>2</sub>-Ar gas mixture was allowed to flow for approximately 12 hours at a flowing rate of 300 ml/min STP in order to flush the system completely. Then, the furnace was heated to the predetermined experimental temperature, 1873 K. After the attainment of this temperature, the furnace was allowed to stabilize for at least 1 hour. Then, the image of the liquid metal drop was captured by using the high resolution camera.

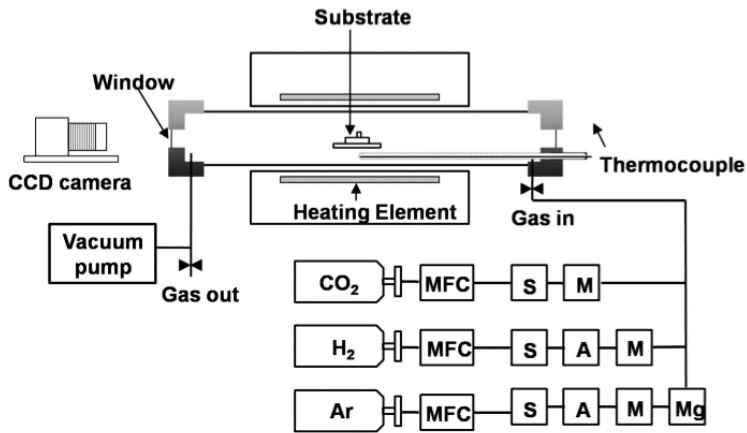


Figure 1: Experimental apparatus of the wetting measurements

In order to investigate the effect of oxygen partial pressure, predetermined  $H_2$ - $CO_2$  gas mixture was allowed to flow at a flowing rate of 300 ml/min STP. After oxide layer fully covered the metal sample, the gas switched to purified argon gas, and the sample was solidified in the furnace by switching off the furnace. The samples after experiments were subjected to SEM-EDX analysis.

## RESULTS AND DISCUSSION

In Figure 2 is shown the contact angle between liquid Fe-19%Cr-10%Ni alloy and solid alumina under different oxygen partial pressure. When the oxygen partial pressure is less than  $4.23 \times 10^{-12}$  atm, the contact angle is almost constant in the range of  $122 \sim 125^\circ$ . As the oxygen partial pressure increases

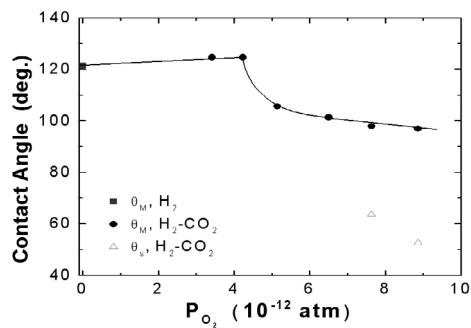


Figure 2: Effect of oxygen partial pressure on the contact angle of a liquid Fe-19%Cr-10%Ni alloy drop on the alumina substrate

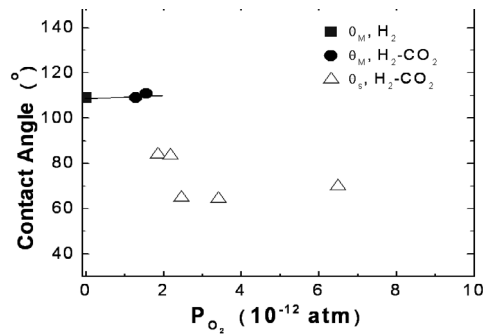


Figure 3: Effect of oxygen partial pressure on the contact angle of a liquid Fe-19%Cr-10%Ni alloy drop on the dolomite substrate

To  $5.14 \times 10^{-12}$  atm, the contact angle suddenly decreases to  $106^\circ$ . As the oxygen partial pressure increases, the contact angle gradually decreases. It is also noteworthy that the oxide formation on the metal surface was observed when the oxygen partial pressure reaches  $7.63 \times 10^{-12}$  atm. The contact angle between oxide and the alumina substrate is as low as  $\sim 60^\circ$ .

In Figure 3 is shown the contact angle between liquid Fe-19%Cr-10%Ni alloy and solid dolomite under different oxygen partial pressure. When the oxygen partial pressure is less than  $1.55 \times 10^{-12}$

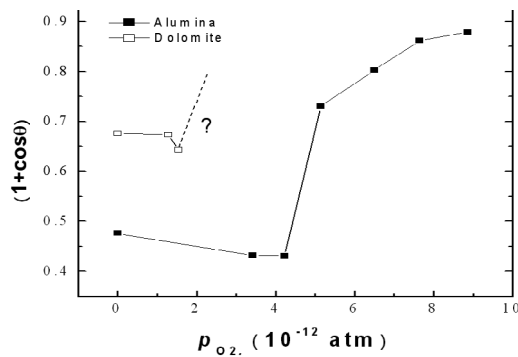


Figure 4: Effect of oxygen partial pressure on  $(1+\cos\theta)$  of a liquid Fe-19%Cr-10%Ni alloy drop on two different substrates: alumina and dolomite

Atm, the contact angle is almost constant in the range of  $109\sim 111^\circ$ . As the oxygen partial pressure increases over  $1.85 \times 10^{-12}$  atm, an oxide layer covers the metal drop and the contact angle measurements could not be carried out. The apparent contact angle between oxide and the dolomite substrate is as low as  $\sim 70^\circ$ .

In Figure 4, the value of  $(1+\cos\theta)$  is plotted as function of the oxygen partial pressure for the two different substrates: alumina and dolomite. It is noteworthy that the value of  $(1+\cos\theta)$  of dolomite is approximately 40% higher than those of alumina. As a result, it is considered that it becomes more difficult to separate liquid metal from the dolomite rather than alumina. Moreover, the oxygen partial pressure to form surface oxide layer on the dolomite is much lower than that on the alumina. Accordingly, it is concluded that the surface oxide formation is much easier for dolomite than alumina. Consequently, it is expected that dolomite forms skull easily than alumina, which is in good agreement with the observations in the practical process.

## CONCLUSIONS

The wetting characteristics of liquid Fe-19%Cr-10%Ni alloys on alumina and dolomite substrates in various oxygen partial pressures were investigated at 1873 K using the sessile drop technique. It was found that the wetting index,  $(1+\cos\theta)$ , of dolomite is approximately 40% higher than those of alumina. In addition, the oxygen partial pressure to generate surface oxide on dolomite, which may capture the liquid metal on the refractory surface, is lower than that on alumina. It is considered that the results of the present study are useful in understanding the mechanism of skull formation after the secondary steelmaking of stainless steels.

## ACKNOWLEDGEMENTS

This study was supported by a grant from the Fundamental R&D Program for Core Technology of Materials funded by the Ministry of Commerce, Industry and Energy, Republic of Korea.

## REFERENCES

- Kapilashrami, E., Jakobsson, A., Lahri, A. K. & Seetharaman, S. (2003). *Studies of the Wetting Characteristics of Liquid Iron on Dense Alumina by the X-ray Sessile Drop Technique*. *Metall. Mater. Trans. B*, Vol. 34B, pp. 193-99. [1]

