Impedance Measurement of Simulated Foaming Slag for Evaluation of Gas Phase Fraction

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The molten CaO-based slags used in modern hot metal pretreatment and steelmaking converters often contain a dispersed gas phase. This is called foaming slag and is created by refining reactions that utilize metallurgical lime. The rheological behavior of foaming slag, which significantly affects the fluid flow of processes associated with the dissolution of lime, has been found to be controlled by the dispersed fraction of the gas phase. In the present study, a simulated slag foam was produced by dispersing inert gas bubbles in various liquids. The effect of varying the volume fraction of the dispersed gas phase on the impedance was then systematically investigated with cylindrical configurations of electrodes. The following equivalent circuit analyses on the semicircular Nyquist plots indicated that electrodes with foaming slag consisted of a series circuit of the solution resistance, a parallel junction of the double layer capacitance, and the resistance of the electric charge transfer. The Nyquist plots displayed semicircular shapes, and their diameters increased with the gas phase fraction despite the various viscosities of the liquid phases, which corresponded to the increase in the charge transfer resistance. The charge transfer resistances were calibrated on the basis of the increase in the electrode surface area and revealed a good linear relationship against the two-thirds power of the gas phase fraction in an ultrapure water and glycerol solution. This suggests a possible approach to quantitatively evaluating the gas phase fraction of a foaming slag by measuring the impedance.

KEY WORDS: foaming slag; impedance; equivalent circuit; gas phase fraction.

1. Introduction

As part of the steelmaking process associated with the decarburization reaction of molten iron and steel, a massive amount of carbon monoxide gas is generated at the interface of the molten iron and slag phases. This is the result of a reaction between oxygen in the slag phase and carbon in the metal phase, which is the main impurity of hot metal produced in a blast furnace. The generation of carbon monoxide gas creates both liquid and gas phases in the slag, which is called foaming slag. This presents a very real hazard of molten slag and steel projection. Accurately predicting the formation behavior and evaluating the foaming height of foaming slag is therefore essential to the safe and efficient operation of a converter and hot-metal pretreatment.

Molten CaO–SiO–FeO-based slags are affiliated with unreacted and undissolved CaO, which is a major refining medium for the desulfurization and dephosphorization of hot metal. Such slags act as a matrix for the dispersion of generated carbon monoxide. The rheological behavior of foaming slag, which significantly affects the fluid flow of processes associated with the dissolution of lime, is controlled by the dispersed fraction of the gas phase. Previous studies have examined the rheological behavior of multiphase gas/liquid fluids under ambient temperatures. Sibree was the first to confirm that the viscosity of a liquid phase is increased by a vapor phase suspension. Later, Uhira measured the viscosity of a silicone oil and bubble suspension with various volume fractions of the dispersed gas phase and similarly reported an increase in viscosity with a multiphase gas/liquid fluid. Recently, some of the present authors have similarly reported that the relative viscosity increases with the volume fraction of the gas phase, and the fluid ultimately transitions from Newtonian to pseudoplastic at a higher gas phase fraction. That was the result of evaluating the rheological behavior of simulated foaming slag, which is affected by a number of different factors including variation in the viscosity of the liquid phase, the size of the gas bubbles, and the shear rate.

Although understanding the physical and interfacial properties (e.g., viscosity, density, and surface tension) of the liquid slag phase is essential because this determines the lifetime of any bubbles, the viscosity of the multiphase gas/liquid slag also represents a critical parameter for understanding the mass and heat transfer phenomena in the converter. Furthermore, quantitatively evaluating the level of foaming slag in the converter process is essential because this is a fundamental controlling factor for the practical operation of steelmaking. This is not easy to measure because of the harsh environment in the steelmaking converter. Thus, previous studies have focused on developing techniques...
to measure the foaming level. Kobayashi et al.\textsuperscript{9)} and Maki et al.\textsuperscript{10)} separately reported the utilization of microwaves to evaluate the foaming level. Yamauchi et al. applied an electrical methodology to detect the foaming level; they employed two parallel electrodes driven under a direct current field.\textsuperscript{11)}

In the present study, experimental apparatuses were constructed to evaluate the level and gas phase fraction of a simulated foaming slag. The impedance technique was utilized under an alternative current field associated with equivalent circuit analyses. The relationships between the alternative current parameters and volume fraction of the dispersed gas phase were then quantitatively investigated.

2. Experimental

Figure 1 shows a schematic of the impedance measurement apparatus. This consisted of an outer cylinder of acrylic pipe with a porous bottom (fine porous alumina, Krosaki Harima Corp.) that allowed N\textsubscript{2} gas bubbles to be generated in the liquid phase contained within. As previously mentioned, CO gas was generated during the actual converter process; however, N\textsubscript{2} gas exhibits similar thermophysical properties to CO gas,\textsuperscript{12–14)} such as the density, viscosity, and specific heat, because of their similar bonding natures. In the present study, therefore, the variation in the gas phase fraction with different gas species was negligible. A Pt-20mass\%Rh alloy rod with a 2 mm diameter and a stainless steel tube with a 0.3 mm thickness and ~40 mm diameter were placed within the outer acrylic pipe in a concentric fashion, which produced a cylindrical electrode configuration. Both were connected to a commercially available impedance analyzer (IM3570, HIOKI E. E. Corp.). The initial immersion depth of the rod electrode was 10 mm, and the flow rate of N\textsubscript{2} gas was controlled by using a mass flow controller (FLOW COMPO, Kojima Instruments Inc.).

The impedances and phase angle difference of the simulated foaming slag with various liquid matrices were measured in a frequency range of 4.0–5 MHz at 1.0 V. The imaginary and real impedances were calculated as follows:

\text{Imaginary Impedance: } Z'' = |Z| \sin \theta \quad \text{(1)}
\text{Real Impedance: } Z' = |Z| \cos \theta \quad \text{(2)}

where \( Z'' \) is the imaginary impedance, \( Z' \) is the real impedance, \( Z \) is the measured impedance, and \( \theta \) is the phase angle difference. Nyquist plots of the imaginary and real impedances were reproduced on the basis of equivalent circuit analyses by utilizing EIS Spectrum Analyzer software.\textsuperscript{15)}

Table 1 presents the electrical and physical properties (i.e., specific resistivity, surface tension, viscosity, and density) of the matrix liquids for the simulated foaming slags. Ultrapure water, 0.01 N KCl aqueous solution, 2-propanol, soluble glass, glycerol, and glycerol aqueous solutions were employed as foaming slag matrices. A manometer set on the sidewall of the outer cylinder provided the volume fraction of the gas phase dispersed in the simulated foaming slag. A schematic diagram is shown in Fig. 1. The detailed calculation procedure for the volume fraction of the dispersed gas phase is reported elsewhere.\textsuperscript{3)}

Figure 2 shows a schematic of the alternative impedance measurement apparatus employed in the present study. It consisted of an outer cylinder of carbon steel that con-

<table>
<thead>
<tr>
<th>Specific resistivity (( \Omega \cdot \text{cm} ))</th>
<th>Surface tension (mN/m) at r.t.</th>
<th>Viscosity (mPa\cdot s) at r.t.</th>
<th>Density (g/cm\textsuperscript{3}) at r.t.</th>
</tr>
</thead>
<tbody>
<tr>
<td>ultrapure water</td>
<td>1.75–1.82\times10\textsuperscript{7}</td>
<td>72.8</td>
<td>1.79</td>
</tr>
<tr>
<td>0.01N KCl aq. solution</td>
<td>9.0</td>
<td>72.8</td>
<td>1.79</td>
</tr>
<tr>
<td>2-propanol</td>
<td>2.5\times10\textsuperscript{3}</td>
<td>62.4</td>
<td>1.77</td>
</tr>
<tr>
<td>soluble glass</td>
<td>–</td>
<td>70</td>
<td>1.43</td>
</tr>
<tr>
<td>glycerol</td>
<td>4.0\times10\textsuperscript{3}</td>
<td>63.4</td>
<td>1.4\times10\textsuperscript{3}</td>
</tr>
<tr>
<td>95% glycerol aq. Solution</td>
<td>–</td>
<td>–</td>
<td>5.2\times10\textsuperscript{2}</td>
</tr>
<tr>
<td>90% glycerol aq. Solution</td>
<td>–</td>
<td>–</td>
<td>2.2\times10\textsuperscript{2}</td>
</tr>
<tr>
<td>80% glycerol aq. Solution</td>
<td>–</td>
<td>64.8</td>
<td>6.0\times10\textsuperscript{2}</td>
</tr>
</tbody>
</table>
obtained a perforated plate of carbon steel, which allowed N₂ gas bubbles to be generated in the liquid phase contained within. A Pt–20mass%Rh alloy rod with a 2 mm diameter was placed within the outer steel electrode in a concentric fashion. This led to a cylindrical electrode configuration. Both were connected to a commercially available impedance analyzer (IM3570, HIOKI E. E. Corp.). The impedances and phase angle difference of the simulated foaming slag with various liquid matrices were similarly measured in a frequency range of 4.0–5 MHz at 1.0 V. This steel apparatus was made with the aim of realizing a practical evaluation of foaming behavior of steelmaking slags at elevated temperatures. The volume fraction of the gas phase was simply estimated on the basis of the height difference between the foaming liquid and uniform liquid. The initial immersion depth of the rod electrode was 10 mm, and the flow rate of N₂ gas was similarly controlled by using a mass flow controller (FLOW COMPO, Kojima Instruments Inc.).

3. Results and Discussion

3.1. Detection of Foaming Behavior by Using the Acrylic Apparatus and Equivalent Circuit Analysis

Figure 3 depicts the Nyquist diagrams calculated from

![Fig. 3. Changes in the Nyquist diagrams of various liquids with the gas phase fraction (vol%) characterized by using the acrylic apparatus: (a) ultrapure water, (b) glycerol, (c) 0.01N KCl aqueous solution, and (d) soluble glass.](image)
the measured impedance of the simulated foaming slags with various gas phase fractions (vol%): (a) ultrapure water, (b) glycerol, (c) 0.01N KCl aqueous solution, and (d) soluble glass. Note that the impedance of the 2-propanol-based foaming slag could not be detected under the present experimental conditions because of its high resistivity. Two different tendencies were found in the Nyquist plots: one was semicircular, and the other was not semicircular but rather linear. For ultrapure water and glycerol, the diameter of the Nyquist plot semicircle increased with the gas phase fraction within the liquid matrices. This can be attributed to the variation in the electric characteristics of the liquid matrices when the dispersed gas phase was present. For the ultrapure water, the scatter in the Nyquist plot increased with the gas phase fraction because of the unstable nature of the dispersed gas bubbles at the electrode interface. For the glycerol, however, the scatter was much smaller. For the 0.01N KCl aqueous solution and soluble glass, negligible variations regarding the gas phase fraction and linear dependencies were found. This was attributed to the lower resistivity of those liquid matrices.

Equivalent circuit analyses were performed only on the Nyquist plots shown in Figs. 3(a) and 3(b) for ultrapure water and glycerol. This is because the Nyquist plots shown in Figs. 3(c) and 3(d) for 0.01N KCl aqueous solution and soluble glass revealed little variation with the gas phase fraction. Figure 4 reveals the fitting results for the Nyquist plots of simulated foaming slags with various gas phase fractions (vol%) of (a) ultrapure water and (b) glycerol. The fitting lines successfully reproduced the measured impedances in all cases. The fitting results indicated that the equivalent circuit consisted of a series circuit of the solution resistance (\(R_{\text{sol}}\)), a parallel junction of the double layer capacitance (\(C_{\text{dl}}\)), and the electric charge transfer resistance (\(R_{\text{et}}\)). This is illustrated in Fig. 5. The solution resistance (\(R_{\text{sol}}\)) is the resistance of entire simulated foaming slag, which is represented by the left side intercept of the Nyquist curve at the real impedance axis. The double layer capacitance (\(C_{\text{dl}}\)) is the capacitance of the electrical double layer at the surface of the electrode. The electric charge transfer (\(R_{\text{et}}\)) is the charge transfer resistance at the interface of the electrode and electrical double layer, which is represented by the diameter of the Nyquist plots.\(^{16}\)

Tables 2 and 3 summarize the parameters estimated with the equivalent circuit analyses. In both cases, the

![Figure 5](image)

**Fig. 5.** Schematic diagram of the estimated equivalent circuit.

<table>
<thead>
<tr>
<th>Gas phase fraction (vol%)</th>
<th>Charge transfer resistance (kΩ)</th>
<th>Solution resistance (Ω)</th>
<th>Double-layer capacitance (pF)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>221</td>
<td>1380</td>
<td>97.7</td>
</tr>
<tr>
<td>10</td>
<td>233</td>
<td>1390</td>
<td>96.2</td>
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<td>20</td>
<td>251</td>
<td>1280</td>
<td>91.6</td>
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<tr>
<td>30</td>
<td>265</td>
<td>1420</td>
<td>87.9</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Gas phase fraction (vol%)</th>
<th>Charge transfer resistance (kΩ)</th>
<th>Solution resistance (Ω)</th>
<th>Double-layer capacitance (pF)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>18.5</td>
<td>19.0</td>
<td>190</td>
</tr>
<tr>
<td>10</td>
<td>25.7</td>
<td>24.0</td>
<td>206</td>
</tr>
<tr>
<td>20</td>
<td>29.1</td>
<td>20.0</td>
<td>149</td>
</tr>
<tr>
<td>30</td>
<td>33.5</td>
<td>23.0</td>
<td>139</td>
</tr>
</tbody>
</table>

![Figure 4](image)

**Fig. 4.** Typical examples of fitting results after the equivalent circuit analyses: (a) ultrapure water and (b) glycerol.
charge transfer resistance increased and the double layer capacitance decreased as the gas phase fraction increased. This was attributed to the decrease in surface area of the electrode with the presence of the gas phase at the electrode surface. As the electrode area decreased, the charge transfer resistance increased in accordance with Ohm’s law (cf. Eq. (3)). The double layer capacitance decreased with the electrical double layer owing to the presence of the gas phase at the electrode surface.

\[ R = \frac{V}{I} = \frac{\rho l}{S} \] ............................... (3)

Here, \( R \) is the electrical resistance, \( V \) is the voltage, \( I \) is the current, \( \rho \) is the electrical resistivity, \( l \) is the length of the conductor, and \( S \) is the surface area of the conductor. However, the solution resistance revealed no significant dependence on the gas phase fraction and was found to be two or three orders of magnitude smaller than the charge transfer resistance. Thus, the charge transfer resistance was considered for further examination, as discussed in the next section.

### 3.2. Detection of Foaming Behavior with the Steel Apparatus

Figure 6 shows the Nyquist diagrams calculated from the impedance measured with the steel apparatus of simulated foaming slags with various gas phase fractions (vol%): (a) ultrapure water, (b) glycerol, (c) 0.01N KCl aqueous solution, and (d) soluble glass. The impedance of the 2-propanol-based foaming slag again could not be measured under the present experimental conditions because of its high resistivity. Similar to the Nyquist diagrams characterized with the acrylic apparatus, two different tendencies were found. For ultrapure water and glycerol, the diameter of the Nyquist plot semicircle increased with the gas phase fraction within the liquid matrices, which can be attributed to the variation in electric characteristics of liquid matrices when the dispersed gas phase was present. The scatter in the Nyquist plot also increased with the gas phase fraction for the glycerol, but the scatter for ultrapure water was modest. This indicated the opposite tendency described in the previous section with the acrylic apparatus and would be due to the difference in interfacial reaction mechanisms. For the 0.01N KCl aqueous solution and soluble glass, negligible variations with the gas phase fraction and rather linear dependencies were found. This was attributed to the lower

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**Fig. 6.** Changes in the Nyquist diagrams of various liquids with the gas phase fraction (vol%) characterized by using the steel apparatus: (a) water, (b) glycerol, (c) 0.01N KCl aqueous solution, and (d) soluble glass.
resistivity of those liquid matrices.

**Figure 7** shows the Nyquist diagrams calculated from the measured impedance of simulated foaming slags (glycerol aqueous solutions) with various gas phase fractions (vol%) and liquid phase viscosities (Pa·s): (a) 1,410 mPa·s (100% glycerol), (b) 520 mPa·s (95% glycerol), (c) 220 mPa·s (90% glycerol), and (d) 60 mPa·s (80% glycerol). Varying the liquid phase viscosity allowed broad changes in viscosity to be simulated for steelmaking slags depending on the temperature and composition. For all concentrations of glycerol aqueous solution, the Nyquist plots displayed a semicircular shape, and their diameters increased with the gas phase fraction. This corresponds to the increase in the charge transfer resistance. Additionally, the diameter of the Nyquist plots and the charge transfer resistance decreased with the viscosity of the glycerol aqueous solution. This can be attributed to the decrease in resistivity of the glycerol solution with increasing H₂O content.

**Figure 8** shows the Nyquist diagrams calculated from the measured impedance of simulated foaming slags (glycerol aqueous solutions + potassium chloride) with various gas phase fractions (vol%) and liquid phase viscosities (Pa·s): (a) 1,410 mPa·s (100% glycerol) + 0.01 N KCl and (b) 60 mPa·s (80% glycerol) + 0.01 N KCl. In addition to the effect of the liquid phase viscosity on the Nyquist diagram of the simulated foaming slag (cf. Fig. 7), the effect of adding an electrolyte to the simulated foaming slag was examined. This is because the electrical resistivity of practical steelmaking slags (CaO–SiO₂–FeO) is generally in the range of $10^{-1}$–$10^{2}$ Ω·cm, which is much lower than that of ultrapure water and glycerol as given in Table 1. As shown in Fig. 8(a), the semicircular shapes of the Nyquist plots and their diameters increased with the gas phase fraction, as expected. This corresponds to the increase in the charge transfer resistance. However, with the KCl addition, the charge transfer resistance was much less than that of glycerol without KCl. This was simply due to the decrease in resistivity of the simulated foaming slag. This tendency was also found in the case shown in Fig. 8(b) and revealed a drastic evolution in the shape of the Nyquist plots. The semicircular parts of the plot were similar and showed a negligible dependence on the gas phase fraction. However, the linear parts of the plot in the low-frequency region appeared as the resistivity of the simulated foaming slag decreased. This is known as the Warburg impedance and is associated with the diffusion process of certain species in a solution. Thus, the proposed impedance technique in the present study can be applied to practical steelmaking slag to

![Fig. 7. Changes in the Nyquist diagrams of the glycerol aqueous solution with the gas phase fraction (vol%) characterized by using the steel apparatus: (a) 1,410, (b) 520, (c) 220, and (d) 60 mPa·s.](image-url)
determine the slag viscosity and resistivity.

3.3. Approaches to Quantitative Analysis of the Gas Phase Fraction

Figures 9(a) and 10(a) show the estimated charge transfer resistance at the interface between the electrodes (steel apparatus, cf. Figs. 6(a) and 6(b)) and ultrapure water and glycerol, respectively, as a function of the gas phase fraction according to the equivalent circuit analyses. The charge transfer resistance drew a convex curve (dotted line) against the linear regression line (solid line). Note that, under the present experimental conditions, the interfacial area between the electrodes and simulated foaming slag increased with the gas phase fraction simply because of the increased height of the foaming slag. As shown in Eq. (3), increasing the interfacial electrode area decreases the resistance, which should be considered for accurate estimation of the gas phase fraction. The height of the foaming slag can be used to express the gas phase fraction as follows:

\[
\phi = \left( \frac{\pi r^2 (h_0 + \Delta h)}{\pi r^2 h_0} - 1 \right) \times 100 = \frac{100 \Delta h}{h_0} \quad \text{(4)}
\]

where \( \phi \) is the volume fraction of the dispersed gas phase, \( r \) is the radius of the outer electrode, \( h_0 \) is the initial height of the slag phase, and \( \Delta h \) is the increment of the slag height after foaming. The change in the interfacial electrode area can be written as

\[
S' = \frac{2\pi r (d + \Delta h)}{2\pi r d} = 1 + \frac{\Delta h}{d} \quad \text{(5)}
\]

where \( S' \) is the increment of the interfacial electrode area associated with slag foaming and \( d \) is the initial immersion depth of the inner electrode into slag phase. Consequently, the calibration factor for the electrode area increment can be rearranged with Eqs. (4) and (5) to become

\[
S' = 1 + \frac{h_0}{100d} \phi \quad \text{(6)}
\]

By multiplying the estimated charge transfer resistivity by the calibration factor calculated with Eq. (6), the decrease in resistivity due to the increase in electrode area can be corrected.

Figures 9(b) and 10(b) show the calibrated charge transfer resistances at the interfaces of ultrapure water and glycerol, respectively, as a function of the gas phase fraction. The
linearity of the data improved but still revealed convex upward curvatures. An additional calibration was applied to the horizontal axis; the volume fraction of the gas phase dispersed. Thus, the charge transfer resistance should be a function of the electrode area as described above; it basically depends on the two-dimensional variation of the electrode area as the gas phase fraction increases. On the other hand, the volume fraction of the gas phase naturally varies in three dimensions. This indicated that the actual increment of the electrode area should be a function of the two-third power of the volume fraction of gas phase. Figures 9(c) and 10(c) reveal the calibrated charge transfer resistance at the interfaces of ultrapure water and glycerol, respectively, as a function of the two-third power of the gas phase fraction. The calibrated charge transfer resistance can be linearly expressed against the two-third power of the gas phase fraction, and the correlation factors ($R^2$) were more than 0.99.

The calibration procedure described above can be applied to the other impedance measurement results. Figures 11(a)–11(c) illustrate the estimated and calibrated charge transfer resistances at the interface between the electrodes (steel apparatus, cf. Fig. 7) and glycerol aqueous solutions as a function of the gas phase fraction and two-third power. The calibration worked well even on the impedance of the glycerol aqueous solution. As shown in Fig. 11(c), the calibrated charge transfer resistances revealed excellent linear relationships ($R^2 > 0.99$) against the two-third power of the gas phase fraction at any viscosity of the glycerol solution. At a higher viscosity of the liquid phase, larger calibrated resistances and a larger slope against the two-third power of the gas phase fraction were found. This can be interpreted as an increase in resistivity in the glycerol solution with less water content.

These calibration results suggest a possible approach to the quantitative evaluation of the gas phase fraction of foaming slag by impedance measurement. However, as stated in Eq. (6), the volume fraction of the dispersed gas phase ($\phi$) was required for the calibration process in the present study. Therefore, improving the impedance measurement system to develop alternative current parameters that do not depend on the variation in electrode surface area will be essential for further application of and high-temperature measurement with the proposed technique in the future.

4. Conclusion

Experimental apparatuses were constructed and used to evaluate the volume fraction of the gas phase dispersed in a foaming slag simulated by ultrapure water and glycerol solutions containing N$_2$ gas under various experimental conditions encompassing those of the steelmaking converter process. The major findings of this study can be summarized
as follows:

(1) Two different tendencies were found in the Nyquist plots calculated from the measured impedances: semicircular and linear depending on the resistivity.

(2) Equivalent circuit analyses on the semicircular Nyquist plots estimated that cylindrical electrode configurations with foaming slag consisted of a series circuit of the solution resistance ($R_{\text{sol}}$), parallel junction of the double layer capacitance ($C_{\text{dl}}$), and resistance of the electric charge transfer ($R_{\text{ct}}$).

(3) The charge transfer resistance increased and the double layer capacitance decreased as the gas phase fraction increased. However, the solution resistance revealed no significant dependence on the gas phase fraction and was two or three orders of magnitude less than the charge transfer resistance.

(4) For all concentrations of the glycerol aqueous solution, the Nyquist plots displayed a semicircular shapes, and the diameter increased with the gas phase fraction regardless of the viscosity of the liquid phase, which corresponds to an increase in the charge transfer resistance.

(5) The calibrated resistances of the charge transfer based on the increasing electrode surface area revealed a good linear relationship with the two-third power of the gas phase fraction in the ultrapure water and glycerol solution. This suggests a possible approach to quantitatively evaluating the gas phase fraction of foaming slag by measuring the impedance.

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