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Uncompensated Resistance Measurement in Molten Salt with Various Electrode Materials

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1. Introduction

Molten salts are promising media thanks to their high melting point, high boiling point, and excellent heat transfer properties They are being investigated for various applications under development, such as pyroprocessing, molten salt reactors, and thermal energy storage. Especially, their high melting and boiling points enable operation at elevated temperatures, which leads to improved thermal efficiency.

However, these high operating temperatures limit the use of conventional sensing techniques due to material compatibility issues. Therefore, most sensing techniques developed for molten salt systems are based on electrochemical methods, as they can operate under high temperature and corrosive environments.

When conducting electrochemical measurements, electrode materials are one of the key factors in terms of stability under such conditions. In addition to durability, electrode materials can also affect the electrochemical signals because additional reactions may occur at the electrode surface [1,2].

Several additional factors must also be considered. The resistance arising from the experimental setup, the solution resistance (R_s), and the intrinsic resistance of the electrode itself can influence the electrochemical signal. The sum of these resistive contributions is generally referred to as the uncompensated resistance (R_u) in electrochemical measurements. When this resistance is large, the charge transfer resistance can be distorted, leading to misinterpretation of the electrochemical response.

In this work, the R_u term was investigated to identify further factors that should be considered in electrochemical experiments. In particular, various electrode materials were employed to examine the effect of their electrical properties. Other parameters, such as the electrical connections, were strictly controlled to isolate the effect of the electrode material. Electrochemical impedance spectroscopy (EIS) was introduced to separate the contributions of charge transfer resistance and R_u.

2. Experimental

All experiments were conducted inside the glovebox filled with argon (Ar, 99.999%) gas. The temperature was controlled at 580°C using an electrical furnace placed under the glovebox.

Anhydrous NaCl (99.999%, Sigma-Aldrich) and $MgCl_2$ (99.99%, Sigma-Aldrich) salts were mixed with the ratio of [NaCl] = 45mol% in this experiment. Glassy carbon cells were utilized as a salt container.

EIS measurements were conducted by the potentiostat (SP-240, BioLogic) with the three-electrode system. Tungsten (W), molybdenum (Mo), and platinum (Pt) rods with 1mm diameter were utilized as working electrodes (WE). W rod with a 1.5mm diameter and Pt rod with a 1mm diameter were used as the counter electrode (CE) and the reference electrode (RE), respectively. The EIS data were measured only in the high frequency region (10^5 Hz to 10^2 Hz) to focus on the R_u part. EIS was conducted with the AC signal (amplitude = 10mV) at the open circuit potential.

One of the important parameters to be measured is the surface area, as the resistance values are normalized to the electrode surface area. To determine the surface area, the immersed depth of the electrode must be accurately measured. In this work, chronopotentiometry was conducted after each experiment to mark the immersed depth. A current of -20 mA was applied for 30 seconds to intentionally deposit magnesium (Mg) onto the electrode surface so that the immersed region could be identified. Fig. 1 shows the electrode after chronopotentiometry. The immersed part was clearly distinguished, allowing the surface area to be calculated as follows: W = 0.12 cm^2; Mo = 0.18 cm^2; and Pt = 0.17 cm^2.

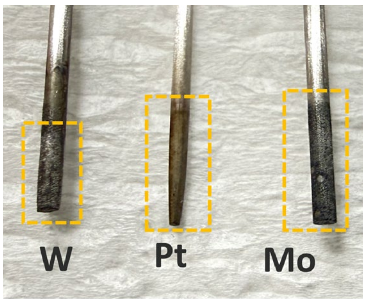

Fig. 1. Surface state of electrodes after chronopotentiometry

3. Results and Discussion

The R_u can be obtained from the Nyquist plot of the EIS results, specifically from the high-frequency x-intercept. Fig. 2 shows the measured Nyquist plots with the various electrodes. The high-frequency x-intercept, corresponding to the R_u, exhibited clearly different values depending on the electrode material.

If the R_s constituted the major contribution to the R_u, the differences would be small because same solution was utilized. However, the significant variation indicates that resistance from the experimental setup and the intrinsic properties of the electrode substantially contribute to the R_u.

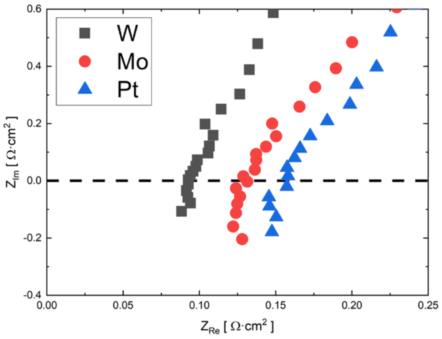

Fig. 2. Nyquist plot as EIS results in NaCl-$MgCl_2$ salts ([NaCl] = 45mol%) measured with different electrode materials at 580°C

To obtain R_u values, the EIS results were fitted using the equivalent circuit shown in Fig. 3. L is the inductance induced by the connection of wires, Q_f is the film constant phase element (CPE), R_f is the film resistance, Q_{dl} is the double layer CPE, and R_{ct} is the charge transfer resistance [3].

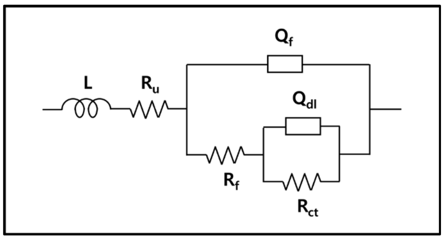

Fig. 3. Equivalent circuit used for fitting of EIS data

The R_u values obtained from the fitting results are summarized in Table I. The R_u values exhibit a clear difference, with nearly a 50% variation between the W and Pt electrodes.

Solution conductivity was utilized to compare the R_u and R_s quantitatively. The relationship between solution conductivity and R_s is written in Equation 1 [4].

$$Solution\ conductivity = \frac{L}{A \times Solution\ Resistance\ (R_S)} \quad (1)$$

L is the distance between the WE and the RE, which is 0.8cm in this experiment. A is the surface area of the WE. The solution conductivities calculated from the R_u values are listed in Table I under the assumption that the R_s is the sole contribution to R_u.

The specific conductivities of NaCl–$MgCl_2$ binary solution were reported by K. Grojotheim et al.; however, the investigated NaCl–$MgCl_2$ molar ratios and temperature ranges were limited [5]. Despite the limited dataset, the reported solution conductivities of the NaCl–$MgCl_2$ binary systems ranged from 1.4 to 3 $\Omega^{-1}\cdot cm^{-1}$ under the examined conditions [5]. These values show a significant difference compared to the solution conductivities calculated in this work, indicating that R_s is not the dominant contribution to R_u.

Table I: Solution resistance and solution conductivity of NaCl-$MgCl_2$ salts ([NaCl] = 45mol%) measured with different electrode materials at 580°C

Electrode Materials	R_u [$\Omega\cdot cm^2$]	Solution Conductivity [$\Omega^{-1}\cdot cm^{-1}$]
W	0.90×10^{-1}	8.94
Mo	1.23×10^{-1}	6.49
Pt	1.43×10^{-1}	5.59

Several factors must be examined to identify the origins of the observed difference. The first factor to consider is the intrinsic properties of the electrode material. Each electrode has its own electrical resistivity. Although metal electrodes are generally regarded as highly conductive materials, their conductivities vary significantly depending on the material. As shown in Table II, W and Mo exhibit similar resistivity values, whereas Pt shows lower conductivity [6]. Since the uncompensated resistance is not measured strictly at the electrode-electrolyte interface, the intrinsic resistance of the electrode can contribute to the measured uncompensated resistance.

Table II: Resistivity of metal at 580°C [6]

Electrode Materials	Resistivity [$10^{-8}\ \Omega\cdot m$]
W	~20.19
Mo	~19.08
Pt	~29.02

In addition, the CE and the RE can influence the measured resistance. A typical electrochemical cell consists of a three-node system that includes a WE, a CE, and an RE. S. Fletcher converted the three-node system into an equivalent two-node representation to analyze the resistance measured at the WE [7]. It was shown that the resistance between the CE and RE, as well as the intrinsic resistance of the RE, can be included in the resistance commonly regarded as the resistance between the WE and RE. Therefore, the overall electrode configuration can affect the resistance of interest.

4. Conclusions

This work conducted the EIS experiments with W, Mo, and Pt electrode as WE to obtain the R_u values in NaCl-$MgCl_2$ ([NaCl] = 45mol%) molten salts at 580°C. It was found that the R_u values showed significant differences depending on the electrode material. Solution conductivity calculation confirmed that R_s is not the only factor that contributes to R_u. These results highlight that multiple factors must be carefully controlled to avoid misinterpretation of the results, including electrode materials and electrode configurations.

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