Oxygen Reduction Activity Indicator for Fuel Cell Catalysts at Rated Voltage

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The available indicators of catalysts for oxygen reduction reaction (ORR) activity are typically characterized at high potentials (commonly at 0.9 V), whereas fuel cells generally operate at 0.85–0.6 V in its practical application. In our work, charge-transfer resistance (Rct) was proposed to evaluate the ORR activity of catalysts for fuel cells at rated voltage. Firstly, the Rct of MEA with Pt/C catalysts was measured in the range of 0.68 V–0.92 V. Tafel slopes calculated from Rct were approximately 80 mV/decade at high potentials, whereas the value increased with the decline of potentials below 0.8 V. It showed that Rct as the ORR indicator of catalysts has certain rationality preliminarily. Secondly, the Rct of MEA with PtₓCoᵧ/C catalysts was compared with Pt/C catalysts. It was lower than that of Pt/C in the high potential region, indicating that the higher ORR activities than that of Pt/C catalysts in higher potential region. Whereas they tended to be the same in the low potential region, indicating that the same ORR activities in rated potential region. Thirdly, Rct of MEA with Pt/C catalysts during durability test was monitored. It was interesting that the Rct increased by nearly 125.6% after 900 hours.

It is generally believed that the catalyst activity severely affects fuel cell performance. As low Pt loadings, along with high performance and long life, are crucially required for commercialization of fuel cells now, the catalyst with higher catalytic activity is urgently needed. U.S. Department of Energy (DOE) proposed technical targets of electro-catalysts for transportation applications, that is, the mass activity of catalyst ≥ 0.44 A/mgPGM@0.9VIr-free.1

However, 2016 DOE annual merit reports2 showed that, pure Pt catalysts and most of the novel catalysts can meet this requirement in the rotating disk electrode (RDE) system at 0.9 V, while the performances of the membrane electrode assemblies (MEAs) with novel catalysts are far from the ultimate targets of low Pt loading and high performance. Although the catalytic activity of Pt alloy catalyst is higher than that of Pt/C, the performance of Pt alloy MEA has no advantage.3,4 Therefore, some researchers think that the activity of Pt has not much to do with fuel cell performance. Debe even put forward his idea that “I therefore consider that realizing target ORR activities may no longer be the most important goal”.5

We believe that there is still a need for prominent catalysts with outstanding activity, more precisely, catalysts with high ORR activity at rated voltage (0.6 V–0.8 V), as shown in Fig. 1. In other words, the available indicators of catalysts for ORR activity may not be suitable.

Characterization of the catalytic activity of oxygen reduction catalysts in the rated voltage range is difficult, because it is hard to distinguish three major polarizations from polarization curve, that is, activation polarization, ohm polarization and mass transfer polarization. Thus we turned to electrochemical impedance spectroscopy (EIS), which can distinguish three polarizations clearly and give information about catalytic activity - charge-transfer resistance (Rct).6

In this work, charge-transfer resistance of Pt/C catalysts in MEA at different potentials were studied, especially in rated voltage range, and verified the rationality of Rct as the catalyst activity indicator with Tafel slope. The oxygen reduction activity of PtₓCoᵧ/C catalysts in MEA was evaluated with Rct and compared to Pt/C catalysts. Rct of MEA with Pt/C catalysts during durability test was monitored.

Fundamental of Charge-Transfer Resistance

Definition of charge-transfer resistance.—The definition of the charge-transfer resistance is:

\[ R_{ct} = \frac{\partial \eta}{\partial j} \]  \hspace{1cm} [1]

Where:

\[ \eta \] is the activation overpotential, \( j \) is the current density.

According to the Butler-Volmer equation:7

\[ j = j_0 \left( \exp \left( \frac{\alpha F \eta}{RT} \right) - \exp \left( \frac{1 - \alpha F \eta}{RT} \right) \right) \]  \hspace{1cm} [2]

Where:

\( R \) is the ideal gas constant;
\( F \) is the Faraday constant;
\( \alpha \) is the charge transfer coefficient, for oxygen reduction reaction,
\( n \) is the number of electrons transferred in the rate-determining-step of oxygen reduction reaction,
\( j_0 \) is the exchange current density.

The exchange current density \( j_0 \) can be expressed by the following formula:8

\[ j_0 = \rho_0 a_L \exp \left[ \frac{E_0}{RT} \left( 1 - \frac{T}{T_0} \right) \right] \]  \hspace{1cm} [3]

Where:

\( a_L \) is the area specific activity of the catalyst, the unit is cm²mg⁻¹;

Power-law: \( j = j_0 \left( \frac{E}{E_0} \right)^n \)  \hspace{1cm} [4]

The exchange current density \( j_0 \) is a constant, the exchange current density \( j_0 \) of the cathode and anode is different, the exchange current density \( j_0 \) of the anode is smaller than that of the cathode.

Figure 1. Schematic of rated voltage range for PEMFC.
\( \eta \) is the catalyst loading, unit mg/cm²; Then \( q_i \eta \) represents the roughness factor (Ri) of the electrode.

\( T^0 \) is the reference temperature,
\( P_i^0 \) is the reference pressure,
\( P_i \) is the cathodic gas pressure;
\( \gamma \) is the oxygen reduction reaction kinetic series,
\( j_0 \) represents the reference exchange current density,
\( E_c \) is the activation energy barrier for oxygen reduction.

From Equations 1, 2, 3, it can be derived that:

\[
R_{ct} = \frac{1}{j_0 \left( \frac{\alpha nF}{RT} \right) \cdot \frac{\eta}{RT}} = \frac{1 - (1 - \alpha) \eta}{\alpha nF \eta RT} \tag{4}
\]

**Charge-transfer resistance at near-open circuit voltage.** — When \( \eta \) is small enough, or rather, infinitely close to the open circuit voltage, the Butler-Volmer equation is expanded by the Taylor series:

\[
j = j_0 + \frac{\alpha n^2 F^2}{2RT^2} - \frac{(1 - \alpha)^2 n^2 F^2}{2RT^2} \tag{5}
\]

Ignoring second-order and higher-order infinitesimals, it can be obtained that:

\[
j = j_0 + \frac{\alpha n^2 F^2}{2RT^2} - \frac{(1 - \alpha)^2 n^2 F^2}{2RT^2} \tag{6}
\]

According to the formula 1 and 6, it can be obtained that:

\[
R_{ct} = \frac{\partial \eta}{\partial j} = \frac{RT}{nFj_0} \tag{7}
\]

**Charge-transfer resistance at high overpotentials.** — Generally, in the case of high overpotential, the reverse reaction rate of oxygen reduction reaction is relatively small, thus the reverse reaction rate can be ignored. Butler-Volmer equation is simplified to:

\[
j = j_0 e^{-\frac{\alpha nF \eta}{RT}} \tag{8}
\]

Tafel equation can be obtained whereby:

\[
\eta = \frac{2.303RT}{\alpha nF} \ln \left( \frac{j}{j_0} \right) \tag{9}
\]

Where \( \frac{2.303RT}{\alpha nF} \) is commonly referred to as the Tafel slope \( \beta \).

And the charge-transfer resistance can be expressed as:

\[
R_{ct} = \frac{\partial \eta}{\partial j} = \frac{RT}{\alpha nF} \frac{1}{j_0 e^{-\frac{\alpha nF \eta}{RT}}} \tag{10}
\]

It can derive from Formula 9 and 10 that:

\[
R_{ct} = \frac{b/2.303}{j} \tag{11}
\]

**Experimental**

All electrochemical tests in this experiment were performed on a homemade 25-cm² single fuel cell unit with gold coated stainless steel end plates, and the 890e fuel cell test system was provided by Shanghai Hephys Energy Corporation. All the potentials described in the paper are referenced to DHE (dynamic hydrogen electrode).

The catalyst coated membrane (CCM) used in this experiment was produced from Wuhan WUT New Energy Co., Ltd, and was embedded between two gas diffusion layers (GDLs) to constitute a required MEA. Commercial Pt/C catalysts were used on the cathode of MEA 1, while commercial Pt, Co/C catalysts (47 wt% of Pt, 5.4 wt% of Co) were used for MEA 2. The charge-transfer resistances (Rct) of MEA 1 were measured via EIS to verify the scientific rationality of the indicator for ORR activity. And ORR activities were compared between two MEAs at rated voltage with Rct and power density. Then the Rct was used to monitor the change of activity of MEA 3 with commercial Pt/C catalysts during durability test, in order to analyze the catalysts degradation. Note that, commercial Pt/C catalysts are used on anode of all MEAs, and Pt loading information for three MEAs are displayed in Table I.

Prior to electrochemical tests, the assembled cell was fully activated in the hydrogen-oxygen gas supply mode at high current densities. Until the performance no longer increased, the activation of the fuel cell was deemed to be complete.

In order to determine the test conditions for charge-transfer resistance, EIS under different oxygen partial pressures and different flow rates were compared. It was found that oxygen partial pressure and flow rates affected the total value of impedance, as shown in Fig. 2. The lower partial pressure and lower flow rate, the larger the total impedance. But impendence consists of the ohmic resistance (Rohn), Rct and mass-transport resistance (Rd). The variation of impendence was mainly contributed by Rd. Once we used the pure oxygen with high flow rates, Rd disappeared, and only Rct remained (Fig. 2c). Therefore, the pure oxygen with high flow rate was used to perform EIS in the following experiment.

**Results and Discussion**

**Indicator verification by ORR activity analysis of Pt/C catalysts.** — Fig. 3a shows the electrochemical impedance spectroscopy of MEA 1 at different current densities in the range of 40–1400 mA/cm², which is performed in the hydrogen-oxygen gas supply mode with high flow rate. The anode has a rapid rate of hydrogen oxidation reaction (HOR), and the charge-transfer resistance of the anode is generally negligible. It can be clearly seen that the diameter of the semicircle decreases with the increasing current density. That is, the cathodic charge-transfer resistance decreases with the increasing current density, which has been confirmed in a large number of literature. However, it is worth pointing out that when the current density increases to a certain value about 400 mA/cm², the charge-transfer resistance decrease at a more gradual rate, as shown in Fig. 3b. The values are obtained by EIS with a widely accepted equivalent circuit model. This is because the rate of increase of the activation overpotential is slowed down, which means the driving force of the electrochemical reaction increases at a relatively gentle rate.

The corresponding Tafel slopes can be calculated from charge-transfer resistance according to Equation 11, as shown in Fig. 4. The voltage value comes from the stable potential corresponding to the fixed current density before the disturbance signal is applied. From Fig. 4, it can be clearly seen that Tafel slopes calculated from \( R_{ct} \) are approximately 80 mV/decade at high potentials, belonging to the plateau area. Whereas the value increased with the decline of potentials below 0.8 V. Compared with the Tafel slope obtained from polarization curve in the literature, it is found that the calculated slope value is slightly larger (a common value is around 70mV/dec., which has a certain relationship with the test conditions and temperatures). And this difference is likely due to errors introduced by different test methods. It is worth emphasizing that the trend of Tafel slope with potential is basically the same. In a word, it shows that \( R_{ct} \) as the ORR indicator of catalysts has certain rationality preliminarily.

| Pt loading MEA 1 | MEA 2 | MEA 3 |
|-----------------|------|------|
| Pt/C | Pt, Co/C | Pt/C |
| Anode loading (mgP/cm²) | 0.4 | 0.4 | 0.1 |
| Cathode loading (mgP/cm²) | 0.2 | 0.2 | 0.4 |

**Table I. The composition of MEAs.**

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Figure 2. EIS of Pt/C catalysts @ 1000 mA/cm², operated under low (air) partial pressure of oxygen (a), high (oxygen) partial pressure of oxygen with variable (stoichiometric ratio 2.0 (anode)/2.0 (cathode), 440 mL/min (anode) and 220 mL/min (cathode)) flow rates of oxygen (b), and high (oxygen) partial pressure of oxygen with fixed (1000 mL/min (anode) and 2500 mL/min (cathode)) flow rates of oxygen (c), respectively, at a back pressure of 150 kPa for anode and cathode, 75°C, 100%/100% RH, frequency ranged from 10000 Hz – 0.1 Hz.

Figure 3. EIS (a) and Rct (b) of Pt/C catalysts at different current densities, operated at 75°C, 100%/100% RH, at a back pressure of 150 kPa H₂/O₂, fixed flow rates of 1000 mL/min (anode) and 2500 mL/min (cathode), respectively, frequency ranged from 10000 Hz – 0.1 Hz.

Activities comparison of Pt/C catalysts and PtₓC₀y/C catalysts by Rct—After the above experiment result and discussion, the indicator of charge-transfer resistance for ORR activity is verified. Now charge-transfer resistance is used to reevaluate the ORR activity of PtₓC₀y/C catalysts and compared with Pt/C catalysts. The EIS of MEA 1 and MEA 2 at 1400 - 40 mA/cm² (0.68–0.92V) with the disturbance amplitude of 10% cell output current, in the range of 10000Hz-0.1Hz, are both measured. Fig. 5 displays the EIS at 0.78 V (MEA 1@60mA/cm², MEA 2@80mA/cm²) and 0.90V (MEA 1@700mA/cm², MEA 2@800mA/cm²). The values of Rct derived from the diameter of the semicircle in EIS are compared. The diameter of the semicircle in EIS for MEA 1 shows no significant difference with that of MEA 2 at 0.78 V, while it is higher than that of MEA 2 at 0.9 V.

In order to compare ORR activities of these two catalysts more accurately, the normalized Rct was introduced. The Equations 3 and 4 reveal that, the Rct has something to do with the electrochemical area of catalysts, more accurately, Rct and ECSA are inversely related. Above all, CV (cyclic voltammetry) measurements of MEA 1 and MEA 2 have been conducted as shown in Fig. 6. The electrochemical surface area (ECSA) of the catalysts could be calculated according to the measured CV curves (hydrogen adsorption peak area). The value of ECSA for Pt/C and PtₓC₀y/C catalysts are 60.7 m²/g and 40.2 m²/g, respectively, shown in Fig. 6.

For better comparative analysis of ORR activity on MEA, besides ECSA, we also consider the Pt loadings and geometric area of the electrode. After these factors were normalized, Fig. 7a was obtained. It shows charge-transfer resistances of the Pt/C catalyst and the PtₓC₀y/C catalyst versus potential in range of 0.68 V to 0.92 V. In the high potential region, Rct of Pt/C catalysts is distinctly greater than that of PtₓC₀y/C catalysts, which demonstrates PtₓC₀y/C catalysts have...
Figure 5. EIS of Pt/C catalysts and Pt\textsubscript{x}Co\textsubscript{y}/C catalysts at 0.78 V (a) and 0.9 V (b).

Figure 6. CV curves of Pt/C catalysts (a) and Pt\textsubscript{x}Co\textsubscript{y}/C catalysts (b), operated at 30°C, 100%/100% RH, anode (H\textsubscript{2}) and cathode (N\textsubscript{2}) stoichiometry of 2.0/2.0, respectively.

Figure 7. R\textsubscript{ct} of Pt/C catalysts and Pt\textsubscript{x}Co\textsubscript{y}/C catalysts at different potentials (a) and the difference between R\textsubscript{ct} of Pt/C catalysts and Pt\textsubscript{x}Co\textsubscript{y}/C catalysts in the range of 0.7 V–0.9 V (b).
better catalytic performance. In the low potential region, when the PEM fuel cells work under rated conditions, $R_{ct}$ of Pt/C catalysts has no obvious difference with that of Pt$_{x}$Co$_{y}$/C catalysts. As shown in Fig. 7b, the difference between the two values of $R_{ct}$ for Pt/C and Pt$_{x}$Co$_{y}$/C catalysts is weakened from 37.77 Ω cm$^2$ to 0.72 Ω cm$^2$ gradually, as potential decreases from 0.9 V to 0.7 V. In view of the above results, Pt$_{x}$Co$_{y}$/C alloy catalysts do have certain advantages in ORR activity at high potentials. However, when the fuel cell is operated at the rated voltage (0.6 V–0.8 V), the advantages of Pt$_{x}$Co$_{y}$/C catalysts are weakened.

The polarization curves and power density curves of the MEA with Pt/C and MEA with Pt$_{x}$Co$_{y}$/C are also compared. As shown in Fig. 8, the power density of MEA with Pt/C is slightly lower than that of PtxCo$_{y}$/C. For example, the values of power density for MEA with Pt/C and MEA with Pt$_{x}$Co$_{y}$/C are 1.034 and 1.056 W/cm$^2$ at 1500 mA/cm$^2$, respectively, which display no obvious performance difference at rated voltage. It is interesting that the result of power density at rated voltage is consistent with that of $R_{ct}$, but against the common misconception, which is that PtxCo$_{y}$/C is much better than Pt/C. Because $R_{ct}$ is more accurate and easier than power density, $R_{ct}$ will become an effective tool to help the community to design a better catalyst in future.

The dynamics durability of Pt/C catalysts by $R_{ct}$.—When the life time is one of most critical factors for ORR catalysts, the dynamics degradation of catalysts in the PEMFC single cell is investigated in the long-term test. The single cell with MEA 3 is operated galvanostatically at a constant current density 600 mA/cm$^2$ and 75°C (0.722 V at the beginning of life (BOF), 0.627 V at the end of life (EOF), the voltage is always within the operating voltage range during the test). The $R_{ct}$ of Pt/C catalysts in the durability experiment is used as the activity indicator, combined with the loss of ECSA (Rf) of electrode, to research the catalysts degradation. EIS is tested every 100 hours during the durability experiment, altogether ten measurements data ranged from 0–900 hours are shown in Fig. 9.

Fig. 9a shows a time dependency of two semicircles, both charge-transfer resistance (in the high frequency region) and mass-transport resistance (in the low frequency region) increase with time, even mass-transport resistance appears more significant change, which should be ascribed to the degradation of mass transportation medium reported by other groups. Here we concentrated more on the former one, and $R_{ct}$ was obtained by EIS with a widely accepted equivalent circuit model in literature.

After the original data is fitted according to the equivalent circuit model, charge-transfer resistances at 600 mA/cm$^2$ in the period of 0–900 h are obtained, as shown in Fig. 9b. As can be clearly seen that, the charge-transfer resistance increased with time linearly, from 4.5 mOhm at the BOF to 10.11 mOhm at the EOF, it is increased by nearly 125.6% after 900 hours. It may be attributed to the growth of the particle size of Pt particles, the loss of platinum and other influence factor. M. Schulze et al. shows comparable results in their paper at a constant current density of 500 mA/cm$^2$. Therefore, the catalytic activity of the catalyst is speculated to reduce greatly during durability test.

According to the measured CV, the ECSA of the catalyst in the corresponding time period are obtained. As shown in Fig. 10, the overall electrochemical area of the catalyst is found to change over time and show a downward trend of about 26.4%. During the 0–900 h durability test, the increase of the charge-transfer resistance is not entirely caused by the reduction of the electrochemical active area, which accounts for approximately only 1/5. Other factors contributing to the increment of $R_{ct}$ remain further study.
1. The Rct of MEA with Pt/C catalysts was measured in the range of 0.68V~0.92V. Tafel slopes calculated from Rct were approximately 80 mV/decade at high potentials, whereas the value increased with the decline of potentials below 0.8 V. It showed that Rct as the ORR indicator of catalysts has certain rationality preliminarily.

2. Rct of MEA with Pt_xCo_0.5/C catalysts was lower than that of Pt/C and the same in the low potential region, indicating that the same ORR activities in rated potential region. The difference between the two values of Rct for Pt/C and Pt_xCo_0.5/C catalysts is weakened from 37.77 Ω·cm² (@0.9 V) to 0.72 Ω·cm² (@0.7 V) as potential decreased.

3. Rct of MEA with Pt/C catalysts during durability test was monitored. The Rct increased by nearly 125.6% after 900 hours. While the reduction of ECSA accounts for approximately only 1/5 of the increment of Rct. Other factors contributing to the increment of Rct remain further study.

Conclusions

In our work, charge-transfer resistance (Rct) was proposed to evaluate the ORR activity of catalysts for fuel cells at rated voltage.

1. The Rct of MEA with Pt/C catalysts was measured in the range of 0.68V~0.92V. Tafel slopes calculated from Rct were approximately 80 mV/decade at high potentials, whereas the value increased with the decline of potentials below 0.8 V. It showed that Rct as the ORR indicator of catalysts has certain rationality preliminarily.

2. Rct of MEA with Pt_xCo_0.5/C catalysts was lower than that of Pt/C and the same in the low potential region, indicating that the same ORR activities in rated potential region. The difference between the two values of Rct for Pt/C and Pt_xCo_0.5/C catalysts is weakened from 37.77 Ω·cm² (@0.9 V) to 0.72 Ω·cm² (@0.7 V) as potential decreased.

3. Rct of MEA with Pt/C catalysts during durability test was monitored. The Rct increased by nearly 125.6% after 900 hours. While the reduction of ECSA accounts for approximately only 1/5 of the increment of Rct. Other factors contributing to the increment of Rct remain further study.

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