

Overpotential analysis with various anode gas compositions in a molten carbonate fuel cell

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Abstract

The effect of anode gas composition on the overpotential in a 100 cm² class molten carbonate fuel cell is investigated. A total of five different gas compositions are used. They are applied to cross-check the effect of flow rate and composition, e.g., a given composition with different gas flow rates and a total flow rate with different gas compositions. The overpotential at the anode is analyzed via steady-state polarization, inert gas step addition and reactant gas addition methods. The analyses reveal that the anodic overpotential depends on the flow rate of the reactant species rather than the composition. Two identical gas compositions show less overpotential when a larger flow rate is applied. Compositions with large flow rates of CO₂ and H₂O also yield less overpotential due to the gas species. Overpotential analyses show that the three measurements have complementary relationships.

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1. Introduction

The molten carbonate fuel cell (MCFC) is an electrochemical energy-conversion system, and as such, the resistance in the electrochemical reactions determines the performance. In general, the performance is limited by the electrical resistance of the constituent materials, e.g., electrodes, electrolyte, bipolar plates, and the resistance of the electrochemical reactions on the electrode surfaces. The electrochemical reaction is controlled by the charge-transfer process in a slow reaction system and by the mass-transfer process in a fast reaction system. Thus far, it has been revealed that the electrode reactions in molten carbonates are mainly mass-transfer control processes, even in the case of the oxygen reduction reaction, which is much slower than the hydrogen oxidation reaction [1–3]. These reaction characteristics derive from the use of molten carbonate as an electrolyte.

The eutectic carbonates melt at temperatures beyond 773 K [4], and thus the electrochemical reactions in this temperature range are highly activated. The reaction rate at the electrode thereby becomes very high, and the electrode reaction occurs in the mass-transfer controlling region.

The electrodes of the MCFC are covered with thin electrolytes comprised of porous materials, and the gas-phase reactants are supplied to the MCFC. Thus, the mass-transfer process is comprised of two processes: mass-transfer of active species in the liquid electrolyte film and gas-phase mass-transfer in the gas channels. It was previously reported [1] that the electrode reactions in the MCFC are significantly affected by the gas-phase mass-transfer process at the anode and cathode. In addition, they suggested that the anode reaction is mainly a gas-phase mass-transfer control process, because of the very thin electrolyte film on the electrode surface. In contrast, the cathode involves mixed mass-transfer control processes in the gas- and liquid-phases due to the relatively thick electrolyte film.

According to Ang and Sammells [5], the reaction rate of hydrogen oxidation in molten carbonate has a positive relation

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with the partial pressure of H₂, CO₂, and H₂O, i.e.,

$$i_o \propto p(\text{H}_2)^{0.25} p(\text{CO}_2)^{0.25} p(\text{H}_2\text{O})^{0.25} \quad (1)$$

Therefore, the anode requires three gases for H₂ oxidation. At present, the source of hydrogen for the MCFC is typically natural gas reforming, and thus the composition of the feed gas is taken to be that of the reformed gas, and is mainly: H₂:CO₂:H₂O=69:17:14 mol%. A previous study [1] pointed out that the overpotential at the anode is inversely proportional to the mass-transfer coefficient in the gas-phase (Eq. (2)) and the low flow rate of CO₂ and H₂O gives rise to a considerable overpotential at the anode.

$$\eta_{\text{an}} = \sum_A \eta_{\text{an,A}} \cong \sum_A i \frac{RT}{n^2 F^2 a c_{0,A}} \left(\frac{1}{k_{G,A}} \right) \quad (2)$$

where $k_{G,A} (\equiv 0.664(v_{f,A}/L)^{1/2} (D_{G,A})^{2/3} (v_A)^{-1/6})$ is the gas-phase mass-transfer coefficient of the A species, the subscript A denotes the reactant species; i is the current; a is the geometrical electrode surface; c_0 is the bulk concentration; v_f is the linear velocity of the gas feed; L is the electrode length; D_G is the gas diffusivity; ν is the kinematic viscosity; the remaining symbols have their usual meanings.

It was also reported that an equal gas composition (H₂:CO₂:H₂O = 33:33:33 mol%) delivered a much lower anodic overpotential, because the overpotential due to the CO₂ and H₂O species decreases as a result of the increased partial pressures and flow rates [6]. The results, however, were obtained with only two different gas compositions, and thus they cannot fully clarify the effects of flow rate and partial pressure on the overpotential. In this work, a total of five different gas compositions are employed to investigate the dominant factors for the overpotential determination in the MCFC.

2. Experimental

A 100 cm² class MCFC single cell was used. The anode was made of Ni–Al alloy metal and the cathode was in situ oxidized NiO. The matrix was alumina fibre reinforced LiAlO₂, and the electrolyte was an eutectic of 62 mol% Li₂CO₃ and 38 mol% K₂CO₃. The gas channels were set between the electrode and cell frame at the anode and cathode, respectively. The cell frame and gas channel was made of STS 316L stainless steel. More details of the cell components and cell preparation can be found in a previous study [1].

Five different inlet compositions were used for the anode gas (Table 1). Composition 1 (C1) is a simulated gas composition

Table 1
Gas flow rates and partial pressures of five gas compositions at anode inlet

	H ₂ :CO ₂ :H ₂ O flow rate/L min ⁻¹ (partial pressure/atm)
C1	0.174:0.043:0.035 (0.69:0.17:0.14)
C2	0.174:0.058:0.058 (0.60:0.20:0.20)
C3	0.174:0.522:0.174 (0.20:0.60:0.20)
C4	0.522:0.174:0.174 (0.60:0.20:0.20)
C5	0.174:0.174:0.522 (0.20:0.20:0.60)

based on natural gas reforming. Composition 2 (C2) is a simplified composition of C1 at a fixed H₂ flow rate. Compositions 3 (C3), 4 (C4), and 5 (C5) have the same total flow rate but the component gas compositions are different. C3 has a three fold larger CO₂ concentration compared with H₂ and H₂O. Thus, C3 is a CO₂-rich composition. By comparison, C4 and C5 are H₂- and H₂O-rich compositions, respectively. Thus, the effects of composition on the anodic overpotential can be evaluated with C3, C4, and C5. On the other hand, C2 and C4 have the same gas composition but the flow rate of C4 is three times larger than that of C2. Thus, the effect of flow rate on the overpotential can be observed with these compositions. The flow rates of H₂ and CO₂ were controlled by mass flow controllers (MFCs). The water content in the gases from C1 to C5 was controlled with a temperature-controlled bubbler.

The cathode gas was a mixture of 70 mol% air and 30 mol% CO₂ and its total flow rate was 0.883 L min⁻¹. The flow rate of air and CO₂ was controlled by a mass flow controller (MFC). The operation pressure corresponded with atmospheric conditions and the temperature was 923 K.

Overpotential behaviour was measured by means of steady-state polarization (SSP), inert gas step addition (ISA), and reactant gas addition (RA) methods. SSP measures output voltage with increasing current. Thus, the difference between open-circuit voltage (E_{OCV}) and voltage in a polarization state gives the total overvoltage of the cell. ISA induces a spontaneous increase of a reactant's flow rate by the addition of inert gas to the electrode, which results in a voltage change in a polarization state. The subsequent variation in overpotential is then analyzed with respect to the flow rate and partial pressure [1]. ISA measurement provides quantitative information on the overpotential due to the gas-phase mass-transfer resistance at the electrode. In this work, nitrogen was used and the length from the inlet port to the cell was about 4.0 m, with a metal tube of 1/4 in. outer diameter. In the RA method, a reactant gas is added to an electrode that results in a flow rate and concentration change of the species at the electrode [2]. This leads to a shift in overpotential due to the flow rate and partial pressure changes of the species. Thus, RA yields information on the overpotential due to the reactant species.

3. Results and discussion

3.1. Steady-state polarization

The results of SSP under the five gas conditions given in Table 1 are presented in Fig. 1. E_{OCV} can be expressed by the gas partial pressure according to the Nernst equation, namely:

$$E_{\text{ocv}} = E^0 + \frac{RT}{2F} \ln \left(\frac{[\text{H}_2]}{[\text{H}_2\text{O}][\text{CO}_2]_{\text{an}}} [\text{O}_2]^{0.5} [\text{CO}_2]_{\text{ca}} \right) \quad (3)$$

where E^0 is the standard potential, and other symbols have their usual meanings. The gas condition of C1 shows the highest E_{OCV} , because it has the largest H₂ partial pressure among the five gas compositions. By increasing the CO₂ and H₂O partial pressures, a decrease of E_{OCV} is observed. A more correct esti-

Table 2
Gas flow rates inside cell, calculated and measured E_{OCV} , and total overpotential (η_{tot})

	H ₂ /(atm) (L min ⁻¹)	CO ₂ /(atm) (L min ⁻¹)	H ₂ O/(atm) (L min ⁻¹)	CO/(atm) (L min ⁻¹)	Calculated E/V	Measured E/V	η_{tot} (mV)
C1	0.591 (0.149)	0.075 (0.019)	0.237 (0.060)	0.097 (0.025)	1.073	1.058	274
C2	0.505 (0.146)	0.105 (0.030)	0.295 (0.086)	0.095 (0.028)	1.044	1.039	267
C3	0.105 (0.091)	0.505 (0.439)	0.295 (0.257)	0.095 (0.083)	0.920	0.920	219
C4	0.505 (0.439)	0.105 (0.091)	0.295 (0.257)	0.095 (0.083)	1.044	1.037	203
C5	0.174 (0.152)	0.174 (0.152)	0.626 (0.544)	0.026 (0.022)	0.952	0.950	227

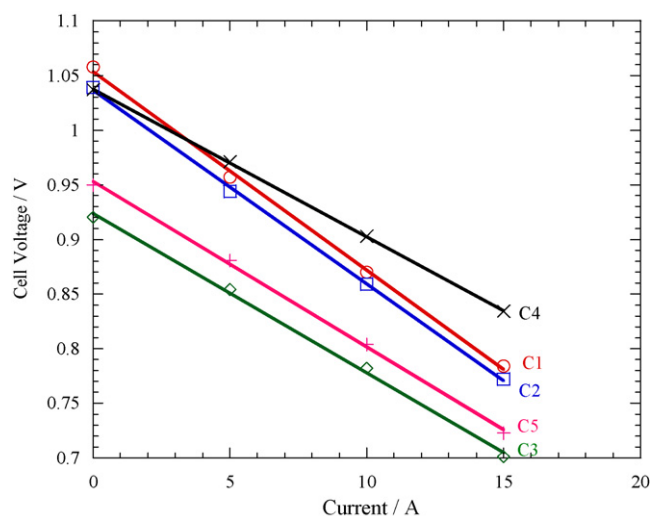


Fig. 1. Steady-state polarization for five gas compositions listed in Table 1 at $u_{ox} = 0.4$ (at 15 A), 923 K, 1 atm.

mation of E_{OCV} requires compensation of the gas composition by the water gas shift reaction (Eq. (4)), which is the equilibrium reaction among the H₂, CO₂, and H₂O gases.



The equilibrium constant of reaction (4) at 923 K is about 0.52 [4], and thus four gases, H₂, CO₂, H₂O, and CO, flow in the anode. Table 2 compares the gas flow rates and partial pressures at open-circuit inside the cell, the calculated and measured E_{OCV} , and the total overvoltage (η_{tot}). The value of η_{tot} was obtained as the difference between E_{OCV} and the voltage at a current of 15 A. The measured E_{OCV} of C1 shows a slight deviation from the calculated value, but other gas compositions demonstrate a very close agreement between the measured and calculated values. The C1 composition gives the largest total overvoltage, although it has the highest E_{OCV} . The difference in E_{OCV} between the C2 and C1 compositions is very small, because these compositions are very similar, and C3 gives the lowest E_{OCV} due to a high CO₂ flow rate. Although C2 and C4 have the same gas composition, different η_{tot} values are obtained. In addition, there are no significant discrepancies in the total overvoltage among C3, C4, and C5, while they show differences in composition. The data in Table 2 indicate that η_{tot} is not solely a function of gas composition.

The current–voltage behaviour of a MCFC is described by a linear line whereas that of a low-temperature fuel cell shows an exponential decrease in voltage with current. This is

closely related to the fast charge-transfer processes in the high-temperature molten carbonates. Thus, the voltage of the MCFC in a polarization state is a function of the mass-transfer rate, and the following relation holds.

$$V = E_{OCV} - \eta_{tot} \quad (5)$$

where $\eta_{tot} = \eta_{IR} + \eta_{an} + \eta_{ca}$, η_{IR} is the ohmic loss, and η_{an} and η_{ca} are the overpotentials due to the respective electrochemical reactions at the electrodes. In addition, the overpotential at the electrodes is determined in terms of the charge-transfer rate (k_0), the mass-transfer rate in the liquid-phase (k_L), and the mass-transfer rate in the gas-phase (k_G) [1]. The anode has a relatively very thin electrolyte film on the electrode surface due to the high contact angle between the carbonate electrolyte and the electrode material [7]. Accordingly, the charge-transfer and liquid-phase mass-transfer rates will be very fast at the anode, and the gas-phase mass-transfer rate controls the reaction rate. Therefore, the gas-phase transport effect would be dominant in the overpotential, and the anodic overpotential is expressed by Eq. (2). The equation indicates that the anodic overpotential is a function of the gas concentration and the gas flow rate at a fixed temperature. Since the cathodic condition of the cell was maintained at a constant gas condition and the internal resistance is generally independent of the gas composition, it can be concluded that the various slopes of the gas composition result from the anode gas composition and the gas flow rate.

3.2. Inert gas step addition (ISA) measurement

Fig. 2 shows the characteristic voltage shift of the ISA by the addition of 0.3 L min⁻¹ to the C2 composition at various current densities. The E_{OCV} slightly increases with N₂ addition because the partial pressures of the reactant gases are reduced and, according to Eq. (2), this enhances E_{OCV} . With increasing current, the generation of two positive and negative voltage peaks is observed. These voltage peaks are due to an overpotential shift resulting from N₂ addition, which is related to the flow rate change of the reactants by the addition [1]. The flat peaks, observed at a current density of 150 mA cm⁻², were also observed in previous work [8], and indicate that the voltage shifts produced by the N₂ addition are fully developed and the distance from the inert gas inlet port to the cell is sufficient to measure the overpotential shift of the ISA measurement. Given that the added nitrogen does not arrive at the cell during the positive voltage peak, it can be concluded that the peak height (ΔV_I) is solely the result of the reactant flow rate change [1].

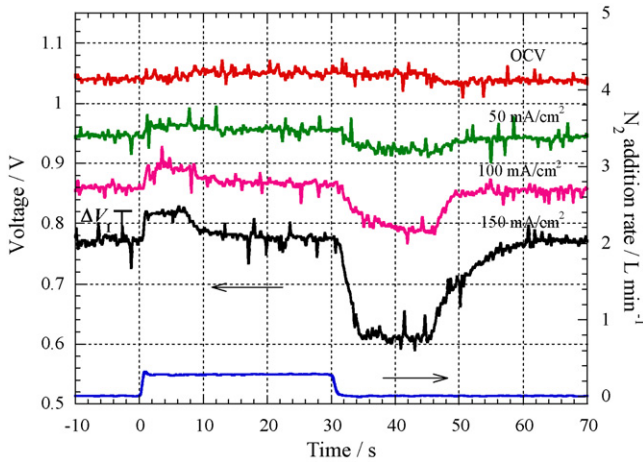


Fig. 2. ISA results with addition of $0.3 \text{ L min}^{-1} \text{ N}_2$ to various current densities at anode at $u_{\text{ox}} = 0.4$ (at 15 A), 923 K, 1 atm.

The voltage peak (ΔV_I) originates from the overpotential reduction due to the increase in the flow rate, because the overpotential is a function of the reactant's flow rate, as shown in Eq. (2). Thus, an increase in the N_2 addition rate results in enhancement of the flow rate and peak height (ΔV_I). On the other hand, the overpotential at the anode is a function of each reactant species' flow rate, and thus a low flow rate results in large overpotential. Our previous work [2] showed that the overpotential due to the CO_2 and H_2O species is larger than that of H_2 in the C1 composition, because of the lower flow rate of CO_2 and H_2O relative to H_2 . Therefore, the largest overpotential is obtained in the C1 composition, having the lowest flow rates of CO_2 and H_2O , as shown in Fig. 3. Consequently, among the five gas compositions, the highest ΔV_I is measured with the C1 composition.

The voltage peak of ΔV_I represents the overpotential difference before and after the increase in flow rate. Since the flow rate of a fixed current can be expressed by utilization, ΔV_I can

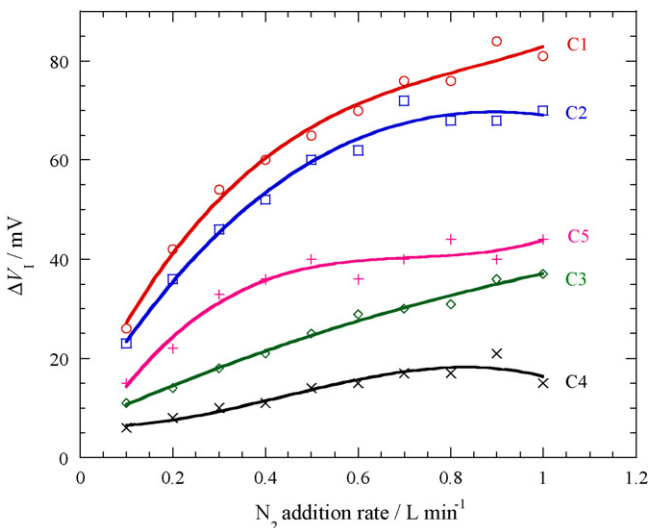


Fig. 3. Plots of ΔV_I vs. N_2 addition rate to anode at $u_{\text{ox}} = 0.4$ (at 15 A), 923 K, 1 atm.

be represented by utilization, as given in Eq. (6) [1].

$$\Delta V_I = \eta_1 - \eta_2 = qu_1^{0.5} - qu_2^{0.5} = m - qu_2^{0.5} \quad (6)$$

where η is the overpotential, the subscripts 1 and 2 indicate before and after addition, respectively

$$q = \frac{1.51 RT (iLs)^{1/2} v^{1/6}}{(n^3 F^3 a^2 c_0)^{1/2} D_G^{2/3}}$$

is a constant, $m = qu_1^{0.5}$ is a constant, and s is the cross-sectional area of the gas channel; other symbols have their usual meanings.

Utilization is the ratio of consumed gas to the gas amounts fed to the cell at a fixed current. This work employs five different anode gas compositions. The anode utilization is generally defined by the hydrogen utilization. Although CO_2 and H_2O are reactant species according to the hydrogen oxidation mechanism [5], the utilization before N_2 addition (u_1) is determined by the H_2 utilization. The reactant's flow rate is increased by N_2 addition during the positive voltage peak, and thus utilization is reduced by the flow rate increase. The reduced utilization (u_2) is expressed by the following equation:

$$u_1 = \frac{V_C}{V_F}, \quad u_2 = \frac{V_C}{(V_F + V_I)} \quad (7)$$

where $V_C = u_1 V_F$ is the consumed gas amount, V_F is the fed gas amount, and V_I is the added N_2 amount.

Fig. 4 shows the relation of Eq. (6) with five gas compositions. The gas compositions show a satisfactory relation with Eq. (6), regardless of the composition change, as shown in Fig. 4. The slope of the relation, q , is the overpotential due to the gas-phase mass-transfer at 100% gas utilization [1]. Therefore, the q values in Fig. 4 represent the overpotential due to the gas-phase mass-transfer of the gas compositions. The q values indicate that the anode has considerable overpotential for the given gas compositions. This is contradictory to the findings of previous investigations [9–11], and indicates that the anode has a very small overpotential, which is a function of the H_2 partial pressure. The q values of the given gas compositions, are listed in Table 3.

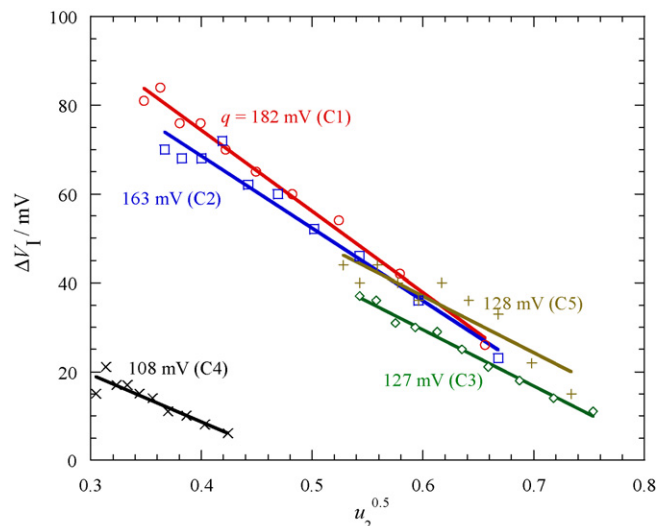


Fig. 4. Relationships of Eq. (6) with data obtained from Fig. 3.

Table 3
The q values of Eq. (6) obtained in Fig. 4

	q values (mV)
C1	182
C2	163
C3	127
C4	108
C5	128

The q value of C1 is 182 mV, which is considerably larger than those (155 mV and 159 mV) reported in a previous study [8]. This difference is attributed to the various parameters for mass-transfer in the cell, e.g., pore distribution, electrolyte film thickness, electrolyte amount, corrosion. Thus, the cell in this work has a larger mass-transfer resistance in the gas-phase than the cells used previously. The q value of C2 is slightly less than that of C1, which indicates that the overpotential of C2 is smaller than that of C1. The difference is ascribed to the enlarged CO₂ and H₂O flow rates of C2 compared with C1. This is in line with a report that a large anodic overpotential was obtained at low CO₂ and H₂O flow rates [2]. The larger flow rates of CO₂ and H₂O in C3, C4, and C5 yield very low q values. C2 and C4 have different flow rates while they have the same gas composition. Consequently, they only vary with respect to utilization. Eq. (6) states that the anodic overpotential is a function of utilization for the same constant q . Therefore, the q values of C2 and C4 should be the same. The reason for the low q value of C4 (see Table 3) is not clear, but it is evident that the low values of ΔV_I interfere with the correct measurement of the q value.

3.3. Reactant gas addition (RA) measurement

Fig. 5 shows two RA results obtained with the addition of 0.3 L min⁻¹ H₂ to the anode for the C2 and C4 compositions. As discussed above, RA is also an overpotential measurement method that involves the addition of a reactant gas. The difference in E_{OCV} change (ΔE) and voltage change in a polarization state (ΔV_p) as a result of the addition of a reactant gas represents the overpotential shift produced by the addition, as given by

$$\Delta V_A = \Delta V_{p,A} - \Delta E_A \quad (8)$$

where A is the reaction species. A positive value of ΔV_A implies that the addition of reactant gas reduces the overpotential. By contrast, a negative value indicates an increase in overpotential. Fig. 5 shows a voltage increase in the open-circuit state (ΔE_{H_2}) and a voltage increase at 15 A ($\Delta V_{p,H_2}$) on H₂ addition. The same amount of H₂ addition results in larger ΔE_{H_2} and $\Delta V_{p,H_2}$ in the C2 than in the C4 composition, due to the smaller reactant flow rate of the former.

The difference in ΔE_{H_2} and $\Delta V_{p,H_2}$, ΔV_{H_2} , is plotted as a function of H₂ addition rate in Fig. 6. The positive values of ΔV_{H_2} show that the H₂ species causes mass-transfer resistance in the gas-phase and its addition reduces the resistance. The values of ΔV_{H_2} at C4 are consistent for the addition range, although there is some negligible deviation, which shows that H₂ addition successfully reduces the gas-phase resistance. On the other hand,

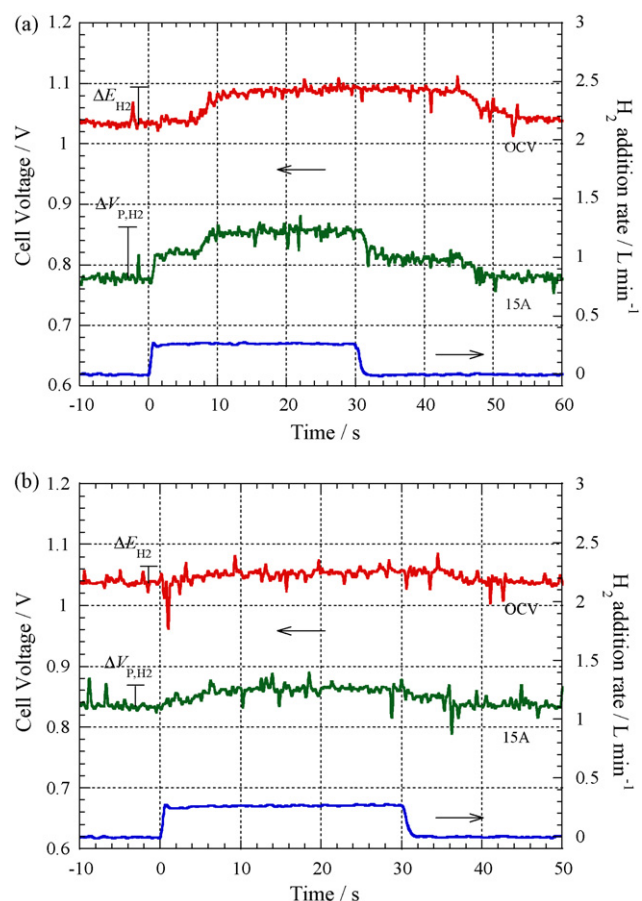


Fig. 5. RA measurements by addition of 0.3 L min⁻¹ H₂ to anode at (a) C2 (H₂:CO₂:H₂O=0.174:0.058:0.058 L min⁻¹) and (b) C4 (H₂:CO₂:H₂O=0.522:0.174:0.174 L min⁻¹) compositions.

ΔV_{H_2} in C2 increases with addition, i.e., a low H₂ flow rate has a relatively large mass-transfer resistance, and thus ΔV_{H_2} shows a steady value over a certain range of additions. The larger flow rate of C4 results in a lower ΔV_{H_2} than for C2 over the addi-

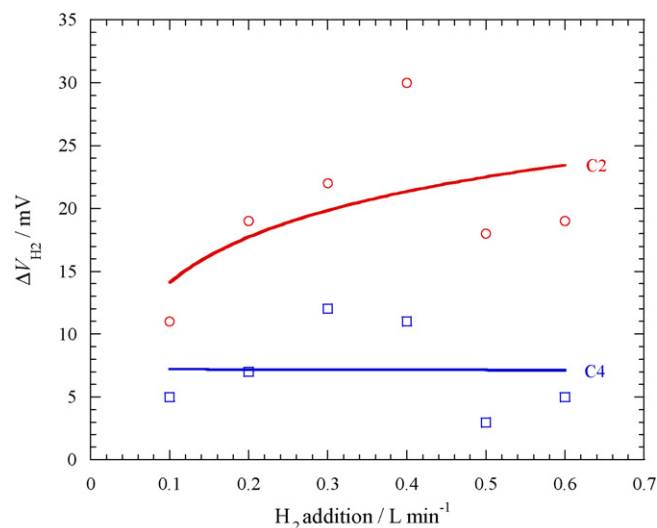


Fig. 6. ΔV_{H_2} vs. H₂ addition rate at C2 and C4 gas compositions.

tion range, which indicates that C4 has a smaller mass-transfer resistance due to the H₂ species.

Fig. 7 shows the RA results for the addition of 0.3 L min⁻¹ CO₂ to the C2 and C3 compositions. According to Eq. (3), the addition of CO₂ results in a negative shift of E_{OCV} , so that negative values of ΔE_{CO_2} and $\Delta V_{p,CO_2}$ are observed. Since the reactant flow rate of C2 is smaller than that of C3, larger ΔE_{CO_2} and $\Delta V_{p,CO_2}$ values are obtained for the C2 composition. This indicates that low values of ΔE_{CO_2} and $\Delta V_{p,CO_2}$ in C3 will yield a smaller overpotential due to the CO₂ species than in C2.

Fig. 8 shows the overpotential reduction caused by the addition of CO₂ to the C1, C2, C3, and C4 compositions. The CO₂ flow rate in the cell is shifted from the inlet gas composition, as shown in Table 2. The minimal CO₂ flow rate of the C1 composition results in the largest ΔV_{CO_2} , whereas C3 gives the lowest ΔV_{CO_2} value. This indicates that C1 has the largest overpotential among the compositions due to the mass-transfer resistance of the CO₂ species. By increasing the CO₂ flow rate, a decrease in ΔV_{CO_2} is observed among all compositions. For four compositions, ΔV_{CO_2} appears to be independent of the CO₂ addition rate above 0.2 L min⁻¹. This implies that the resistance due to the CO₂ species is readily recovered over the addition rate. While the C2 and C4 compositions have the same composition, the flow rate of C4 is three times that of C2. As such, the comparison pro-

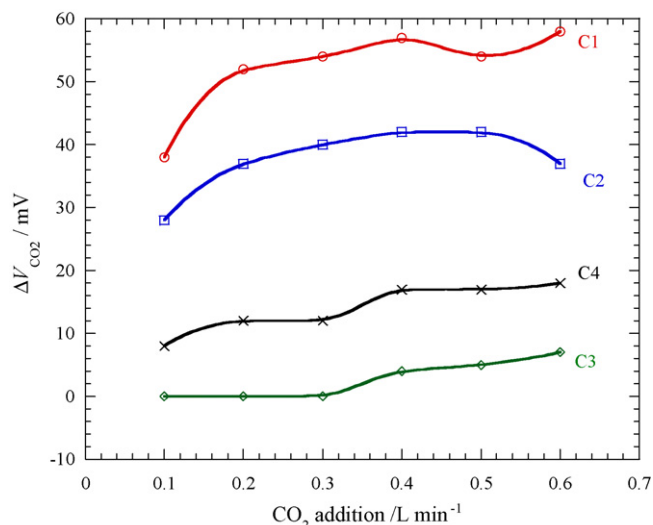


Fig. 8. ΔV_{CO_2} vs. CO₂ addition rate at C1, C2, C3, and C4 compositions.

vides information on the overpotential at the anode in terms of the flow rate and the composition. Fig. 8 shows that ΔV_{CO_2} of C2 is much larger than that of C4, thus clearly showing that the overpotential strongly depends on the flow rate rather than on the gas composition.

4. Conclusions

Five different gas compositions with varying flow rates and composition have been employed to investigate their effects on the overpotential at the anode of a MCFC. Based on overpotential analyses with SSP, ISA, and RA measurements of the five different gas compositions, the following conclusions have been drawn.

- (i) Among the tested gas compositions, the C1 composition causes the largest overpotential due to the low CO₂ and H₂O flow rates.
- (ii) A lower CO₂ or H₂O composition yields a higher E_{OCV} ; but low flow rates result in a large overpotential at the anode.
- (iii) Although the C3 and C5 compositions show very low values of E_{OCV} , a higher gas flow rate results in a lower anodic overpotential.
- (iv) The C4 composition gives a much lower overpotential than C2. C4 has three fold higher flow rates than C2, despite the fact that they share the same gas composition. Thus, the results indicate that the overpotential at the anode strongly depends on the reactant's flow rate rather than on the composition.
- (v) RA measurements reveal that the overpotential at the anode is affected by the flow rate of the reactant species. This implies that the overpotential is the sum of each overpotential due to the H₂, CO₂, and H₂O species.

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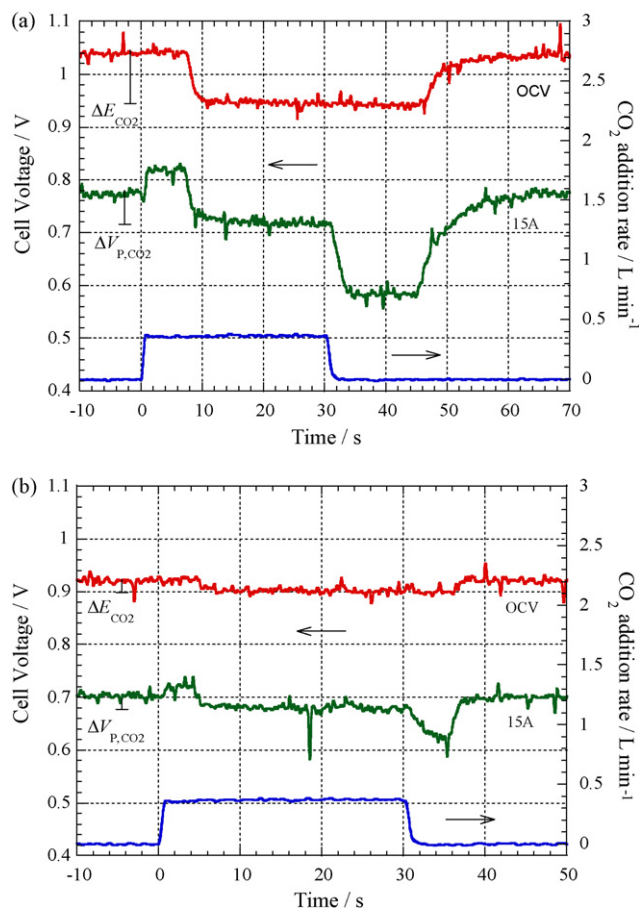


Fig. 7. RA measurements by addition of 0.3 L min⁻¹ CO₂ to anode at (a) C2 (H₂:CO₂:H₂O=0.174:0.058:0.058 L min⁻¹) and (b) C3 (H₂:CO₂:H₂O=0.174:0.522:0.174 L min⁻¹) compositions.

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