

Fabrication and Operation Characteristics of Electrolyte Impregnated Matrix and Cathode for Molten Carbonate Fuel Cells

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In this work, an electrolyte impregnated cathode and matrix were fabricated using (Li/Na)₂CO₃ powders for use in a molten carbonate fuel cell (MCFC). 87% of cathode pores and 90% of matrix pores were filled with electrolyte. A 25 cm² single cell was used with electrolyte impregnated components. Cell performance of the single cell with electrolyte impregnated components showed a similar performance to a conventional single cell. After cell operation, electrolyte was found to have moved to the anode, cathode and matrix. The remaining electrolyte in matrix pores, cathode pores and anode pores are 94.62%, 42.75%, was 21.56%, respectively. By using electrolyte impregnated components, the change of the wet-seal height was decreased 86.78% (to 0.23 mm) comparing with the conventional cell. Electrolyte impregnated components will provide simplified pretreatment process and remove problems in the pretreatment condition such as non-uniform electrolyte melting.

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1. Introduction

Molten carbonate fuel cells (MCFCs) are high-temperature fuel cells that use liquid electrolytes composed of molten carbonates such as Li₂CO₃, Na₂CO₃, and K₂CO₃.¹ A ceramic matrix composed of LiAlO₂ was used to contain the liquid between the anode and the cathode. Carbonate ions are conducted from the cathode to the anode. In the anode side, H₂ reacts with carbonate ions (CO₃²⁻) to produce H₂O, CO₂ and ions. In the cathode side, O₂ reacts with CO₂ and ions and produces CO₃²⁻.

A schematic figure of the conventional assembly of MCFCs is shown in Fig. 1(a). The anode and the cathode for MCFCs are fabricated via the tape casting method.² After tape casting, the green sheet is sintered at 800°C for the cathode and 1100°C for the anode. The matrix and the electrolyte are also fabricated via the tape-casting process. The matrix and the electrolyte are used in the green sheet form. Electrolyte green sheets are placed between matrix green sheets.

During cell heat-up, binders and other compounds in the green sheets are removed and the electrolyte in the green sheets melt.³ Liquid electrolyte is then immersed into the anode, cathode and matrix. There are subsequent changes in the thickness of the stack due to the melting of electrolyte.

In the operation of a single cell of MCFCs, the thickness change in the single cell due to electrolyte green sheets does not influence the operation of the single cell. However, in the stack of MCFCs, which is composed of hundreds of cells, the thickness change due to the electrolyte green sheet induces thermal stress and the stress concentration due to non-uniform melting of electrolyte.⁴ These issues constitute significant problems in the use of MCFCs such as the fracture of the matrix and the gas crossover between the anode and the cathode. In addition, long pretreatment time is required to burn out organics and binders in the matrix and electrolyte green sheets.³

To avoid non-uniform melting of electrolytes and simplify the pretreatment process, an electrolyte impregnated components were

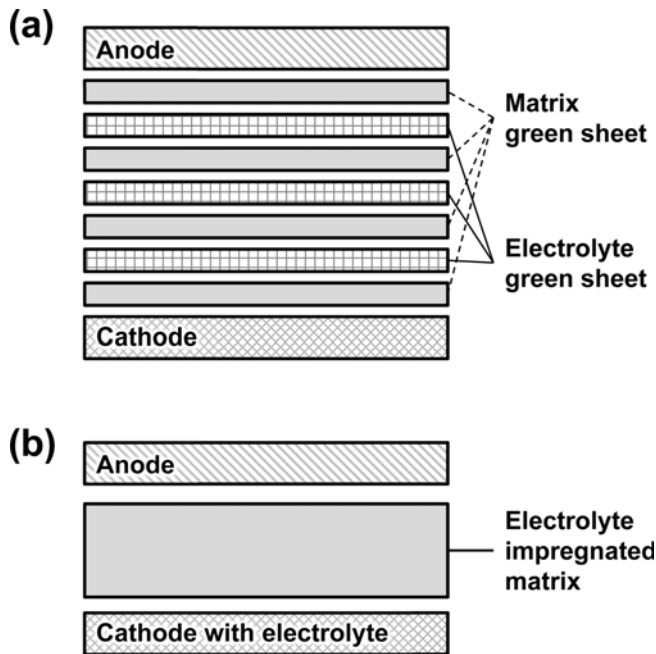


Fig. 1 Comparison between (a) conventional MCFC single cell and (b) MCFC single cell with the electrolyte impregnated matrix and cathode

proposed. The schematic figure of the single cell of MCFCs with electrolyte impregnated matrix and cathode is presented in Fig. 1(b). In this type of MCFCs, additional electrolyte green sheets are not necessary.

By employing the electrolyte impregnated matrix, the structure of MCFCs was simplified as shown in Fig. 1(b). In MCFCs with electrolyte impregnated components, tape-casted green sheets were not used. Because binders and organics found in the green sheets were not included in the cell components, the pretreatment process could be simplified and shortened. Also, the thickness change due to electrolyte melting did not occur.

In this work, the electrolyte impregnated components for MCFCs were fabricated using $(\text{Li}/\text{Na})_2\text{CO}_3$ powders. Fabricated electrolyte impregnated components were investigated and employed in the 25 cm^2 single cell operations. Operation characteristics of the 25 cm^2 single cells with electrolyte impregnated components were studied. The thickness changes of the conventional single cell and the single cell with electrolyte impregnated components were then compared.

2. Experimental Method

2.1 Fabrication of the Electrolyte Impregnated Cathode and Matrix

In MCFCs, a Ni-Ni5wt%Al (60 : 40) anode, a lithiated NiO cathode, a LiAlO_2 matrix and $(\text{Li}/\text{Na})_2\text{CO}_3$ electrolytes⁵ in a eutectic composition were employed for components of MCFCs. These components were fabricated via the tape-casting process. Slurry with a viscosity of 5,000-10,000 cP was used in the tape-casting process. A binder (Butvar B76), a plasticizer (Sigma Aldrich, Dibutyl phthalate), a defoamer (San Nopco, SN-D348) and a dispersant (BYK, BYK-110) were employed in the fabrication of the slurry. A green sheet with the

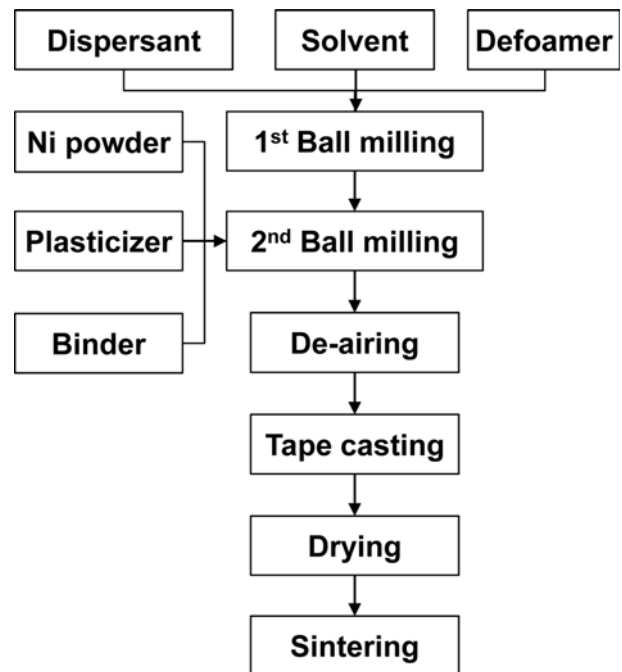


Fig. 2 Manufacturing process of the cathode

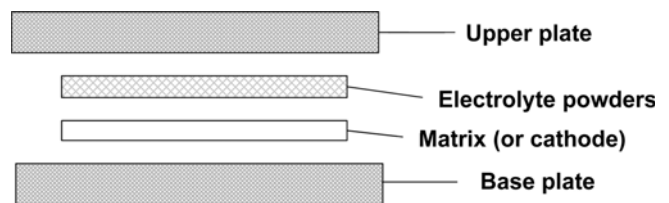


Fig. 3 Schematic figure of the electrolyte impregnation process

desired thickness was fabricated with a doctor blade.²

The tape casting process enabled the mass-production of green sheets for the anodes, cathodes and matrices. Fig. 2 presents the manufacturing process of the cathode. But, in order to make slurry, dispersant was added to make the slurry disperse evenly. Binder and plasticizer ensured the proper viscosity. In the conventional cell, the anode and the cathode were sintered at 1100°C and 800°C , respectively, under the reduced atmosphere. In the sintering process, binders and organic compounds in the green sheets were removed.

In the case of the matrix and the electrolytes, tape-casted green sheets were employed in the cell directly. In the pre-treatment process, binders and organics in the green sheets were removed. Electrolyte green sheets were melted, and electrolyte was immersed to the matrix. Before the impregnation process, the matrix was burnt at 650°C in the air atmosphere to remove binder and organics.

The schematic figure of the electrolyte impregnation process is presented in Fig. 3. For the upper plate and the base plate, alumina plates were used. In the impregnation process, electrolyte powders and green sheets were acceptable. In this work, electrolyte powders were employed in the impregnation process for the simplicity of the impregnation process. Electrolyte powders were placed directly on the components.

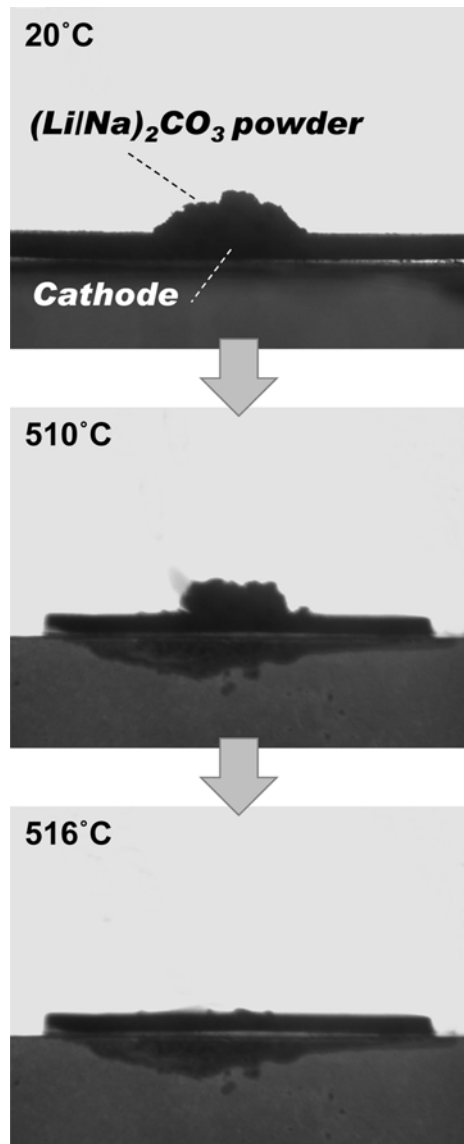


Fig. 4 Electrolyte impregnation process of the cathode

The amount of the electrolyte impregnated into components was determined from the objective filling ratio (normal operating condition)⁶ of MCFCs. Typically for MCFCs, Electrolyte is impregnated into 20% of anode pores, 40% of cathode pores and 100% of matrix pores. The average porosity of the anode, the cathode and the matrix was 0.5, 0.8 and 0.55, respectively. From the size of the components and the average porosity, the required weight of electrolytes was calculated.

When the electrolyte powders were applied to the matrix to fill the pores fully, liquid electrolytes spilled over matrix. Consequently, some parts of the matrix were stuck to the base plate. In order to avoid this problem, it was determined that the filling ratio of the matrix and the cathode should be less than 100%.

The electrolyte impregnation process was conducted in a heat chamber. The chamber was heated up to 650°C which was the operating temperature of MCFCs and kept for one hour. Gas atmospheres were reduced atmosphere with compositions of H₂:N₂ of 30:70. In the electrolyte impregnation process, the sintered anode and

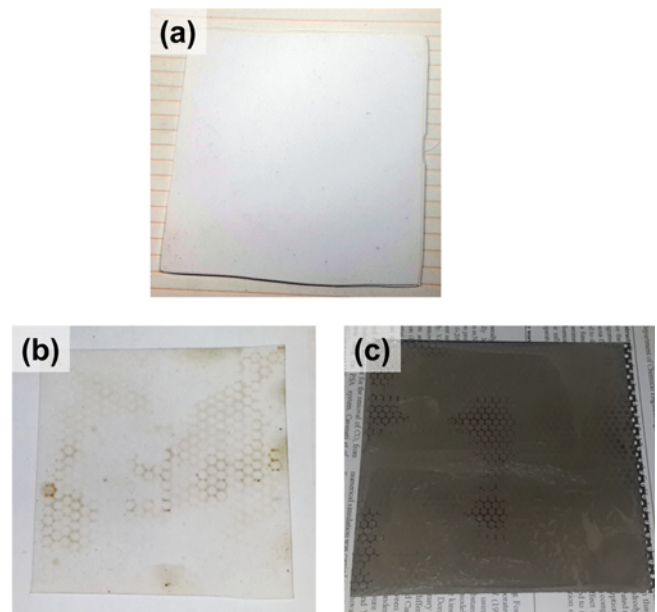


Fig. 5 Pictures of the electrolyte impregnated components (a) Matrix after burn out at 650°C (b) Li/Na electrolyte impregnated matrix; 80% of volume is filled with Li/Na electrolyte (c) Li/Na electrolyte impregnated cathode; 80% of volume is filled with Li/Na electrolyte

cathode and the burnt-out matrix were used.

The electrolyte impregnation process of the cathode was investigated using a contact angle analyzer (OCA 25, Dataphysics).^{7,8} Fig. 4 presents pictures of the electrolyte impregnation process. The cathode with the size of 10 mm 10 mm was placed on the quartz plate. Li₂CO₃ and Na₂CO₃ powders with the molar ratio of 52:48 (eutectic composition) were distributed on the cathode. After that, the chamber was heated up to 650°C with a heating rate of 2°C/min. The electrolyte powder on the cathode did not melt until the chamber was heated up to 510°C. After the chamber was heated to 516°C, most of the electrolyte had melted and immersed into the cathode.

For example, 80% of the cathode pores and the matrix pores were filled with electrolyte. Fig. 5(a) shows the results of the matrix after burn out. Fig. 5(b) shows (Li_{0.52}/Na_{0.48})₂CO₃ electrolytes impregnated matrix. Fig. 5(c) shows the electrolyte impregnated cathode. Using these electrolyte impregnated components, the characteristics of the electrolyte impregnation process were studied. There was no large difference between the conventional components and electrolyte impregnated components in terms of the external appearance.

Fig. 6(a) presents the scanning electron microscope (SEM) images of the matrix after burn-out at 650°C. Fig. 6(b) presents the SEM image of the electrolyte impregnated matrix. Fig. 6(c) and (d) present SEM images of the cathode before and after the electrolyte impregnation process. Both in the matrix and the cathode, electrolytes on the components were melted and impregnated into the components. Finally, the melted electrolytes were solidified and the pores of the matrix were filled with (Li/Na)₂CO₃ electrolyte after the electrolyte impregnation process.

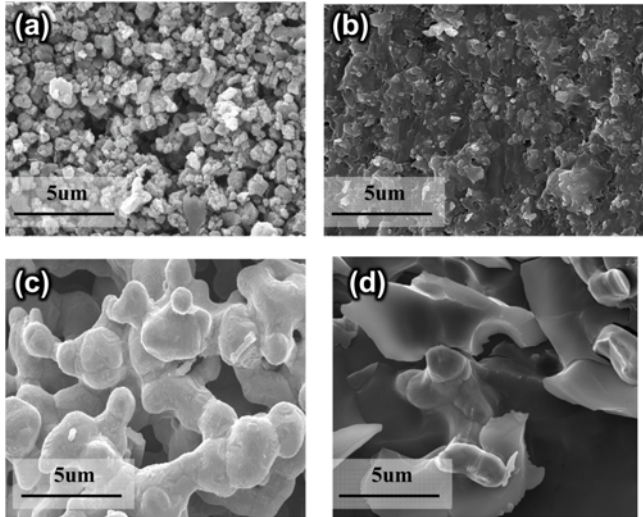


Fig. 6 SEM images of the electrolyte impregnated components (a) Matrix after burn out at 650°C (b) Li/Na electrolyte impregnated matrix; 80% of volume filled with Li/Na electrolyte (c) NiO Cathode after sintering at 800°C (d) Li/Na electrolyte impregnated cathode; 80% of volume filled with Li/Na electrolyte

Table 1 Operating condition of the 25 cm² single cell

Pressure	1 atm	
Sealing pressure	0.2 MPa	
Gas utilization (Anode H ₂ / cathode O ₂ and CO ₂)	0.1 at 150 mA/cm ²	
Gas flow rate	Anode	357 sccm
	Cathode	952 sccm
Input gas Composition	Anode	H ₂ /CO ₂ /H ₂ O = 0.72:0.18:0.1
	Cathode	Air/CO ₂ = 0.7:0.3

2.2 Operation of the 25 cm² Single Cell with Electrolyte Impregnated Cathode and Matrix

The operating conditions and gas compositions are summarized in Table 1. Fig. 7 presents the cell frame of the 25 cm² single cell with electrolyte impregnated components. A perforated sheet was employed as the current collector.⁹ In the operation of the single cell, a Ni - Ni5wt%Al anode, a (Li_{0.52}/Na_{0.48})₂CO₃ electrolyte impregnated cathode, and a (Li_{0.52}/Na_{0.48})₂CO₃ electrolyte impregnated γ -LiAlO₂ matrix were used.

Operating conditions are summarized in Table 1. The single cell was operated at 650°C, 620°C, 600°C, 580°C and 550°C. The gas utilization for the anode side (H₂) and the cathode side (O₂ and CO₂) were fixed to 0.1 at 150 mA/cm². The reaction of the single cell is 25 cm². In order to secure close contacts among components, a sealing pressure with a value of 0.2 MPa was applied to the single cell. The current collector was inserted into the cell frame.

Table 2 presents the amount of electrolytes in the standard 25 cm² single cell and the 25 cm² single cell with electrolyte impregnated components. In the standard 25 cm² single cell, 11.8 g of tape-casted electrolyte green sheets were used. In electrolyte green sheets, the

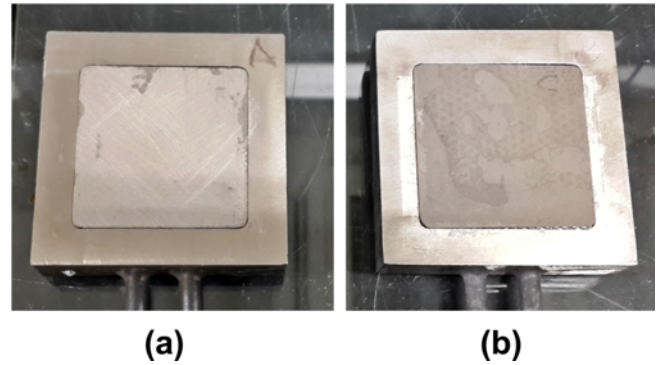


Fig. 7 Cell frame of the 25 cm² single cell (a) Anode cell frame (b) cathode cell frame with (Li/Na)₂CO₃ electrolyte impregnated cathode

Table 2 Comparison of components weight of the standard single cell and the single cell with electrolyte impregnated components

	Standard 25 cm ² single cell	25 cm ² single cell with electrolyte impregnated components
Anode	6.02 g	5.98 g
Matrix	10.44 g (green sheet)	12.60 g (6.00 g of electrolyte)
Electrolyte	11.80 g (green sheet) 8.40 g of electrolytes	8.40 g of electrolyte
Cathode	3.46 g	6.76 g (2.4 g of electrolyte)

*Li₂CO₃ and Na₂CO₃ electrolytes with the composition of 52:48

amount of (Li_{0.52}/Na_{0.48})₂CO₃ electrolyte was 8.4 g. This quantity of electrolyte can fill 20% of anode pores, 40% of cathode pores, and 100% of matrix pores.

In the case of the single cell with electrolyte impregnate components, a total of 8.4 g of electrolytes was required. First, 90% of matrix pores were filled with electrolyte. In that case, 6.0 g of electrolyte powders were employed in the matrix. Residual electrolytes of 2.4 g were impregnated to the cathode. 87% of cathode pores were filled with electrolytes. By impregnating electrolytes into the matrix and the cathode, the required electrolyte can be loaded in the single cell. As a result, it was not necessary to impregnate the anode with electrolyte.

3. Results

Fig. 8 presents the IV characteristics of the 25 cm² electrolyte impregnated single cell and the 25 cm² conventional cell. As the operating temperature decreases, the performance of the cell decreases. The gas utilization is 0.1 and reactive gases are inserted to the cell frame in excess. As a result, the effect of the mass transfer resistance is not dominant and I-V curves are nearly linear. At 650°C, 620°C, 600°C, 580°C and 550°C, cell voltages at 160 mA/cm² were 0.860 V, 0.835 V, 0.802 V, 0.749 V, and 0.641 V, respectively.

Compared with the conventional 25 cm² single cell using electrolyte

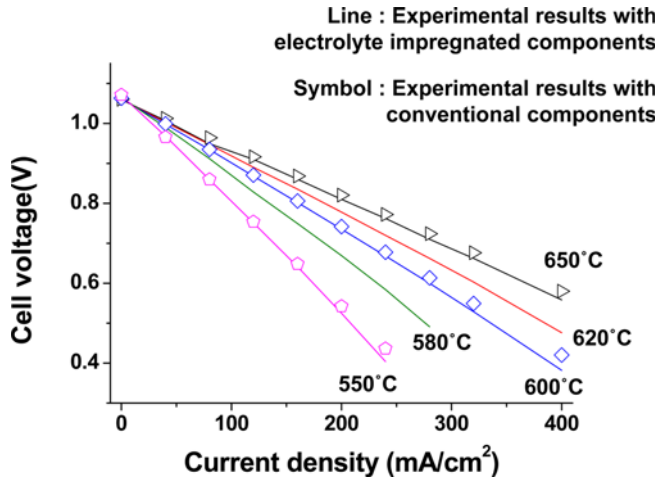


Fig. 8 IV characteristics of the electrolyte impregnated single cell and conventional cell at various operating temperatures

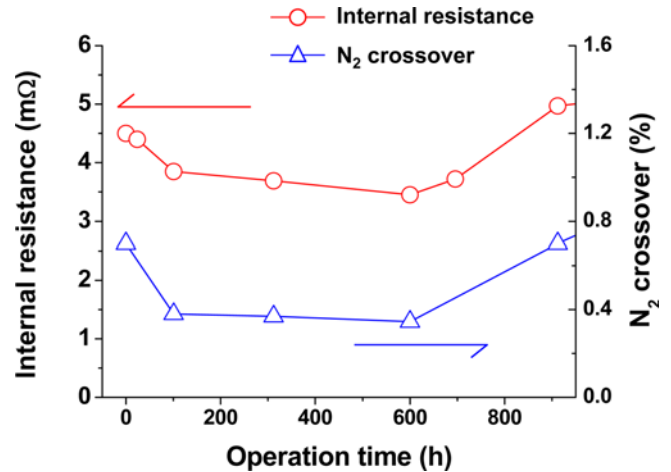


Fig. 10 Distribution of the internal resistance and the N_2 crossover at the anode gas outlet

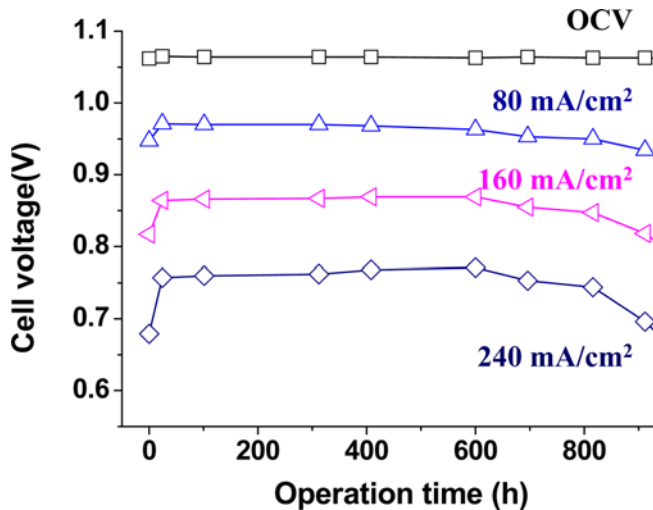


Fig. 9 Results of the long-term operation of 25 cm^2 single cell for electrolyte impregnated components

green sheet, the performance of the electrolyte impregnated cell was slightly lower; the cell voltage of the conventional 25 cm^2 single cell at 650°C and 160 mA/cm^2 was 0.868 V , giving a difference of only 8 mV .

The performance of the electrolyte impregnated cell with respect to operation time at 650°C is presented in Fig. 9. During long-term operation, the current density of 160 mA/cm^2 was applied. Fig. 10 presents the distribution of the internal resistance and the N_2 crossover at the anode gas outlet. At the first stage of operation, the performance of the cell is very low compared to normal condition. As the operating time increases, the performance of the cell increases until 600 hours of operation.

In the earlier stage of the operation, N_2 crossover and internal resistance is large compared to normal condition. As the internal resistance and the N_2 crossover at the anode gas outlet decreases, the performance of the cell increased. However, after 600 hours of operation, the internal resistance of the cell and the N_2 crossover at the anode gas outlet increased. At the same time, the performance of the cell decreased. Generally, due to the electrolyte loss in the matrix cause

gas crossover which is a critical type of damage in the long-term operation. Lack of electrolyte in the matrix made internal resistance increase and the performance of the fuel cell decrease.^{10,11}

At the first stage of operation, the electrolyte in the cathode and the matrix (87% of cathode pores and 90% of matrix pores) were not repositioned to the anode, cathode, and matrix. As the operation time increases, liquid electrolytes were repositioned with objective impregnation ratio (20% of anode pores, 40% of cathode pores, and 100% of matrix pores) due to the capillary force of porous components. Finally, the performance of the cell stabilized.

The performance difference between the conventional cell and the electrolyte impregnated cell was the difference of the internal resistance. For the conventional cell, the internal resistance was less than $3 \text{ m}\Omega$. Increased internal resistance of the electrolyte impregnated cell reduces performance of the electrolyte impregnated cell than conventional cell.

4. Discussion

4.1 Analysis of the Components After Cell Operation

Fig. 11 presents the SEM images of electrolyte impregnated cell components after 1000 hours of operation. After cell operation, only microstructure of the cathode was changed from Ni to NiO due to cathode atmosphere (70% Air + 30% CO_2).^{12,13} The microstructure of Ni and NiO can be distinguished from the SEM images. Microstructure of Ni is connected to each other due to the sintering process as shown in Fig. 6(c). However, microstructure of NiO was not connected each other as shown in Fig. 11(d). The size of the NiO sphere is smaller than that of Ni.

Microstructures of the matrix and the anode did not change. The SEM image of the matrix presented in Fig. 11(a) is not significantly different to that observed in a conventional matrix after cell operation.

Around LiAlO_2 particles, solidified electrolyte was placed. Most of pores were filled with electrolyte. Fig. 11(c) presents the SEM image of the anode after cell operation. Before the cell operation, electrolyte

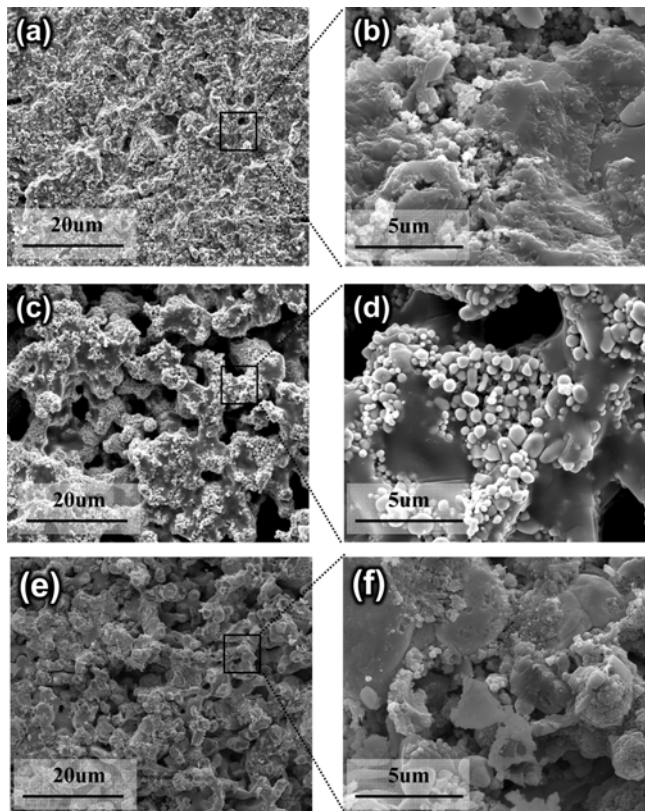


Fig. 11 SEM images of components of 1000hours operation (a) Matrix, (b) detailed image of matrix, (c) cathode, (d) detailed image of cathode, (e) anode, and (f) detailed image of anode

was not present in the anode. However, after cell operation, electrolyte was found near Ni-Ni5wt%Al particles.

Fig. 11(b) presents the SEM image of the cathode after cell operation. Before cell operation, pores of the cathode were filled with electrolyte as shown in Fig. 6(d). After cell operation, the Ni particles were oxidized to NiO and electrolyte was found near NiO particles. Electrolyte filled in the cathode side moved to the matrix and the anode. Microstructure of Ni is connected to each other due to the sintering process as shown in Fig. 6(d). However, microstructure of NiO was not connected each other as shown in Fig. 11(b). The shape of NiO is spherical shape. The size of the NiO sphere is smaller than that of Ni.

The amount of remaining electrolyte in the components after cell operation was measured by the ASTM C373-88 procedure.^{14,15} Using acetic acid which did not affect the particles of matrix, anode and cathode, remaining electrolyte in components was removed. By measuring the weight of removed electrolyte in components, the remaining electrolyte in the components after the cell operation was obtained. The remaining electrolyte was measured by using 5 specimens after cell operation.

Before cell operation, the electrolyte was impregnated in the matrix pores and the cathode pores are 90% and 87%, respectively. After cell operation, the average remaining electrolyte in the matrix pores, the cathode pores and the anode pores are 94.62%, 42.75%, and 21.56%, respectively. Liquid electrolyte was relocated in the operating condition

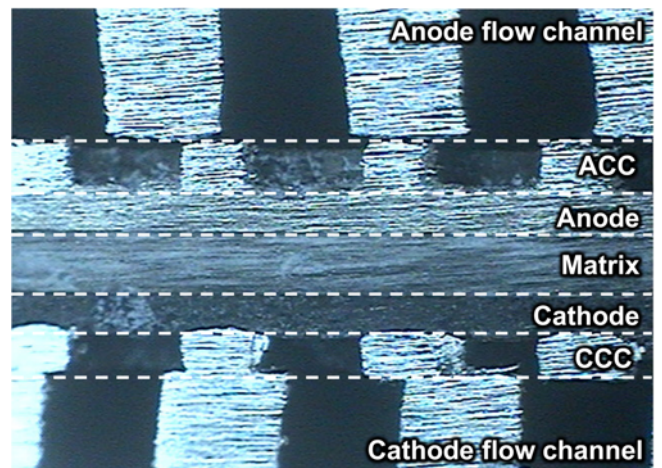


Fig. 12 Sectional view of the cell frame after operation of 1000 hours

Table 3 Change of the thickness before and after operation with electrolyte impregnated components

	Anode	Matrix with electrolyte	Cathode	Wet-seal
Initial thickness (mm)	0.72	1.10	0.70	1.22
Thickness after operation (mm)	0.71	0.98	0.66	0.99
Variation (mm)	0.01	0.12	0.04	0.23
Magnitude of change (%)	1.39	10.91	5.71	18.85

of MCFCs, and the amount of remaining electrolyte was close to the target electrolyte impregnation ratio (100% of matrix pores, 40% of cathode pores, and 20% of anode pores). These changes of the remaining electrolyte in pores are the evidence of the electrolyte reposition. From these results, it was found that the impregnated electrolytes in the cathode and matrix moved between the anode, cathode and matrix due to capillary forces.

4.2 Change of the Thickness After Operation of 25 cm² Single Cell

In order to investigate the change of thickness for each component such as anode, matrix, and cathode, the cell frame was cut after 1000 hours of operation to produce a cross-section for analysis. Fig. 12 shows this cross-sectional view of the cell frame. With this visual inspection, the thickness of anode, matrix, and cathode was measured.

The thickness of electrolyte impregnated components before and after cell operation is listed in Table 3. The initial thickness of the electrolyte impregnated matrix was 1.10 mm. After cell operation, the thickness was 0.98 mm and decreased by 10.91% from the initial thickness. The change in the thickness of the matrix was the largest among all components, because the matrix was composed of γ -LiAlO₂ which is not sintered at the operating condition of MCFCs with a sealing pressure of 0.2 MPa. The thickness of the anode and the cathode was decreased by 1.39% and 5.71% respectively. The change in the thickness of the anode was very small, because Ni-Ni5wt%Al was employed after sintering at 1000°C in the reduced atmosphere in the anode. The change in the thickness of the cathode was 5.71% and

was as a result of oxidation from Ni to NiO in the cathodic atmosphere with $(\text{Li}/\text{Na})_2\text{CO}_3$ electrolyte.¹⁶ The distance from the cathode side wet-seal and the anode-side wet seal was initially 1.22 mm and decreased to 0.99 mm after cell operation.

In the conventional single cell, tape-casted electrolyte green sheets with a thickness of 1.5 mm were used between matrix green sheets.

These tape-casted electrolyte green sheets were melted in the pre-treatment process and to introduce them into the anode, cathode, and matrix. Before cell operation, the thickness of the matrix and electrolyte green sheets were 2.7 mm. After cell operation, the thickness was 1.06 mm and decreased by 60.74%.

In the conventional single cell using electrolyte green sheets, the distance from the cathode side wet-seal and the anode-side wet seal was decreased by 1.74 mm. This change of the distance due to electrolyte melting was acceptable in the operation of the single cell. However, in the stack of MCFCs, hundreds of cells were assembled. In the stack, the cumulative thickness of electrolyte green sheet was very large. Also, in the pre-treatment process, there can be problems related to the melting of electrolyte. These problems include: non-uniform electrolyte melting and thickness change of the MCFC stack. These non-uniform deformations could result in critical problems in operation of MCFCs such as gas crossover in the matrix due to fractures.

By employing electrolyte impregnated components, these problems resulting from electrolyte melting and immersion to components can be avoided. Fractures of components in the assembling process of stacks and operations can be avoided.

In addition to the reduced change in the thickness, the pre-treatment was simplified by employing electrolyte impregnated components. In the conventional cell, removal rates and the temperatures of binders and organics in the green sheets of the matrix and electrolyte should be considered. In the 100 cm² single cell, the pre-treatment time was 1 week.¹⁷ However, the pre-treatment schedule can be simplified by employing electrolyte impregnated components. It was decreased to 48 hours.

5. Conclusion

In this work, the electrolyte impregnated cathode and matrix for MCFCs were fabricated and employed in single cell tests. In the cathode and the matrix, the electrolyte was filled with 90% of matrix pores and 87% of cathode pores. The characteristics of a 25 cm² single cell with electrolyte impregnated components were investigated. In the early stage of operation, N₂ crossover at the anode gas outlet and the internal resistance of the cell was lower. As the operating time increased (120 hours), electrolyte in the cathode and the matrix relocated and as a result, N₂ crossover and the internal resistance decreased. Finally, the performance of the cell was improved to be similar to that of the conventional single cell.

The thickness change of components was found to be minimized by employing electrolyte impregnated components. In the single cell with electrolyte impregnated components, the change of the thickness in components was reduced by 86.78% (from 1.74 mm to 0.23 mm) compared to the conventional cell. Through the use of electrolyte impregnated components, minimized thickness change will decrease the cumulative impact of thickness change in stacks of MCFCs. Also,

problems in the pre-treatment such as non-uniform impregnation of electrolytes and thermal stress of components can be prevented.

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