Fabrication of electrolyte-supported solid oxide fuel cells using a tape casting process

Youngjin KWON¹ and Youngbae HAN¹,†

¹Department of Mechanical System Engineering, Korea Military Academy, 574 Hwarang-ro, Nowon-gu, Seoul, Republic of Korea

The high energy density of hydrogen, in addition to its convenience for transportation and infinite resource base, make it a promising energy carrier. Solid oxide fuel cells (SOFCs), in particular—which utilize the oxidation of hydrogen at high temperatures to generate electricity—have been studied widely because of their high efficiency and relatively low cost. However, the lack of a suitable mass production method currently precludes the commercialization of SOFCs. To address this, we herein evaluate tape-casting as a means to reduce the cost of SOFC mass production. A simple de-airing technique is used to simplify the production process and an electrolyte-supported SOFC is produced without employing a buffer or functional layers. The rheological properties of green tape slurries are explored to improve tape completeness and electrolyte performance. Electrolyte conductivity is measured for a fabricated half-cell; the fine structural details are analyzed via scanning electron microscopy. As a result, a unit cell with an open-circuit voltage of 1.05 V and an electric power density of 0.476 W cm⁻² at 800 °C was fabricated.

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1. Introduction

Recently, increasing numbers of studies on new and renewable energy are being performed because of fossil fuel depletion and environmental issues. Although there are various new and renewable energy sources including solar and wind power that have numerous ecological benefits, these sources necessitate additional energy storage systems due to the time gap between generation and consumption.¹,² The numerous advantages of H₂, e.g., its production of water as the only combustion by-product, high natural abundance, and ease of storage/transportation, make it a promising energy carrier.³,⁴,⁵

Fuel cells have been extensively studied as devices that enable the use of hydrogen energy. The advantages of solid oxide fuel cells (SOFCs), which feature ceramic electrodes and electrolytes, include high energy conversion efficiency and fuel flexibility. However, the high cost of these cells and the lack of materials suitable for use under the harsh SOFC operation conditions hinder the commercialization of SOFCs.⁶,⁷ Hence, a manufacturing process to reduce the production cost per unit of SOFC is required.

Tape-casting is a promising technique that is used in the manufacturing of cells and tapes from ceramic-polymer mixtures and exhibits the advantages of large-scale reproducibility and low cost.⁷ Consequently, tape-casting has been widely studied and is suggested as a manufacturing method that can enable the mass production of SOFCs along with cost reduction.⁸–¹¹

An SOFC consists of an anode, electrolyte, and cathode. In our previous research, an electrolyte was prepared by impregnating a Laₐ₀.Sr₀₂.Ga₀.₈₅Mg₀.₁₅O₃-δ (LSGM) structure from an aqueous solution.¹²,¹³ This method provided enhanced electrode performance, but such a structure cannot be used as a supporter due to its poor mechanical strength. Therefore, in this study, the production of an LSGM electrolyte to support SOFCs using a tape-casting process was explored.

Electrolyte performance is determined by the ionic conductivity and thickness of its constituent material.¹⁴,¹⁵ Although LSGM has a greater ionic conductivity than other widely used materials, such as yttrium-stabilized zirconia, gadolinium-doped ceria, and scandia/ceria-doped zirconia, its use is limited due to performance degradation upon reacting with electrode elements.¹⁶,¹⁷ However, as reported by Yoon et al., LSGM does not react with electrode elements below a certain temperature.¹⁸–²⁰

Accordingly, this study was performed to develop a means of producing thin LSGM electrolyte tapes with a simplified manufacturing process. In our previous studies on tape-casting, we found that the viscosity of the slurries used in the manufacture of tapes has a critical effect on their properties and ease of handling.²¹,²² Excessive slurry viscosity can result in defective tapes with air bubbles and, consequently, poor cell performance caused by irregular-
ities in the structure. Conversely, if the viscosity is too low, tapes cannot be manufactured.

In this study, slurry viscosity was adjusted by controlling the amounts of powder and solvent instead of creating a vacuum environment for de-airing, which is the common method for controlling viscosity. This simplified the manufacturing process. The optimal viscosity profile of the slurry was determined and its shear modulus slurry was measured according to the ratio of powder and solvent. Some materials such as Ni, the material most widely used to create anodes, were producing second phase with the slurry was determined and its shear modulus slurry and the slurry was placed in a plastic tank for deaeration approximately 5 rpm. If the above process is not properly conducted, the presence of air in the slurry may compromise overall tape quality. The completed slurry was cast on Mylar film at a speed of 20 mm min⁻¹ using a doctor blade and it was dried for 24 h to produce tapes. The shear rate and rheological/dynamic properties of the slurry were measured by steady-state and oscillation stress amplitude sweep tests. The tensile strength according to the proportions of solvent and powder was also tested by using a universal testing machine (UTM, Instron 5583, Instron Corporation, USA). The sample size was 10 mm × 10 mm × 1 mm, and its elongation rate was maintained at 2 mm min⁻¹.

The tapes produced through the above process were used to make plates by punching them at a diameter of 3 cm, pressing with zirconia plates (3 cm × 3 cm × 2 mm, 9 g), and sintering at 1450 °C while applying pressure. Heat treatment was conducted by first increasing the temperature at 1 °C min⁻¹ until the solvent was evaporated at 80 °C and maintaining the temperature for 30 min. Then, the temperature was raised at 2 °C min⁻¹ to 1300 °C before sintering. To heat up the solvent to 1450 °C for sintering, the temperature was increased at 1 °C min⁻¹ and the temperature was maintained at 1450 °C for 4 h. Finally, it was decreased to ambient temperature at 3 °C min⁻¹.

The same method as was used in the previous study to produce an LSGM plate. A porous structure of scaffolds was made onto the LSGM plate and an impregnation process was conducted using LSCF and PBMO solvent. The half-cell was produced by impregnating PBMO using the four-electrode method and the single cell was prepared by impregnating the anode and cathode with PBMO and LSCF, respectively. Figure 1 shows the overall cell production process. The compositions of the slurries are summarized in Table 1. SEM was performed at an electron acceleration voltage of 15 kV to confirm that the electrolyte of the cell was densely formed and that the electrode provided sufficient porosity.

| Solvent ratio (%) | Ceramic powder (g) | Solvent (g) | Dispersant (g) | Plasticizer (g) | Binder (g) |
|------------------|--------------------|-------------|---------------|----------------|-------------|
| LSGM MEK Ethanol Triton-100 DBP PEG Butvar | 23 30 6.75 4.5 0.195 2.19 2.36 | 26.5 30 8.25 5.5 0.195 2.19 2.36 | 30 30 9.75 6.5 0.195 2.19 2.36 | 33.5 30 11.25 7.5 0.195 2.19 2.36 |

Table 1. Composition of green tape slurry

![Diagram](image-url) Fig. 1. Single cell production process.

2. Materials and methods
2.1 Slurry preparation, tape-casting, and heat treatment
First, a solvent was prepared by mixing methyl ethyl ketone (J. T. Baker) and ethanol at an azeotropic ratio of 6:4 (w/w) and, after adding LSGM electrolyte powder (fuel cell materials) and Triton X-100 (Sigma-Aldrich) dispersant, ball milling was conducted for 16 h. During the process, the viscosity of the slurry was adjusted by controlling the ratio of the solvent and LSGM powder. Polyethylene glycol (Sigma-Aldrich) plasticizers, dibutyl phthalate (DBP; Junsei), and Butvar (Sigma-Aldrich) binder were then added, and further ball milling was performed for 4 h. Then, the zirconia balls were displaced, and the slurry was placed in a plastic tank for deaeration for 24 h. The de-airing process was conducted to remove internal bubbles by slowing the ball speed to approximately 5 rpm. If the above process is not properly conducted, the presence of air in the slurry may compromise overall tape quality. The completed slurry was cast on Mylar film at a speed of 20 mm min⁻¹ using a doctor blade
2.2 Assessment of electrochemical performance

The half-cell thus produced was loaded on a jig installed on the inside of a quartz holder to create a fueling environment, and Pt mesh and Pt paste were used as the current collector. The holder was placed in the electric furnace in order to reduce the PBMO electrode material, and the temperature was increased to 800 °C to create a 10% hydrogen environment (H₂/N₂ = 1:9, v/v). The half-cell performance was assessed by measuring AC impedance with a Solatron 1287/1260, while sealing it to prevent hydrogen leaks and supplying humidified hydrogen at 1000 sccm. The single cell was sealed using Aremco Ceramabond 571 on the aluminum tube and both Pt mesh and Pt paste were applied. Then, humidified hydrogen at 200 sccm and air at 300 sccm were supplied. The current–voltage (I–V) curve and AC impedance were also measured using a Solatron 1287/1260.

3. Results and discussion

Recently, several studies have been published on electrolyte-supported SOFCs, some of which showed outstanding results, such as thin electrolytes with excellent durability. However, the disadvantages of such cells are performance degradation upon thickening of the electrolyte and the complicated production processes needed to prepare functional layers.8,25–28 Although the functional layer ensures high performance with the expansion of the three-phase boundary, it also leads to increased production time and complexity in additional production phases.29,30 Studies on the co-tape casting method have also been conducted to simplify the production process. However, in this method, it is difficult to conduct heat treatment due to differences in the shrinkage behaviors of the constituent materials. Furthermore, it is time-consuming as the tape produced after first casting must be cast again.24

In the present study, the solvent evaporation process was excluded during de-airing, the slurry viscosity was adjusted by controlling the solute to solvent ratio, and the de-airing was carried out using ball milling to simplify the entire process. The rheological properties were measured and their effects on the quality of tape were investigated. Moreover, the electrochemical characteristics of the single cell produced by impregnating PBMO and LSCF were analyzed.

3.1 Production of green tape

The important properties of tape-casting include viscosity, the shear modulus of slurry, tensile strength, and pecking density of green tape.24 Excessive viscosity limits the ability to control slurries and cast tapes. Conversely, a viscosity that is too low may cause phase segregation to occur. Furthermore, a low shear modulus may hinder the creation of an internal network and a low tensile strength can disrupt slurries because it is difficult to maintain their shapes. Therefore, this study determined the ideal proportions required to produce a slurry with a suitable shear modulus, tensile strength, and viscosity. Slurries were produced using solvent ratios of 33.5, 30, 26.5, and 23%, and their rheological properties were measured and analyzed.

Figure 2(a) shows the changes in viscosity according to solvent ratio. In previous studies, the proper viscosities were found to be 0.5–20 Pa s at 10 s⁻¹.21,22,24,31 In the present study, defects were found in the tape as the slurries overflowed while casting at a 33.5% solvent ratio (4.252
Pas at 10 s^{-1}). Tape-casting was also difficult using a 23% ratio due to the excessive viscosity of 42.01 Pas at 10 s^{-1}. However, the tape-castings were successful at solvent ratios of 30 and 26.5% and the properties were controlled relatively well.

Figure 2(b) shows the storage shear modulus of slurry according to solvent ratio. The storage modulus represents the attractive force caused by particle interactions in slurries; a higher storage modulus indicates a greater electrochemical performance.32),33) As Fig. 2(b) shows, the storage shear modulus increases as the solvent ratio decreases. The comparison of storage modulus values at a shear stress of 10 Pa shows that the slurry with a solvent ratio of 23% has the highest storage modulus value of 25.23 kPa and that with a solvent ratio of 33.5% exhibits a relatively low storage modulus value of 10.12 kPa. However, the tape shape was difficult to maintain when a the latter solvent ratio was applied, and it was difficult to control the tape-casting process with a solvent ratio of 23% due to its excessive viscosity.

When comparing the storage moduli between 26.5 and 30% solvent ratios at a shear stress of 10 Pa, it was found that the value is higher at the 26.5% ratio (19.88 kPa) than that at the 30% ratio (14.12 kPa). Hence, the ideal solvent ratio with proper viscosity and shear modulus to produce optimal tapes was found to be 26.5%.

In addition, tensile strength is critical for maintaining tape shape and preventing slurries from overflowing. In previous studies, green tapes were produced to have tensile strengths of 1.5–2 MPa.34),35) Figure 2(c) shows tensile strength and packing density of green tape as a function of solvent ratio. The tensile strength at a solvent ratio of 33.5% is 1.11 MPa; for ratios of 30, 26.5, and 23%, it is in the range of 1.78–3.25 MPa. We confirmed that a solvent ratio of 33.5% results in a low tensile strength unsuitable for maintaining the shape of the tape. As the packing density of green tape is an index of ceramic particle clustering therein, it is positively correlated with tape sinterability and cell density.24) Figure 2(c) shows that packing density decreases as the solvent ratio increases, and this indicates that the solid content of the slurry has a marked effect on the green tape.

The dynamic and rheological properties of the green tapes were assessed according to solvent ratio in order to produce cells with outstanding performance. Four different solvent ratios (23, 26.5, 30, and 33.5%) were assessed. Viscosity, storage modulus, and tensile strength were measured. The green tape fabricated at a 33.5% solvent ratio failed to maintain its shape as the slurries overflowed during the process and the tensile strength was low. Moreover, the tape-casting process for the 23% slurry was difficult because of excessive viscosity. Although solvent ratios of 26.5 and 30% imposed no limitations on producing green tapes, it was determined that green tape fabricated at a 26.5% solvent ratio is more suitable for producing cells with excellent performance as its storage modulus and tensile strength were higher than those obtained for the tape fabricated at a 30% solvent ratio.

3.2 Assessment of the half-cell

The electrolyte located at the center of an SOFC separates the fuel electrode from the air electrode to prevent fuels from mixing with air; this also moves oxygen ions from the air electrode to the fuel electrode. Hence, the electrolyte for an SOFC should be composed of materials that are not ionically or electrically conductive and exhibit chemical and thermal stability over a wide temperature range.36) Electrolyte conductivity can be obtained using electrochemical impedance spectroscopy data based on the following formula:

\[
\sigma = c(zF)^2 D_0 e^{-\Delta G_{\text{act}}/RT},
\]

where \(\sigma\) is electrolyte conductivity (S cm\(^{-1}\)), \(c\) is oxygen concentration, \(z\) is electron charge, \(F\) is Faraday’s constant (C mol\(^{-1}\)), \(D_0\) is the diffusion coefficient (cm\(^2\) s\(^{-1}\)), \(\Delta G_{\text{act}}\) is the free energy of activation [J mol\(^{-1}\)], \(R\) is the universal gas constant [J K\(^{-1}\) mol\(^{-1}\)], and \(T\) is temperature (K).

Based on the above formula, the gradient of a plot of \(\log \sigma\) vs. \(1/T\) yields the activation energy. Figure 3 shows the results from this study and two earlier ones that used the above method.37),38) As Fig. 3 shows, the conductivity and activation energy of the LSGM were lower in the previous studies, suggesting that the LSGM electrolyte produced by applying the tape-casting method exhibits better performance than those prepared by other methods. This method produces a phase of the cell that has a relatively higher density and purity than the other methods. The thickness of the electrolyte decreases by 300 µm, which is thinner than an SOFC electrolyte produced by applying the LSGM electrolyte layer only. The former is predicted to exhibit better performance.18),39)

Although some studies have concluded that electrolyte performance can be enhanced by applying a buffer layer, this method is used to block the reaction of LSGM and Ni, which are highly reactive together.12),19),20),40),41) Unlike these methods, electrode scaffolds were produced in this study and they were combined with electrocatalysts at low temperature to avoid secondary phase formation. In addition, the same materials were used for both electrolyte and

![Fig. 3. Comparison of LSGM conductivities reported in previous works\(^{37,38}\) with those obtained in the present study.](Image 313x92 to 538x264)
electrode for better junction formation. This is also beneficial for simplifying the production process as it involves forming a single layer instead of dual layers.

**Figure 4** shows cross-sectional and surface SEM images of the electrolyte. The electrolyte and electrode formed are 400 and 25 μm thick, respectively [Fig. 4(a)]. These images also demonstrate that the electrolyte is dense and efficiently separates the fuels from air, as shown in Figs. 4(b) and 4(c). The electrode is porous and the electrochemical catalysts are well infiltrated. Infiling porous scaffolds with electrochemical catalysts is likely to be able to overcome the disadvantages of electrolyte-supported SOFCs with huge ohmic resistance because it expands the electrode three-phase boundary, resulting in better performance.9),14),18),20),42) Figures 4(d) and 4(e) show the well-distributed electrode catalysts in the porous scaffold.24)

### 3.3 Single-cell assessment

An LSGM-electrolyte-supported SOFC single cell was fabricated in this study and used to impregnated electrodes; the anode was PBMO and cathode was LSFC materials. **Figure 5** shows the $I-V$ curve that indicates the performance of the single cell. The maximum open-circuit voltage and power density of the single cell are 1.05 V and 0.476 W cm$^{-2}$ at 800 °C. The measured open-circuit voltage is close to the theoretical maximum value and the $I-V$ curve is a straight line. The single cell exhibits better performance than the LSGM-supported cell with a maximum thickness of 200 μm, as produced in an earlier study.41) The maximum power density observed in that study was 0.25 W cm$^{-2}$.

### 4. Conclusions

In this study, we analyzed the rheological properties and fine structural details of green tapes fabricated by applying
a tape-casting method to produce electrolyte-supported SOFCs and assessed the electrochemical performance of a half-cell. We conducted our analysis by applying four different solvent ratios and found that the best solvent ratio for fabricating optimal green tape is 26.5%.

A porous scaffold electrode was screen printed onto the electrolyte plate produced through the process, and the catalyst phase was combined at a low temperature after impregnating it with electrocatalysts. As suggested by a previous study, the same materials were used for both electrode and other materials. The present method enhances the performance by the expansion of the three-phase boundary as the electrochemical catalysts are dispersed at the nanoscale.

The SOFC half-cell produced with the electrode and electrolyte obtained using the present tape-casting method has higher ionic and electrical conductivities than those of other reported LSGM electrolytes. It was also confirmed that the single cell produced in this study has a maximum power density of 0.476 W cm\(^{-2}\) at 800 °C.

The tape-casting method may represent a critical advance in the commercialization of SOFCs as it is a useful approach for large-cell formation and enables their mass production. This study presents a method that avoids the de-airing process in tape-casting production and guarantees high performance without applying a buffer layer. The ideal conditions for producing optimal tapes were identified by analyzing the rheological properties of the resultant green tapes. Thus, this study provides useful reference data for producing SOFCs via the tape-casting process.

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