

Development of Ce/ $\text{La}_{0.25}\text{Sr}_{0.75}\text{Cr}_{0.5}\text{Mn}_{0.5}\text{O}_3$ Anode Catalyst for Further Application in Solid Oxide Fuel Cell Typed Reactor

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Abstract— $\text{La}_{0.25}\text{Sr}_{0.75}\text{Cr}_{0.5}\text{Mn}_{0.5}\text{O}_3$ (LSCM) and Ce/LSCM were synthesized and investigated for their potential use in an oxidative coupling of methane (OCM) reaction. The catalysts was characterized using XRD, SEM and Impedance. The catalytic activity of catalysts for OCM reaction was both studied in fixed bed and SOFC reactor. A YSZ was used as a solid electrolyte and $\text{La}_{0.8}\text{Sr}_{0.2}\text{FeO}_3$ (LSF) was used as a cathode material. The cell obtained a maximum power density of 221 mW cm^{-2} in humidified methane. The cell was put under a bias voltage of 0.3 V, which C2 hydrocarbon was generated from the cell. At the highest methane conversion rate, the cell achieved a good C2 hydrocarbon selectivity of 78.4% and a C2 yield of 3.4% at 850 °C.

Index Terms—SOFC reactor, cogeneration, C2 hydrocarbon, LSCM.

I. INTRODUCTION

Research on the oxidative coupling of methane (OCM) has increased in intensity over the past decade due to the potential for converting natural gas directly to higher value chemicals, such as ethylene and ethane (C2 hydrocarbon) [1], [2]. The conversion of methane to C2 hydrocarbons remains a challenging problem in heterogeneous catalysis and chemical reaction engineering. The selectivity to C2 hydrocarbon decreases with increasing methane conversion. In addition, the yield of C2 hydrocarbons achieved in a fixed-bed reactor (FBR) was limited to about 20-25% due to the presence of undesired complete oxidation in the gas phase and partially on the catalyst surface [3], [4]. In the last 25 years, numerous works have been reported and several innovative approaches have been proposed, including the use of solid electrolyte membrane reactors [5], [6]. However there are a few researches revealed SOFC reactor to produce C2 hydrocarbons. The SOFC reactor for co-generation of C2 hydrocarbons and electric power was first reported by Pujare and Sammells, they studied the OCM by using the cell $\text{Pt}/\text{Sm}_2\text{O}_3/\text{La}_{0.89}\text{Sr}_{0.10}\text{MnO}_3/\text{YSZ}/\text{La}_{0.89}\text{Sr}_{0.10}\text{MnO}_3/\text{Pt}$. High selectivity to C2 hydrocarbon (90 %) was achieved although the methane conversion was relatively low [7]. Hence, a

development of anode catalysts for an oxidative coupling of methane in SOFC becomes an important goal to improve the conversion, selectivity, yield and stability of the reactor. Addition, the anode material must require good electronic conductivity, high electrochemical activity and structural stability properties [8].

Perovskite structure (ABO_3) anode materials have received much attention because of their mixed electronic and ionic conduction behaviors that make the triple-phaseboundary (TPB) extend to the entirely exposed anode surface. Among the variety of these oxides, LaSrMnO_3 -based perovskite compounds are promising Ni-free anode candidates due to their high chemical stability at high temperatures under both oxidizing and reducing atmospheres, and strong resistance to carbon deposition [9], [10]. However, they suffer from the disadvantages of low electronic and ionic conductivities and poor catalytic activity. The sufficient electronic and ionic conductivities are indispensable to reduce the electrode polarization and thus promote the electrode reaction process. There are many efforts to enhance the electronic and ionic conductivities of LaSrMnO_3 material. For example donor doping on the Mn site of LaSrMnO_3 with perovskite structure could improve the electrical conductivity [11]-[15]. S.W. Tao and co-worker reported the increasing in electrical conductivity of doping on the Mn site of LaSrMnO_3 . Moreover doped-chromium on Mn site is suitable for high temperature operation (750-900°C) because Cr doped LaSrMnO_3 played a vital role in increasing resistance to coking (as well as raising sulphur tolerance) [16]. Moreover, Ceria doped perovskite material, the electrochemical catalytic performance and carbon deposition resistance in oxidation of syngas were significantly improved [17].

In this work, $\text{Ce}/\text{La}_{0.25}\text{Sr}_{0.75}\text{Cr}_{0.5}\text{Mn}_{0.5}\text{O}_3$ (Ce/LSCM) were examined. The surface morphology, composition and surface area were characterised by several techniques. The catalytic activity of catalysts for oxidative coupling of methane reaction and the influence of some operating variables, i.e., the temperature were investigated in detail over a fixed bed reactor. For SOFC cell preparation, the cell components was improved by tape-casting preparation. The ohmic resistance decreased by the reduction of the electrolyte thickness. Finally, the electrochemical performance was investigated in solid oxide fuel cell reactor (SOFC).

II. EXPERIMENTAL

A. $\text{La}_{0.25}\text{Sr}_{0.75}\text{Cr}_{0.5}\text{Mn}_{0.5}\text{O}_3$ Catalyst Preparation

The perovskite oxide, $\text{La}_{0.25}\text{Sr}_{0.75}\text{Cr}_{0.5}\text{Mn}_{0.5}\text{O}_3$ (LSCM),

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was prepared by sol-gel method. In this process, stoichiometric quantities of $\text{La}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ (Aldrich, 99.99%), $\text{Mn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ (Aldrich, 98%), $\text{Cr}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ (Aldrich, 99.99%), and $\text{Sr}(\text{NO}_3)_2$ (Aldrich, 99.99%) precursors were dissolved into water. Hence, an aqueous solution of citric acid (Riedel de Haën, reagent grade) and ethylene glycol (Aldrich, 98%) was added to the metal solution (citric acid: metal nitrates [acetate]: ethylene glycol molar ratios=5:1:4.3). The mixture was stirred at 70 °C for 12 h. Water was slowly evaporated on a hot plate and the resulting brown gel was dried and then heated up in air at 400 °C for 2 h in an oven. LSCM perovskite samples were obtained by ball-milling the decomposed resin for a few hours before calcination at 1000 °C for 2 h in air with heating rate 6 °C/min).

B. Catalysts Characterization

XRD patterns of the $\text{La}_{0.25}\text{Sr}_{0.75}\text{Cr}_{0.5}\text{Mn}_{0.5}\text{O}_3$ (LSCM) catalyst were obtained by using X-ray diffractometers, D 5000 (Siemens AG) using $\text{Cu K}\alpha$ radiation equipped with Ni filter with a range of detection of $2\theta = 20\text{-}80$ and a resolution of 0.02°. X-ray diffractometer connected with a personal computer using Diffract AT version 3.3 for a full control of the XRD analyzer. SEM (model JEM-6400 Jeol Ltd., Japan) techniques were used to study the catalyst morphology. The SEM was operated in the back scattering electron (BSE) mode at 15 kV.

C. Catalytic Activity Measurement

The catalytic of oxidative coupling of methane over LSCM catalysts was performed at atmospheric pressure and at temperatures of 700-900 °C in a quartz micro-reactor (i.d. 6 mm) placed in a ceramic tube furnace. A total of 0.3 g of fresh catalyst was loaded into a reactor that was located in a programmable furnace with a thermocouple placed in the center of the catalyst bed, and 4:1:5 $\text{CH}_4\text{:O}_2\text{:N}_2$ molar ratios of the feed were used. The gas products were sampled every 30 min for measuring their compositions using an on-line gas chromatography equipped with a TCD detector.

D. Preparation of LSF//YSZ//LSCM and LSF//YSZ//Ce-LSCM Single Cell

The single cell was prepared in YSZ 3-layer cells electrolyte and porous similar in somewhere [13]. LSCM was added to anode porous layers using aqueous nitrate solutions until the loading of LSCM was 40 wt.%. Then, the sample was transferred to the furnace where the nitrate decomposition was performed in a stagnant air. The heating treatment consisted of heating rate of 3 °C/min to 1200 °C and dwelling at this temperature for 2 hrs before cooling down to room temperature. After the heating treatment, the perovskite of the electrode material should deposit on the YSZ porous scaffold surface. For cathode side, $\text{La}_{0.8}\text{Sr}_{0.2}\text{FeO}_3$ (LSF) cathode was added after the final 1200 °C calcinations used to produce the LSCM, and was calcined at 850 °C. Additions of CeO_2 at the anode were impregnated in separate stages. In this case, the drying temperature was 400 °C, and they were calcined at 700 °C. Phase composition and metal oxide distribution inside the porous substrate were investigated by SEM-EDS.

E. Performance of SOFC Cell

In this study, H_2 and CH_4 were used as the anode gas. Humidified H_2 and CH_4 were achieved by bubbling gas through room-temperature water resulting in a gas mixture of 3 % H_2O and fuel. The flow rate of gas was controlled by a mass flow meter.

The open circuit voltage (OCV) of the cell under testing condition was recorded as a function of time until the system reached the steady state before performing the electrochemical measurements. The electrochemical measurements were carried out using a frequency response analyzer (Solartron 1255, UK) coupled with an electrochemical interface (Solartron 1287, UK). The voltage-current (V-I) measurement was performed by sweeping potentials across the cell using a scan rate of 2 mV/sec with the applied potentials from 0-0.5 V with respect to the OCV. The AC impedance measurement was obtained in a frequency range of 1 MHz to 0.1 Hz using potential amplitude of 50 mV under open-circuit conditions. In some cases, the polarisation under applied potential was also investigated.

For C2 hydrocarbon synthesis, humidified CH_4 was fed into the anode, during electrolysis, the gaseous products in the anode outlet stream were examined by a mass spectrometer (MS) (Prolab, Thermo Scientific) and GC-8A gas chromatograph (Shimadzu) equipped with a TCD, using a Poropak Q and Molecular Sieve 5A column at every 15 min.

III. RESULTS AND DISCUSSION

A. Catalytic Performance of Catalysts in Fixed Bed Reactor

The results of reactant conversions and product selectivities of LSCM and Ce/LSCM catalysts for OCM are shown in Fig. 1. This compares the catalytic performances of catalysts between 700-900°C. The WHSV and molar ratio of $\text{CH}_4\text{:O}_2\text{:N}_2$ were kept at 10000 ml $\text{g}^{-1}\text{h}^{-1}$ and 4:1:5, respectively. The results show LSCM catalyst was more selective to partial oxidation than the oxidative coupling of methane reaction, as the selectivity to CO was much higher than that to CO_2 . Approximately 54.5% CO selectivity and 43.2% CO_2 selectivity at 43.2% CH_4 conversion with LSCM successfully obtained at 850 °C. For Ce/LSCM catalyst, it was more selective to oxidative coupling of methane reaction, as the selectivity to C2 was much higher than that to CO and CO_2 . Approximately 54.5% CO selectivity and 43.2% CO_2 selectivity at 43.2% CH_4 conversion with LSCM successfully obtained at 850 °C. In an analogous manner, Ce promoter addition increased methane conversion and C2 hydrocarbon selectivity. The C_2^+ selectivity increased when Ce was present in the catalyst. The highest C_2^+ (55.3%) selectivity were measured at 800 °C.

The effect of temperature on products is presented in Fig. 2. C_2^+ selectivity increased with temperature and passed through a maximum at 800 °C. For undesirable products, CO_2 selectivity decreased with Ce addition. The 5 wt% Ce catalyst demonstrated the lowest CO_2 selectivity at 750 °C. The yield of C2 hydrocarbon selective products as a function

of temperature can be seen in Fig. 2. C2 hydrocarbon yield increased and passed through a maximum value at 800 °C. Consequently, the addition of Ce promoter could promote the performance of the catalysts for the OCM reaction, and inhibited the gas phase oxidation of methane.

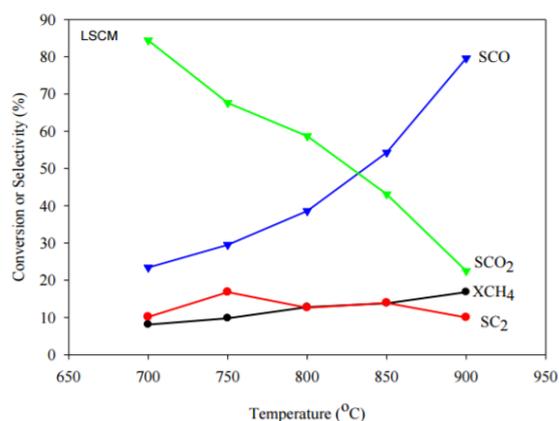


Fig. 1. The catalytic performance of LSCM catalyst as related to operation temperatures at WHSV of 10000 mlg⁻¹h⁻¹ and CH₄/O₂ of 4.

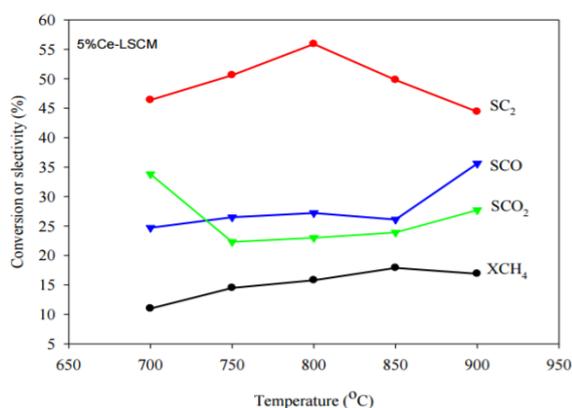


Fig. 2. The catalytic performance of Ce/LSCM catalyst as related to operation temperatures at WHSV of 10000 mlg⁻¹h⁻¹ and CH₄/O₂ of 4.

B. Characterization of SOFC Single Cell

Phase identification of perovskite electrodes prepared from metal nitrates was determined by X-ray diffraction (XRD). Fig. 3 displays the XRD patterns from the surfaces of two electrode composites comprising of LSCM and Ce-LSCM addition into electrodes. For comparison, an XRD pattern of the YSZ porous layer is also included. After the sintered at 1200 °C, XRD peaks of LSCM were showed in Fig. 3. For CeO₂ doping, the CeO₂ peaks can be detected at 2θ as 28.6°. These data indicated that the addition of Ce promoter was not reacted with LSCM phase.

C. Microstructure of Impregnated Electrodes

Firstly, the firing conditions for the impregnation of the LSCM electrode were investigated. According to the literatures [18], [19], the thermal decomposition of nitrate solution in air begins at 300 °C. With an intention of prevent any thermal shock that may happen in this fairly strong ceramic membrane after repeated impregnation, the decomposition temperature of the impregnated nitrate was set at 350 °C with dwell time of 30 min. However after several impregnation cycles, the impregnated samples were completely disintegrated and delaminated. The SEM images in Fig. 4 show a significant change in the microstructure of

porous layers, the SEM images of the structures LSCM indicates that the LSCM forms a uniform coating over the surfaces of the YSZ (Fig. 4a). For CeO₂ loading, the results show the presence of nanocrystalline CeO₂ (Fig. 4b). The estimated particle diameters for CeO₂ were 5-21 nm and uniform coating over the surfaces of the LSCM/YSZ.

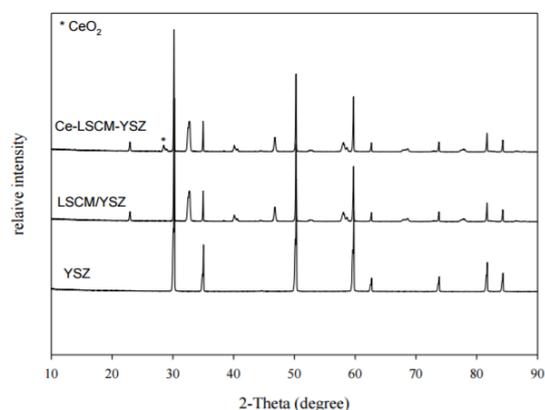


Fig. 3. The XRD pattern of (a) YSZ, (b) LSCM/YSZ and (c) Ce-LSCM/YSZ.

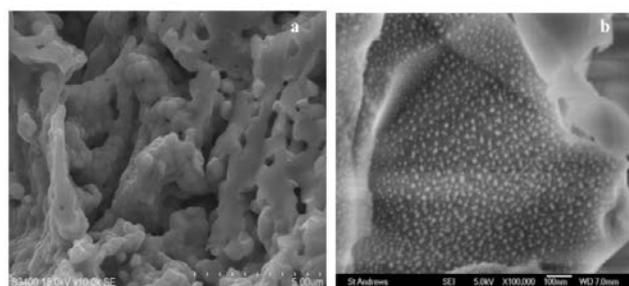


Fig. 4. SEM images of fracture-cross section of YSZ membrane with an impregnated porous layer. (a) porous membrane with LSCM impregnated layer, (b) porous membrane with Ce/LSCM impregnated layer.

D. Electrochemical Performances

Fig. 5 shows voltage, current, and power density properties for tape-cast SOFC cells fabricated by impregnation of LSCM with CeO₂ and without CeO₂ with Ag current collectors. The measurements of these cells at 800 °C are shown in Fig. 5. Their open circuit voltage (OCV) values were ~1.02-1.08 V, which were very close to the theoretical value calculated from the Nernst equation. Such high OCV values indicate that a dense structure is well-formed in the YSZ electrolyte and leakage of the fuel gas is negligible for each sample. As seen in Fig. 6, the power density using a LSCM anode catalyst was 123 mW cm⁻² at 800 °C. When 5 wt% of CeO₂ was impregnated together with LSCM, the power density was 298 mWcm⁻². This is because the added ceria enhanced the conductivity, as well as extended effective active areas. As shown in Fig. 6, the cell had a series resistance, R_s , of 0.65 Ω.cm² and a total resistance, R_T , of 1.05 Ω.cm² at 750 °C. As the temperature increased to 850 °C, the series resistance of the cell decreased to 0.37 Ω.cm² and the total resistances decreased to 0.65 Ω.cm². All the impedance spectra captured at OCV with varying temperatures demonstrated that the resistance of the cell was greatly dominated by the polarization resistance. The power density, with respect to the temperature, for the SOFC systems studied is shown in Fig. 7. In SOFC systems studied, there is an increase in the maximum power with increasing

temperature. The addition of ceria catalysts improve performance across the entire temperature range studied. The highest power density achieved in this study were for the cell containing 5wt% CeO₂ with 356 mW.cm⁻² at 850 °C in humidified H₂. These high powder densities are achieved because ceria is the most active materials for effective catalysis of the different processes in fuel oxidation.

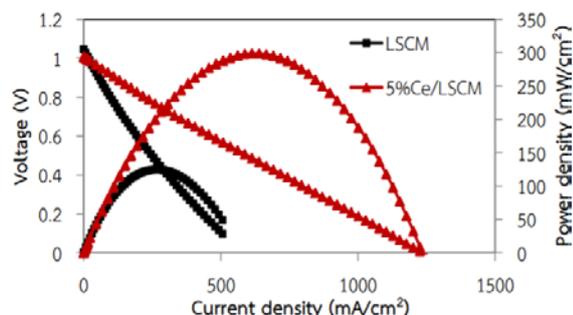


Fig. 5. Performance curves of the 8YSZ cell with 40 wt% LSCM(a) and 5%Ce/LSCM (b) in wet humidified H₂ at 800 °C.

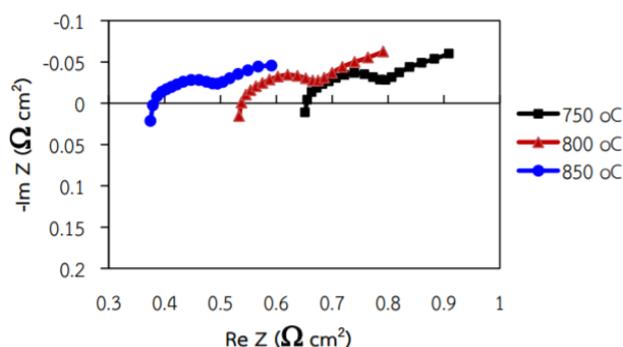


Fig. 6. Impedance spectra of the cell with 5%ceria-40wt% LSCM as anode and 40 wt% LSF as cathode in humidified H₂ at different temperatures.

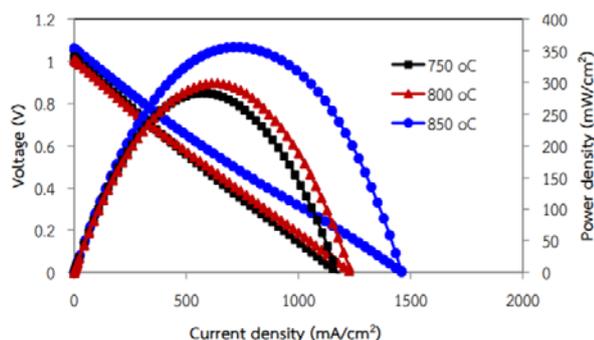


Fig. 7. IV and performance curves of the cell with 5%ceria-40wt% LSCM as anode and 40 wt% LSF as cathode in humidified H₂ at different temperatures.

E. SOFC with 5%CeO₂/LSCM Anode as Cogeneration of Chemical and Electricity

The 8YSZ cell with the 200 μm porous scaffolds were infiltrated with 40 wt% LSF and 5%CeO₂-40 wt% LSCM was used to investigate the performance of the 5%CeO₂-40 wt% LSCM anode as a cogeneration device. The cell was tested in humidified CH₄ following at the rate of 10ml/min at 750-850 °C. Impedance spectra of the cell presented in Fig. 8 shows that the maximum power with increasing temperature. The cell had a low resistance and also the highest maximum power density of 221 mW cm² at 850 °C, the best performance was also obtained at 850 °C in humidified CH₄ (Fig. 9). As shown in Table I, the methane conversion was 3.1% after applying 0.3 V to the cell for 30 minutes. At the

highest methane conversion rate, the cell achieved a good C2 hydrocarbon selectivity of 78.4% and a C2 yield of 3.3% at 850 °C.

TABLE I: THE ARRANGEMENT OF CHANNELS

Temperature (°C)	X _{CH₄} (%)	S _{C₂} (%)	S _{CO₂} (%)	Y _{C₂} (%)
800	3.1	69.8	25.8	2.6
900	3.4	78.4	16.7	3.3

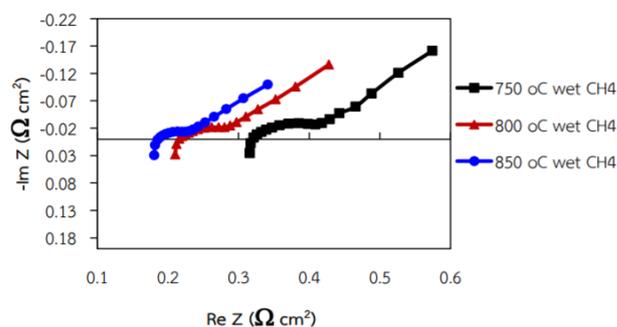


Fig. 8. Impedance spectra of the cell with 5%ceria-40wt% LSCM as anode and 40 wt% LSF as cathode in humidified CH₄ at different temperatures.

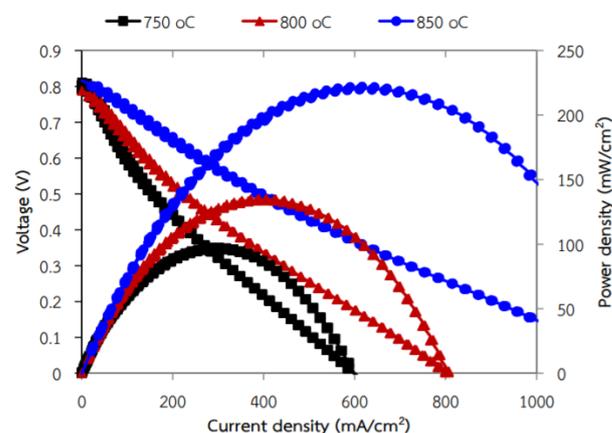


Fig. 9. IV and performance curves of the cell with 5%ceria-40wt% LSCM as anode and 40 wt% LSF as cathode in humidified CH₄ at different temperatures.

IV. CONCLUSION

The catalytic activity for oxidative coupling of methane (OCM) was measured in a fixed bed reactor (FBR) and solid oxide fuel cell reactor (SOFC). Approximately 54.5% CO selectivity and 43.2% CO₂ selectivity at 43.2% CH₄ conversion with LSCM successfully obtained at 850 °C for Ce/LSCM catalyst. The addition of Ce promoter could promote the performance of the catalysts for the OCM reaction, and inhibited the gas phase oxidation of methane. The improvement of cell performance was carried out by decreasing the thickness of the electrolyte and using a high catalytic activity electrode. The tape casting method was employed in order to reduce the electrolyte thickness and prepare porous ceramic support for the electrode material. The electrode materials were introduced into porous YSZ support by ion impregnation technique and a composite electrode/electrolyte was achieved. The impregnated LSCM was synthesized at temperatures over 700 °C and did not react with 8YSZ at 1200 °C. Tape-cast SOFCs with impregnated LSCM exhibited excellent performance when CeO₂ was used as catalysts. A SOFC with a composite anode 5%CeO₂ - 40

wt% LSCM exhibited maximum power density 221 mW cm^{-2} at 850°C in humidified methane. Under a load of 0.3V , 3.4% CH_4 conversion and 78.4% C_2 selectivity were obtained.

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