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Systhesis $La_{0.6}Sr_{0.4}Co_{0.2}Fe_{0.8}O_{3-\delta}$ Perovskite by Microwave Assisted Sol – Gel Method and Properties of LSCF/GDC Composite Cathode for IT-SOFCs

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Abstract—

LSCF 6428 was synthesized by microwave assisted sol – gel method in order to improve the procedure. The single-phase, nanocrystalline structure, specific area were determined by XRD, SEM and BET analyses. Composite of LSCF 6428 and GDC at different ratio were used to find suitable cathode for IT-SOFCs. Properties of this cathode including thermal expansion coefficient, porosity, CH_4 conversion and resistance were studied. The results showed that well-crystalline perovskite was obtained by microwaved at 500 W in 10 minutes, stirred at 90 ^oC in 2 hours, and calcined at 900 ^oC in 1 hours. The surface area of the material was 13.4 m^2/g. The properties of materials were also improved by mixing with GDC at the ratio 7:3 (w/w). At this ratio, thermal expansion coefficient of composite matched with LAMOX electrolyte, porosity of cathode reached 27.7 %. CH_4 conversion of cathode was 20.4 – 24.8 % at air mixed ratio 2:1 and in the range of temperature from 600 – 800 ^oC. The resistance of cathode was 0.17 – 0.2 Ω in the range of temperature from 580 – 700 ^oC. Microwave assisted sol – gel is a time – saving and energy - efficient technique to synthesis LSCF 6428 and composite of LSCF 6428 and GDC is suitable cathode material for IT-SOFCs.

Keywords—IT-SOFCs, LSCF 6428, cathode, perovskite, microwave, sol – gel, GDC.

I. INTRODUCTION

Solid oxide fuel cell (SOFC) is a ceramic device that converts the chemical energy of a fuel gas and an oxidant gas directly to electrical energy without combustion as an intermediate step [1]. The working principle is based on the difference in catalytic activity of the electrodes for the respective anode and cathode reactions. SOFCs are considered as a next generation power generating technology because of their fuel flexibility and high overall efficiency [2]. However, SOFCs operate at high temperature (above 1000 ^oC) impose several challenges on reliability and long-term stability of these fuel cells [2]. So that, reducing their operating temperature is very important to commercialize SOFCs. Intermediate Temperature - Solid Oxide Fuel Cell (IT-SOFCs) operate at temperature from 600 to 800 ^oC in order to lower the cost and to extend the lifetime [3]. However, decreasing the operating temperature makes cell performance decrease due to less catalytic activity of the electrodes and poor conductive of the electrolyte. Many recent studies have focused on improving the electrochemical performance by searching new materials for anode, cathode and electrolyte [2], [3].

Mixed ionic–electronic conductor $La_{0.6}Sr_{0.4}Co_{0.2}Fe_{0.8}O_{3-\delta}$ (LSCF 6428) perovskite is an attractive material as a cathode for IT-SOFCs because it has high catalytic activity as well as good ionic and electronic conductivities at intermediate temperature [2], [3]. LSCF 6428 was synthesized by different methods such as solid state reaction, co-precipitation, combustion, hydrothermal, plasma spray decomposition... [3], [4], [5]. These methods generally require long reaction time and high calcined temperature (above 1000 ^oC). In our previous works [6], LSCF 6428 was synthesized by sol - gel method, which was proved as a simple and economical method for making nano powders [7], with lower calcined temperature (900 ^oC in 1 hour) [6]. In recent studies, microwave synthesis is considered as a technology for high-purity, time-saving and energy-efficient [4], [8]. The problem with LSCF 6428 is its thermal expansion coefficient (TEC) higher than LAMOX electrolyte [9]. TEC mismatches between components can lead to failure of the seals that separate anode, electrolyte, and cathode chambers [2], [10]. The addition of Gadolinium doped ceria (GDC) can reduce the total TEC and yet maintain the high ionic conductivity [2], [10]. Because GDC is one of a class of ceria – doped electrolytes with higher ionic conductivity and lower operating temperatures (<700 ^oC) compared to another electrolyte like YSZ [2], [11]. The electrolyte application of LAMOX in SOFC appears very promising, it denotes the oxide ion conductor family based on its parent crystal $La_2Mo_2O_9$ [9]. This material exhibit high ion conductivity. Their conductivities are comparable with or slightly higher than those of the well-known oxide ion conductors of lanthanum strontium gallium magnesium oxide and GDC at 600 - 800 ^oC [9]. Besides that, the cathode must has sufficient porosity to allow gas transport to the reaction sites, low CH_4 conversion and sufficient electronic conductivity to support electron flow in the oxidizing environment at the operating temperature. In general, maximum possible cathode conductivity is desirable to minimize resistance. Cathode must has low CH_4 conversion because in the SC-SOFC, the reaction include [1], [2]:

Anode: $CH_4 + ½O_2 \rightarrow 2H_2 + CO$ (1)

$H_2 + O_2^- \rightarrow H_2O + 2e^-$ (2)

Cathode: $O_2 + 4e^- \rightarrow 2O_2^-$ (3)

Completely oxidized: $CH_4 + 2O_2 \rightarrow 2H_2O + CO_2$ (4)

In particular, the conversion of CH_4 occurs on the anode according to the reaction (1), create the desired product is H_2 (direct fuel power generation). Reaction (4) is the side effects, should be limited because of CH_4 consumption, completely oxidized to CO_2. Therefore, a mixture of cathode materials need low conversion CH_4 to CO_2.

In this paper, to improve synthesis process, nano-crystalline LSCF 6428 was synthesized by microwave assisted sol - gel method, which was not found in publication about LSCF synthesis at the ratio 6428. This research also focused on mixing LSCF 6428 with GDC at difference ratios to match TEC with LAMOX electrolyte and investigated the conditions that cathode has high porosity, low CH_4 conversion and low resistance.

II. MATERIAL AND METHODOLOGY

A. Material Synthesis Process

The procedure to synthesize LSCF 6428 by sol-gel method was described in our previous work [6] using $La(NO_3)_3.6H_2O$ (99.9%), $Sr(NO_3)_2$ (99.0%), $Co(NO_3)_3.9H_2O$ (98.5%), $Fe(NO_3)_3.9H_2O$ (98.5%), EDTA (98.0%), ethylene glycol (98.0%), ammonia solution (30%) and distilled water. The precursor solution was prepared by mixing individual nitrate salt solution in a molar ratio of 0.6:0.4:0.2:0.8 respectively for LSCF. EDTA was dissolved in NH_4OH solution at the molar ratio of 1:1. This solution was added to nitrate solution in order to form complexes at defined the molar ratio of EDTA/NO_3^- of 1.5:1. Ethylene glycol which was used to create dispersive environment was slowly added into the reaction mixture and the pH 8.0 of mixture was maintained by using ammonia. Then the mixture was heat and continuously stirred at 90 ^oC until a dark brown gel was formed. Gels were dried at 200 °C for 2 h, following by calcination at high temperatures from 700 - 1300 °C. After that, a ball mill in alcohol was occurred. Sample was dried at 105 ° C for 2 h.

The difference here is the short time (5 minutes) microwave application during sol-gel ageing at 90 ^oC which lasts next 60 minutes. Limited by the microwave oven in use, the microwave irradiation was either at 300W, 500W, or 700W. This step was repeated up to total 3 times.

A modified domestic microwave oven (Sanyo 700, China) was used. The frequency of the microwave irradiation was 2.45 GHz, and the maximum output power was 700 W. The modified microwave oven is shown in Fig.1.

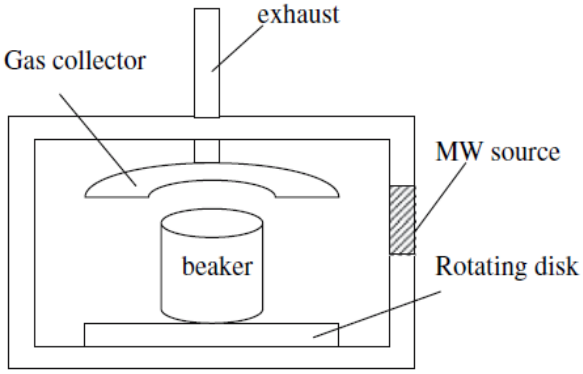

Fig. 1 The modified household microwave oven (vessel size: 16 L, microwave power: 700 W, frequency: 2.45 GHz)

B. Characterization of LSCF 6428

1) Characterization of Synthesized Materials

The synthesized materials were characterized by XRD, SEM and N_2-BET methods, using a D8 advance Bruker equipped with a CuK_α source and scanning angle in region $2\theta = 20 \div 80^o$, a JEOL-JSM-7401F, and a BET NOVA 2200e, respectively.

2) Preparation and Characterization of Cathode Materials

GDC was synthesized according to the sol-gel method described elsewhere [12]. The composites serving as cathode materials were prepared by ball milling the as-prepared LSCF 6428 with GDC at different ratios of 10:0; 8:2; 7:3 and 6:4 (w/w) in ethanol for 10 minutes.

The TEC of cathode materials in form of 5 mm x 20 mm cylindrical pellets (compressed at 7 MPa) were measured by a Netzsch Dil PC 402 in the range from room temperature up to 1000 ^oC at 10 ^oC/minutes heating rate and under open air.

The porosity, CH_4 conversion and electrical resistances of cathode materials were measured in form of 15 mm x 1 mm pellets (compressed at 7 MPa) after 60 minutes calcining at 1000 ^oC in open air. The porosities were determined by difference between weight in water and in air according to Archimedes method.

The CH_4 conversion was studied at the different conditions of mixing ratio gas of CH_4:O_2 (R_{mix} = 1, 1.5, 2) and reaction temperature ranging from 500 - 800 ^oC. Reactor products were characterized using GC-2014 ATF.

The electrical resistances of cathode materials were measured in a home-made apparatus as described in Fig.2. The air mixing ration CH_4/O_2 (R_{mix}) was kept constant at 2 and the temperature was ranged from $400^oC \div 700^oC$. The electrical resistances of cathode were measured by a Wellink HL-1240 multimeter.

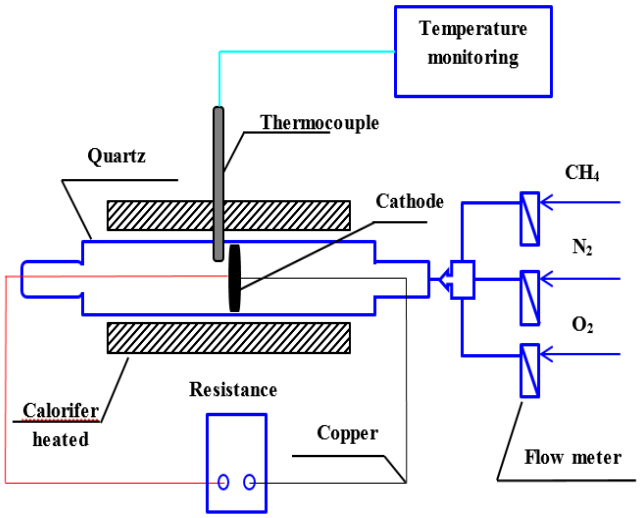

Fig. 2 Schematic diagram of resistance measurement configuration.

III. RESULT AND DISCUSSION

A. Effects of Power and Time of Microwave Irradiation

The microwave irradiation made the temperature of the sol sample raised immediately. Within 1 minute of irradiation, the sol started boiling and then converted into a black solution. After every 5 minutes of irradiation, stirring was applied to maintain the temperature of solution at 90 ^{o}C. Low heating supply to maintain because of the solution was boiled when irradiated.

The XRD pattern of LSCF 6428 cathode material synthesized at difference microwave powers (300, 500 and 700 W) is presented in Fig.3. The samples were microwaved for 5 minutes and stirred at 90 ^{o}C for 1 hours, this step was repeated up to total 3 times for every microwave power. The XRD results showed that at microwave power of 300 W, the perovskite structure was not clear because energy is not enough for reaction to occur. At 500 and 700 W, the perovskite structure became clearer, matched with standard peaks in both location and intensity. The difference between 500 W and 700 W was not clear because of sufficient energy supplied by both irradiations for the reaction. So the appropriate microwave power is 500 W. At this power, microwave assisted sol – gel method provided good condition that helps complexion reaction to occur completely. Compare with our previous work [6], the perovskite structure was obtained by stirring at 90 ^{o}C in 4 hours. In this experiment using microwave assisted sol – gel method, total time of microwave irradiation and stirring was 3 hours and 15 minutes. Total time was studied in next experiment. After sol – gel, both samples were calcined at 900 ^{o}C for 1 hour. In another study, LSCF was prepared by the Pechini method in which temperature of calcination was 1000 °C for 4 h to develop crystalline perovskite phase [3]. So both LSCF were synthesized by sol – gel method and microwave assisted sol – gel which reduced significantly the temperature and time of calcination.

The effect of difference microwave irradiation times on synthesis of LSCF 6428 cathode material is presented by XRD pattern in Fig.4. The microwave power was selected from the previous experiment at 500 W. The results showed that for one time of irradiation, the perovskite structure was not clear because energy is not enough for the reaction to occur. Two and three times, the difference was not much. In these cases, location and intensity of peaks matched with standard peaks. Because of sufficient energy for the reaction, providing higher energy does not make significant difference. Hence, the appropriate time in total for microwave irradiation is 10 minutes at the power of 500 W. At this condition, LSCF 6428appeared in single phase with clear perovskite structure.

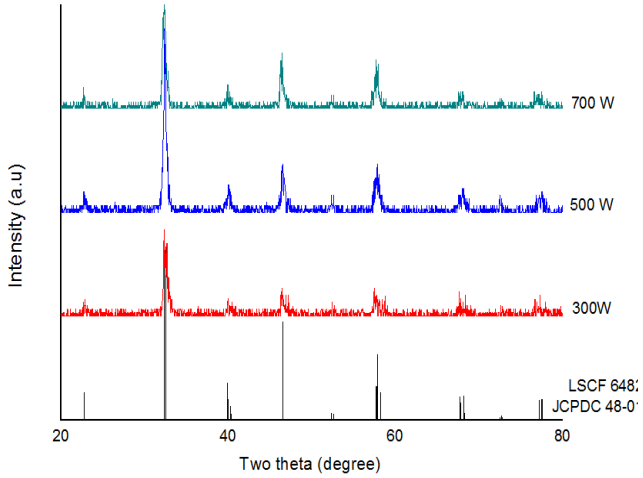

Fig. 3 XRD of LSCF 6428 synthesized at difference microwave powers

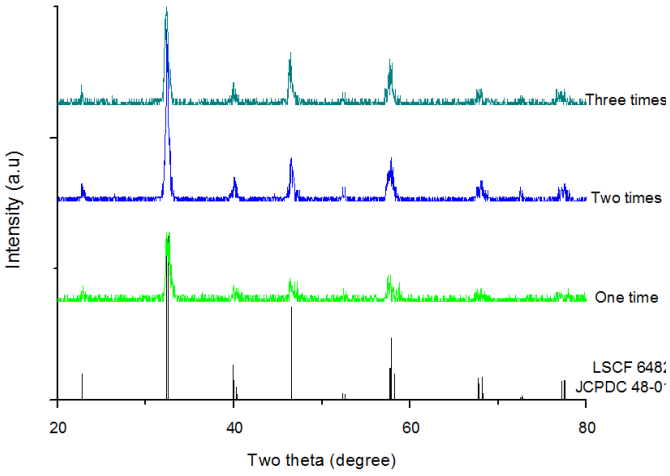

Fig. 4 XRD of LSCF 6428 synthesized at difference microwave times (with the same microwave power of 500 W).

The comparison of XRD results between in this paper with LSCF synthesized only by sol – gel [6] is presented in Fig.5. The traditional sol – gel synthesis was conducted by stirring in 4 hours without microwave irradiating, while the microwave assisted sol – gel (MW sol – gel) synthesis was performed at 500 W in 10 minutes and stirring in 2 hours. Other conditions for preparation of two samples were kept the same. Both location and intensity of peaks in two samples were suitable with standard peak, but in the case of microwave assisted sol – gel synthesis, the peaks were much clearer, less interference, hence the material has better quality.

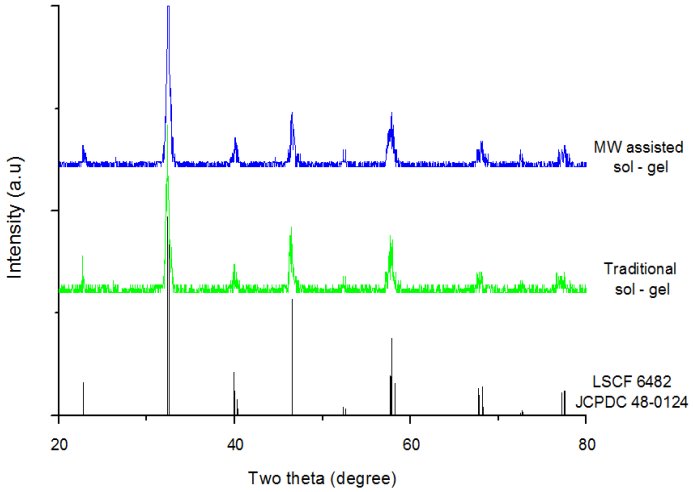

Fig.5 Comparison of XRD results of LSCF 6428 synthesized by traditional sol – gel and microwave assisted sol – gel methods.

In another study, LSCF 6428 was synthesized by sol – gel method that required stirring at 90 °C for 4 hours and after the sol-gel, calcination at 900 °C for 3 hours [7]. With LSCF synthesis processes at another ratio is 8255, material was also synthesized by sol - gel combined with microwave which required stirring at 90 ^{o}C for 3 hours and microwave power of 700 W for 35 minutes [4]. In this study, the synthesis conditions were microwave power of 500 W for 10 minutes, stirred at 90 °C for 2 hours and calcined at 900 °C for 1 hours. The research results showed that well perovskite structure was obtained with the stirred time, calcined time, microwave power, microwave time were decreased. Because microwave assisted sol – gel help complexion reactions were effective, the consumption of energy decreased significantly and time – saving.

The crystallite size of LSCF 6428 synthesized at the intensity of microwave of 500 W for 10 minutes was determined by SEM as shown in Fig.6. The average particle size was in the range of 90 - 120 nm. In other studies, M. Ghouse et al [7] and Changjing Fu et al [13] reported that materials synthesized by sol - gel method also had nanosize in the range of 50 - 200 nm, with the surface area of 2.8 m^2/g. The synthetic material by sol - gel combined with microwave method obtained the smaller sizes compared to the one synthesized by the traditional sol – gel method, which could be due to good contact between the reaction and position O_2. BET results further showed that surface area of material was 13.384 m^2/g. Hence, comparing to the traditional sol–gel routes [7], the microwave sol – gel materials show smaller crystallite size and larger surface areas.

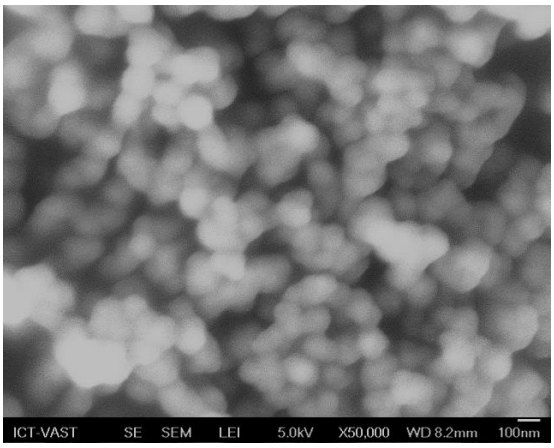

Fig. 6 SEM result of LSCF 6428 microwaved at 500 W in 10 minutes.

B. Effect of the Mixed Ratio on TEC and Porosity

LSCF was mixed with GDC at difference ratios of 10:0, 8:2, 7:3 and 6:4 to match TEC with LAMOX. TEC results of these sample at 800 ^{o}C are presented in table I and porosity of these are presented in table II.

At the ratio of 8:2, TEC of the composite was slightly higher than that of electrolyte α-$La_2Mo_2O_9$ (LAMOX). At the ratios of 7:3 and 6:4, TEC of the composites was matched with electrolyte. The reason is LSCF 6428 has TEC higher than electrolyte, on the other hand, TEC of GDC is lower than that of LSCF 6428 and LAMOX, according to table I. So mixing

with GDC can make TEC of the composite reduce and match with LAMOX. Matching with electrolyte will prevent cracking between cathode and electrolyte. Three ratios of 8:2, 7:3 and 6:4 were used to study in next experiments to find out the suitable condition.

GDC make porosity of composite increased, high porosity will extend gas transport to the reaction sites. Based on the results of porosity, at the ratio of 6:4 is more advantageous than the ratio of 7: 3 and 8:2, more suitable porosity to the IT-SOFCs applications. However, this difference only about 2 – 3 %. Compared to the porosity of the composite cathode materials LSCF-SDC with porosity in the range of 19.62 to 24.92% [14], all three composite at the ratio of 8:2, 7:3 and 6:4 contains higher porosity.

Table I TEC of materials.

Material	**TEC (10^{-6}/°C)**
LSCF	15.5
LSCF:GDC = 8:2	15.1
LSCF:GDC = 7:3	14.8
LSCF:GDC = 6:4	14.5
GDC	12.3 [15]
α-$La_2Mo_2O_9$ (electrolyte)	14 – 15 [9]

TABLE III Porosity of materials.

Material	**Porosity (%)**
LSCF	23.67 ± 0.25
LSCF:GDC = 8:2	26.16 ± 0.31
LSCF:GDC = 7:3	27.68 ± 0.22
LSCF:GDC = 6:4	29.16 ± 0.39

C. Effect of the Mixed Ratio on the CH4 Conversion

R_{mix} were selected from the complete oxidation reaction (4) with R_{mix} = 0,5 and reaction desired product (1) with R_{mix} = 2. A lower limit of the mixing ratio (R_{mix} < 0,85) is imposed by the gas mixture becoming explosive. Carbon deposition has a negative impact on the performance and long-term stability of the system, and $R_{mix} \leq 2$ is recommended to avoid carbon formation. CH_4 conversion of cathode at the difference R_{mix} and the difference composite were showed in Fig. 7 and Fig. 8.

The results showed that, high operating temperature will consume more fuel, in the range of 600 – 800 °C, stable conversions were observed. The lowest conversion is at the R_{mix} = 2 in range of 20.4 to 24.8%. CH_4 conversion of composites at difference ratio of LSCF 6428 and GDC were not significant. CH_4 conversion of composite at the ratio 7:3 was slightly lower than that of 6:4.

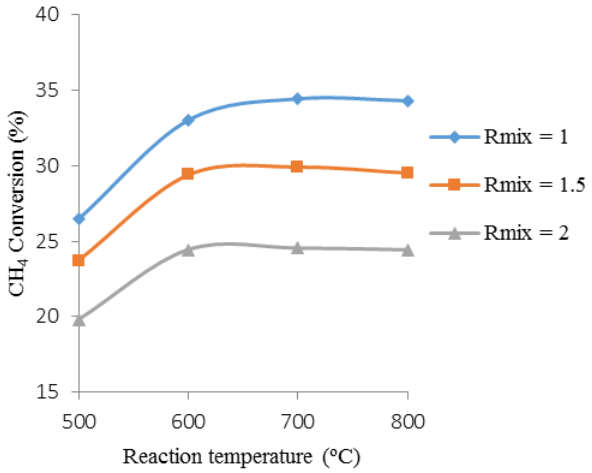

Fig. 7 SEM result of LSCF 6428 microwaved at 500 W in 10 minutes.

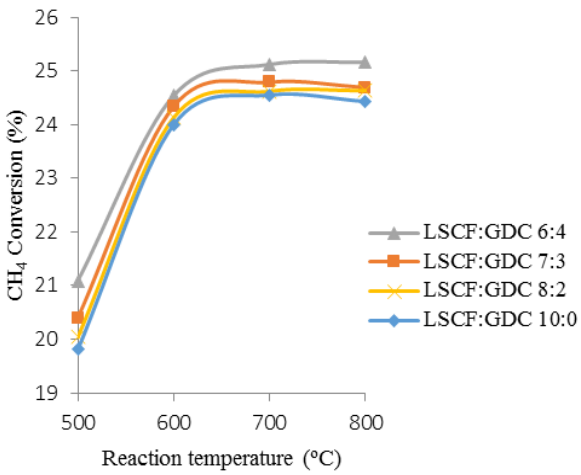

Fig. 8 SEM result of LSCF 6428 microwaved at 500 W in 10 minutes.

Compare with another materials was presented in Table III, results showed that the composite of LSCF 6428 and GDC has lower CH_4 conversion so that this composite can save fuels.

Table IIIII CH_4 conversion of materials.

Material	**Temperature (°C)**	**Conversion (%)**
70% LSCF + 30% GDC	600 – 800	20.4 – 24.8
$La_{0.08}Sr_{0.92}Fe_{0.20}Ti_{0.80}O_{3-\delta}$	600 – 800	26 – 30 [16]

70% SSC + 30 % SDC	600	24 – 33 [17]
30% BSCF + 70% SDC	700	22 – 29 [10]

D. Effect of the Mixed Ratio on the Resistance

Fig. 9 shows the results of resistance of composite material of LSCF and GDC at the ratios of 8:2, 7:3 and 6:4 with R_{mix} = 2, reaction temperature from 450 – 700 ^oC. Sample LSCF 6428, which was not mixed with GDC (10:0), was used to verify.

Results showed that when the operating temperature increased, the resistance of cathode was decreased. From 550 ^oC to 700 ^oC, the resistance was higher because of poor conductivity of cathode materials at low temperature. From 550 ^oC, the resistance of cathode materials was lower. So the suitable temperature for SC-SOFC operation is 550 - 700 ^oC. According to another studies, the temperature is 600 – 800 ^oC [5, 11]. Thus, this study has decreased operating temperature, which potentially lowers the cost while extends the lifetime of SC-SOFC. In another research, the resistance of LSCF/GDC was 0.18 Ω at 800 ^oC [5], or the lowest polarization resistance of 0.17 Ω cm^2 was achieved from LSCF–GDC (40:60 wt%) composite cathode prepared at 600 °C [11]. In this study, at the mix ratio of 7:3, the resistance of cathode is 0.17 – 0.2 Ω in range temperature 580 – 700 ^oC. The higher proportion of GDC in the mixture resulted in the higher resistance of cathode. So, the ratio 7:3 was chosen because of matching TEC with electrolyte, low CH_4 conversion and low resistance.

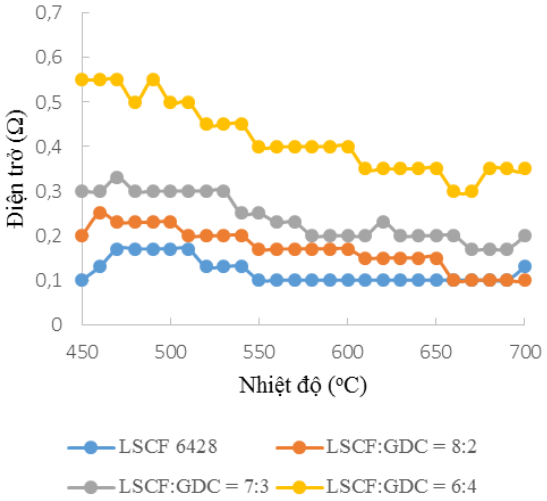

Fig. 9 Resistance of cathode composite made by LSCF and GDC at difference ratios.

IV. CONCLUSIONS

LSCF 6428, with well single-phase and nanocrystalline structure, was successfully synthesized by sol – gel process combined with microwave method. Results in this study showed that the application of microwave assisted sol–gel route for the synthesis of LSCF 6428 is superior in terms of energy and time saving compared to the other methods. LSCF was then mixed with GDC at the ratio of 7:3 and the composite had TEC matched with electrolyte, high porosity, low CH_4 conversion and low resistance in the range of temperature of 550 – 700 ^oC. So it can be concluded that the mixture is suitable material for cathode fabrication in SC-SOFC.

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