

Synthesis and properties of $\text{LaNi}_{1-x}\text{Fe}_x\text{O}_{3-\delta}$ as cathode materials in SOFC

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Abstract The synthesis of $\text{LaNi}_{1-x}\text{Fe}_x\text{O}_{3-\delta}$ (LNF) perovskites with $x = 0.0\text{--}1.0$, for use as cathode materials for an IT-SOFC, was investigated using four combustion methods, Water Citrate (WC), Modified Water Citrate (MWC), Nitric Citrate (NC), and Modified Nitric Citrate (MNC). The structures and homogeneities of the synthesized powders were examined using an XRD, and the particle sizes were examined using an SEM and a particle size analyzer. All four combustion methods gave the single phase perovskites with the same structure. The main difference was shown in a particle size that the smallest to the largest sizes were obtained from MNC, MWC, NC, and WC, respectively. In this LNF series, as x is $0\text{--}0.5$, the crystal structure is cubic and rhombohedral at the calcination temperature of 700 and 900 °C, respectively. Further investigation indicated that the cubic structure changed to rhombohedral structure at 900 °C, and was stable up to 1200 °C. As x is $0.6\text{--}1.0$, the crystal structure is in orthorhombic phase when calcined between 700 and 1000 °C. This orthorhombic phase decomposed above 1100 °C. From the XRD and SEM–EDX results, $\text{LaNi}_{0.6}\text{Fe}_{0.4}\text{O}_{3-\delta}$ (LNF64) has a good chemical compatibility with 8YSZ from room temperature up to 900 °C. In addition, its

thermal expansion coefficient is $13.2 \times 10^{-6} \text{ K}^{-1}$ close to that of 8 mol% Y_2O_3 (8YSZ). Therefore, LNF64 also has a good physical compatibility with 8YSZ.

Introduction

Recently, $\text{LaNi}_{1-x}\text{Fe}_x\text{O}_{3-\delta}$ (LNF, $x = 0.0\text{--}1.0$) perovskites have received considerable interest for industrial applications such as cathodes for solid oxide fuel cells [1–3] and catalyst for hydrogen production from biomass gasification [4], steam reforming [5], and partial oxidation of methane (POM) [6]. Chiba et al. [1] was the first group to investigate LNF ($x = 0.0\text{--}1.0$) and $\text{LaNi}_{1-x}\text{M}_x\text{O}_{3-\delta}$ ($\text{M} = \text{Al, Cr, Mn, Fe, Co, and Ga}$) as cathode materials for IT-SOFC. $\text{LaNi}_{0.6}\text{Fe}_{0.4}\text{O}_{3-\delta}$ (LNF64) exhibits the greatest electronic conductivity over three times greater than the conventional cathode material of $\text{La}_{0.8}\text{Sr}_{0.2}\text{MnO}_3$. Moreover, the thermal expansion coefficient (TEC) of this composition is close to that of the doped zirconia electrolyte [1]. Provendier et al. reported that $\text{LaNi}_{0.3}\text{Fe}_{0.7}\text{O}_{3-\delta}$ (LNF37) was the most active for CH_4 steam reforming [5] and POM [6]. It limits the coke formation with a good aging behavior. It also allows a regeneration of the starting structure. Kharton et al. [7] showed that the oxygen permeation through LNF ($x = 0.0\text{--}1.0$) membranes tended to increase with nickel content due to an increase of oxygen vacancy concentration and bulk ionic conductivity.

The synthesis methods of LNF64 perovskite were investigated and reported by several groups [2, 3, 10, 15]. Various synthesis methods of LNF ($x = 0.0\text{--}1.0$) have been investigated in both conventional solid state method [1, 8, 9] and wet chemical method via citrate using ethanol [3], nitric acid [9], or water [10, 11] as a solvent. From the previous works, the modified citrate method for the synthesis of

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LaFeO₃-based perovskites, which involves the complex formation of metal ions with citric acid and NH₃ and the spontaneous combustion have been used in this study. The advantage of the modified citrate method over the original citrate method is an achievement of fine, homogeneous, and single phase powder [12, 13]. Similar to the approach, Bontempi et al. [14] synthesized LNF37 by a modified citrate route using metal acetates instead of metal nitrates as starting materials and decomposing the organic substances in mild condition at 400 °C. However, 5 days for drying and 2 days to complete decomposition were required.

Basu et al. [16] synthesized LNF64 by citrate and urea routes, in which the metal nitrates were dissolved and mixed with citric acid or urea, respectively. They found that the powder synthesized by citrate route has smaller average particle size than from the urea method. On the contrary, the electronic conductivity of membrane prepared by urea route is greater [10]. Among three different methods; citrate route using ethanol as a solvent, glycine-nitrate route, and coprecipitate route have been investigated by Bevilacqua et al. [3]. The citrate and coprecipitate routes lead to homogeneous powder while the powder prepared by the coprecipitate route was easily sintered. However, the amount of base, tetramethyl ammonium hydroxide used has to be four times of the stoichiometry value to insure full precipitation for this coprecipitate route. The washing and filtering steps had to be done twice to remove any excess base which took 2 days.

In this article, LNF64 perovskite has been chosen for the study of this synthesis method for an application of cathode material. This perovskite was synthesized using citrate and modified citrate methods using water and nitric acid as solvents and investigated for the phase homogeneity and morphology. The suitable method was chosen for a synthesis of all the compositions of LNF ($x = 0.0$ – 1.0). As a cathode, the chemical compatibility of LNF64 with the commercial electrolyte, zirconium oxide doped with 8 mol% Y₂O₃ (8YSZ) was studied using XRD and SEM–EDS [16, 17] and the physical compatibility was studied by comparing the TEC.

Experimental section

Synthesis of perovskite powders

The synthesis methods proposed in this study are based on a citrate route using nitric acid as a solvent, a citrate route using water as a solvent, a modified citrate route using nitric acid as a solvent, and a modified citrate route using water as a solvent, indicated as NC, WC, MNC, and MWC, respectively. High-purity La(NO₃)₃·6H₂O (99.0%, Fluka), Ni(NO₃)₂·6H₂O (97%, UniLab), and Fe(NO₃)₃·9H₂O (98%, Ajax) were used as starting materials.

Citrate route using nitric acid as a solvent

Stoichiometric amount of La, Ni, and Fe nitrates were dissolved in 10 mL conc. nitric acid. Then citric acid was added to a citric:cationic mole ratio of 2:1. The citrate solution was stirred at room temperature for 3 h, and then evaporated on a hot plate at 200 °C until the mixture auto-ignites. The solid was ground using a mortar and pestle, and followed by calcination at 700–1200 °C for 5 h.

Citrate route using water as a solvent

The WC procedure was carried out following the NC method but using water as a solvent instead of nitric acid.

Modified citrate route using nitric acid as a solvent

The MNC procedure was carried out similar to the NC method. Prior to the evaporation step, NH₃ was added until the pH of the solution was 9 as reported in previous works [12, 13]. The homogeneous solution was agitated for 3–5 h before evaporation.

Modified citrate route using water as a solvent

The MWC procedure was carried out following the MNC method but using water as a solvent instead of nitric acid.

Characterization

All the XRD patterns were performed using Rigaku, Dmax 2002 Ultima⁺ diffractometer with Cu-K α radiation (40 kV, 30 mA). The patterns were collected over a range of 20° and 70° 2 θ and a step size of 0.02° with a hold time of 0.5 s per step. For the phase identification, the JCPDS database and the MDI Jade 6.5 search-match software were used.

Morphological images of specimens were recorded using a JSM-5800 LV scanning electron microscope equipped with an EDX system. The micrographs were recorded at 20 kV.

The particle size measurements were carried out using Master Sizer-S. (Malvern Instrument, England). In the sample preparation, 0.2–1 g LNF powder was dispersed in 20 mL solution of sodium pyrophosphate in DI water, and then sonicated for 4 min.

Chemical compatibility of LNF64 with 8YSZ

The chemical compatibility was investigated in two ways, LNF64–8YSZ powder mixture and LNF64–8YSZ electrode coating.

LNF64–8YSZ powder mixture

LNF64 was mixed with 8YSZ from Aldrich in a 50/50 wt ratio. The resulting mixture was then pressed into a disk of 13 mm before heated to various temperatures between 800 and 1200 °C for 5 h. The heated sample was then analyzed using an XRD for a phase identification.

LNF64–8YSZ electrode coating

The electrolyte pellet of 13 mm was made by uniaxially pressing 3 g of 8YSZ and sintering at 1500 °C for 2 h in air. LNF64 prepared by MNC route was calcined at 1000 °C prior to the dispersion in butyl acetate organic binder to obtain a paste. The LNF64 paste was applied on 8YSZ pellet by screen printing. The LNF64–8YSZ pellet was fired at 1000 °C for 2 h in air. The chemical compatibility was characterized using SEM–EDX.

Physical compatibility

The physical compatibility between LNF64 and 8YSZ was investigated in term of the TEC. The selected LNF64 sample, after calcined at 1000 °C, was made into a sample bar by uniaxially pressing 7 g of powder, and then the bar was sintered at 1200 °C for 2 h in air. TEC behavior was determined using Unitherm Model 1161. The thermal study was started from room temperature up to 1200 °C at a heating rate of 3 °C/min.

Electrical conductivity

Symmetrical two-electrode electrochemical test cells were used to perform preliminary impedance spectroscopy investigations on LNF64 cathodes [3]. LNF64 prepared from four synthesis methods were calcined at 1000 °C and dispersed in an organic binder to obtain a paste. The electrode covered a geometric area of 0.70 cm². Two identical electrodes were applied on each side of a 8YSZ pellet and fired at 1100 °C for 1 h. The measurements were carried out with an applied potential of 10 mV, over the frequency range of 0.05 Hz–10 MHz, at 300–950 °C, using a Solartron 1260 Impedance/Gain-Phase Analyser.

Results and discussion

Synthesis method of LNF64

Various synthesis methods on the preparation of LNF including WC, NC, MWC, and MNC have been investigated using XRD, SEM, and particle size analyzer. The XRD patterns (Fig. 1) show that all LNF64 powders

synthesized from every method contain a single phase of rhombohedral structure similar to LaNiO₃ (JCPDS: 33-0711) after calcination at 1000 °C for 5 h.

Despite the same structure, a difference in the morphology for each synthesis method was observed. From SEM micrographs (Fig. 2), all the powder except from WC method show uniform shape with narrow-sized distribution. Furthermore, the powders synthesized by both modified citrate methods have smaller particle size than that of the unmodified ones. The mean particle sizes of ~0.2, ~0.3, and ~0.4 μm, estimated from SEM micrographs were obtained from the powder prepared by MNC, WMC, and NC, respectively. This is due to the nature of powder synthesis process. The exothermic auto-ignition process is fuel-rich for the modified citrate method, where tiny glowing flints propagate within the mixture until the reaction completes. For the unmodified method, the low ignition temperature from the fuel-lean results in a non-uniform heat distribution which leads to a non-uniform morphology [12, 13].

Although the particle sizes shown in SEM micrographs (Fig. 2) are smaller than a micron, their agglomerated particle sizes are larger as measured by the particle size analyzer (Table 1). The powder dispersion process during a sample preparation could not separate individual particles apart because parts of these particles were joined together during the calcination process. In spite of this, the trend of agglomeration size synthesized by different methods still followed that of individual particles.

Even though the smallest average particle size and the smallest agglomeration size are obtained from MNC, the WC and MWC were chosen in this study for investigating

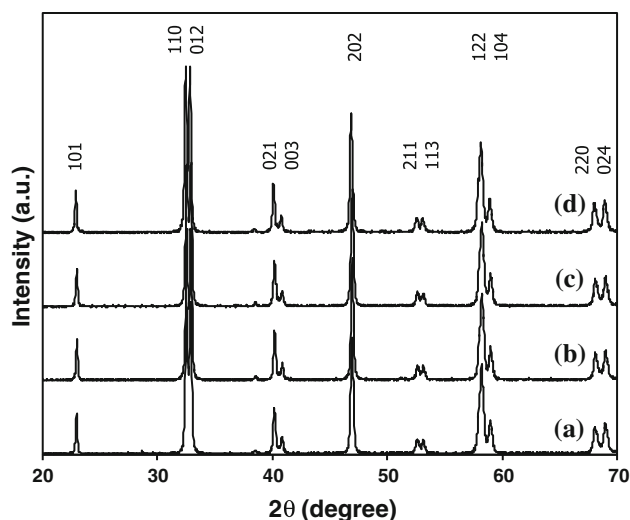
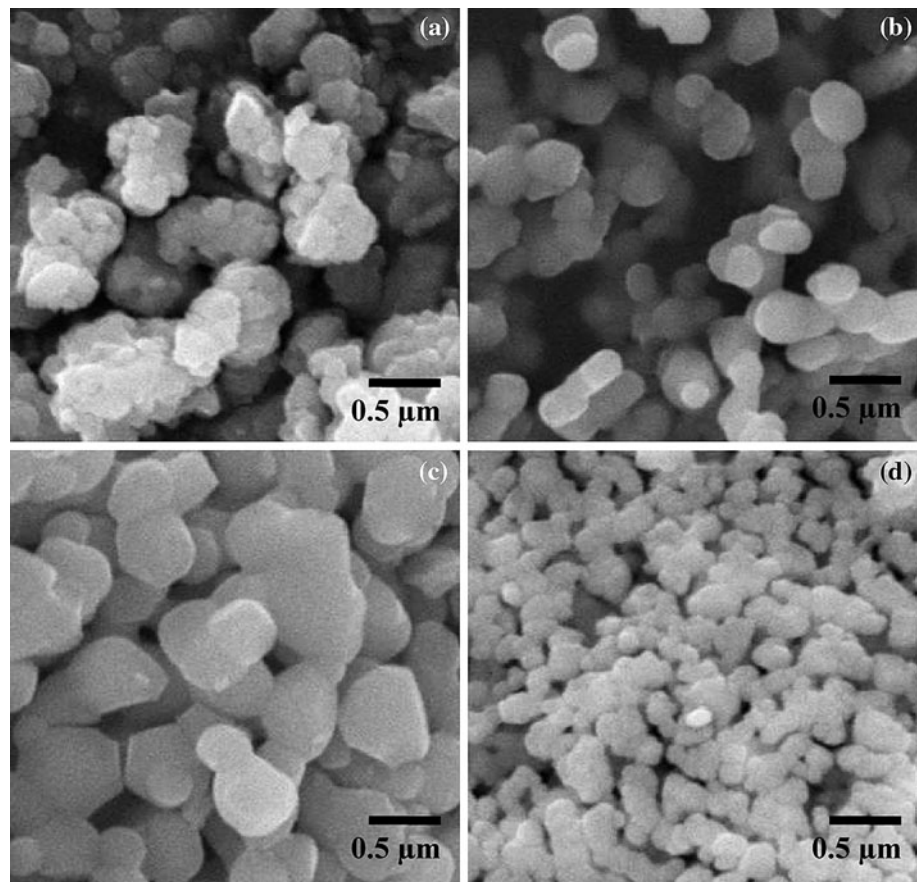


Fig. 1 XRD patterns of LNF64 ($T_{\text{calcination}} = 1000$ °C for 5 h) synthesized by four methods: (a) WC, (b) MWC, (c) NC, and (d) MNC

Fig. 2 SEM micrographs of LNF64 ($T_{\text{calcination}} = 1000\text{ }^{\circ}\text{C}$ for 5 h) synthesized by four methods: (a) WC, (b) MWC, (c) NC, (d) MNC



the synthesis of LNF ($x = 0.0\text{--}1.0$) for the environmental safety and industrial benign.

Synthesis of LNF ($x = 0.0\text{--}1.0$)

The XRD patterns of all LNF perovskites prepared by MWC at $900\text{ }^{\circ}\text{C}$ are shown in Fig. 3. All compositions exhibit a single phase of perovskite structure, which is either orthorhombic or rhombohedral. As $x \geq 0.5$, the compounds have the orthorhombic structure similar to LaFeO_3 (JCPDS: 37-1493), and as $x < 0.4$ the compounds exhibit the rhombohedral structure similar to LaNiO_3 (JCPDS: 33-0711). The previous studies from Chiba and Falcon groups [1, 18] indicated the orthorhombic phase of

Table 1 Particle size-distribution of LNF64 powder synthesized by four methods

Method	Agglomerated particle size (μm)	
	The highest population	The second highest population
WC	4.61	1.71
NC	3.46	1.15
MWC	2.61	0.08
MNC	1.61	–

the composition with high Fe contents, and found the mixed phases of orthorhombic and rhombohedral at $x = 5$ and $x \leq 3$, respectively. Unlike the conventional solid state synthesis that generally uses metal carbonate and metal oxide powder as reactants, the wet combustion method starts with the mixing of metal ion solution at a stoichiometric ratio. Thus, all metallic components can be distributed homogeneously in the solution. Moreover, the citrate anion used in this study acts as a chelating agent of these metal ions. In this study, the self-combustion process releases heat throughout the reaction mixtures to convert the metal citrate complexes to perovskites. The conventional solid state synthesis on the other hand needs high temperature to induce the metal ion in solid lattice of one reactant to diffuse into the lattice of another reactant to form the desired complex. Therefore, this method takes a long time. The purity of the products also depends on the grain size of the starting materials. The starting materials with large grain sizes require higher diffusion ability to be able to be homogeneous. Therefore, desired products which are unstable at high temperature cannot be obtained from this solid state synthesis [1].

As the powder prepared from all the synthesis methods have similar patterns as those obtained from WC methods, they are not presented here.

The lattice parameters and unit cell volume of all x values of LNF perovskites prepared by MWC are presented in Figs. 4 and 5, respectively. The volume of unit cells increases with an increase of x in the LNF system, as Fe ion is larger than Ni ion. The abrupt changes in lattice parameters from $x = 0.4$ – 0.5 is due to a change in the crystal structure. It could be concluded that the modified and unmodified citrate methods do not affect the phase homogeneity and structure system of LNF perovskites, only the morphology and size of particles are influenced.

Effect of calcination temperature on LNF structures

LNF37 and LNF64 were chosen to represent the orthorhombic and rhombohedral systems, respectively. The change of volume in the perovskite phase as a function of calcination temperature, compared with literature results [6, 14, 18] is shown in Fig. 6. It can be seen from this study that the unit cell volume of the samples is not different from those reported in literature, but the phase transformation occurs at lower temperature. Bontempi et al. [14] reported that the structure of LNF37 is cubic between 600

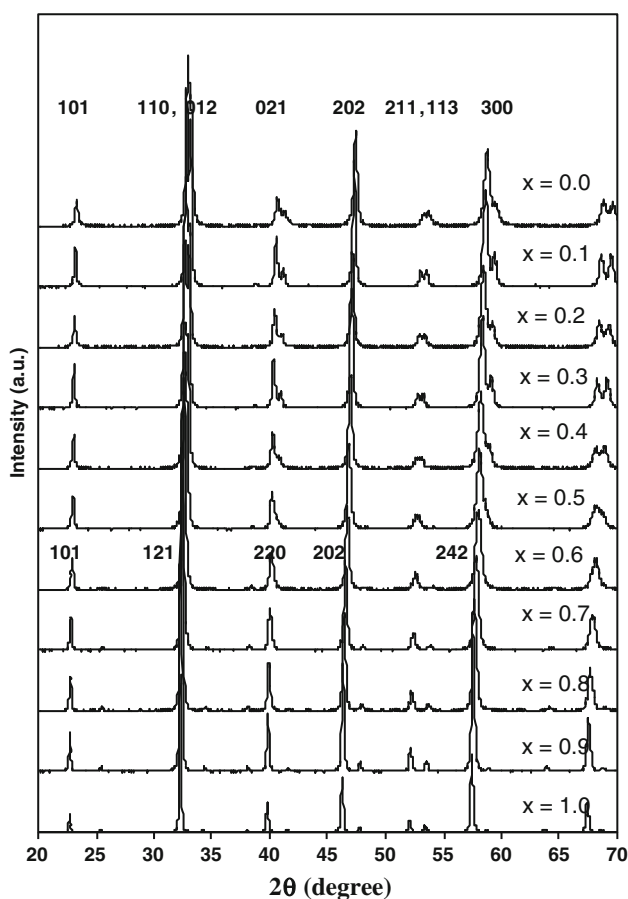


Fig. 3 XRD patterns of LNF ($x = 0.0$ – 1.0) synthesized by MWC method and calcined at $900\text{ }^{\circ}\text{C}$ for 5 h

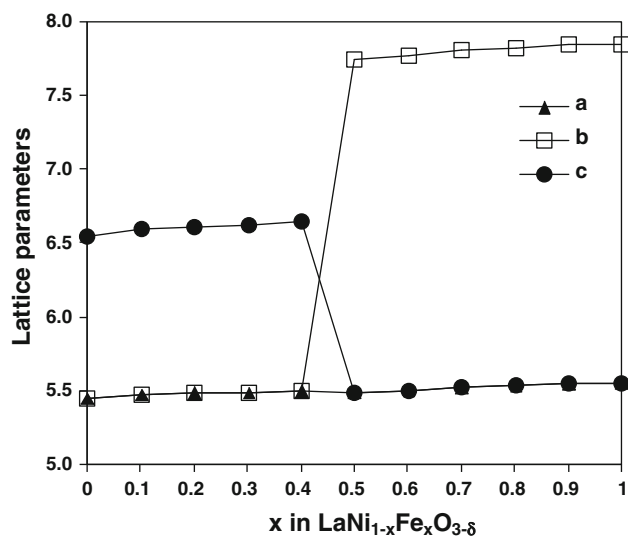


Fig. 4 Lattice parameters a , b , and c of LNF ($x = 0.0$ – 1.0) synthesized by MWC and calcined at $900\text{ }^{\circ}\text{C}$ for 5 h

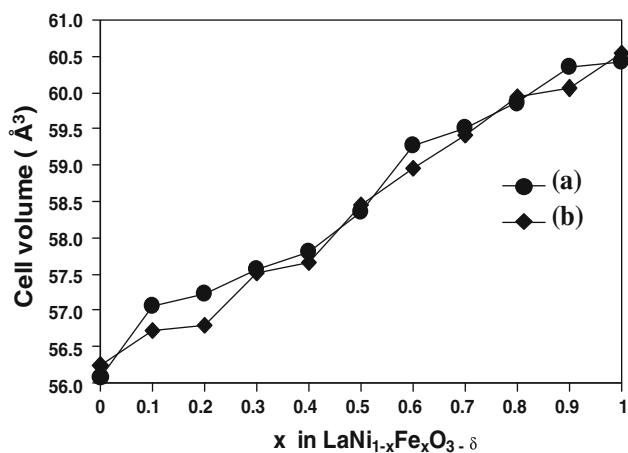


Fig. 5 Unit cell volume of LNF ($x = 0.0$ – 1.0) synthesized by (a) WC and (b) MWC, and calcined at $900\text{ }^{\circ}\text{C}$ for 5 h

and $800\text{ }^{\circ}\text{C}$ and orthorhombic at around 900 – $1000\text{ }^{\circ}\text{C}$. In the study, LNF37 maintains a single phase of an orthorhombic structure in the range of 700 – $1000\text{ }^{\circ}\text{C}$. The structural difference at lower temperature might be due to the preparation procedure. Bontempi et al. used the MWC approach to synthesize LNF37 with evaporation of the precursor solution at $90\text{ }^{\circ}\text{C}$, drying at 70 – $180\text{ }^{\circ}\text{C}$ for 5 days, then heating at $400\text{ }^{\circ}\text{C}$ before calcination at the desired temperature. The evaporation step was simply done by heating the precursor solution directly, and the temperature of precursor mixtures was raised to 300 – $400\text{ }^{\circ}\text{C}$ instantly by auto-ignition. The self-combustion proceeded and completed in less than 2 h. The complete combustion method could allow LNF37 to possess the thermodynamically stable structure. This follows the theory that the ideal perovskite structure is cubic with the tolerance factor of 1.

LNF37 has the tolerance factor of 0.97, leading to the preferable orthorhombic structure [18]. At the temperature above 1000 °C, the structure partially decomposed with the presence of La_2NiO_4 (JCPDS: 34-0314) and La_2O_3 (JCPDS: 24-0554). LNF64 possesses a cubic structure at the low calcination temperature of 700–800 °C and becomes a rhombohedral structure when the temperature is above 800 °C. This temperature is also lower than that reported by Bevilacqua et al. [3] which was at 1000 °C.

SEM micrographs in Fig. 7 show the powder morphology from different calcination temperatures. The mean dimension of particle size ranges from ~ 0.2 to ~ 0.4 μm with an increase of the calcination temperature from 900 to 1000 °C. When compare LNF37 with LNF64, the presence of Fe in the structure seems to favor the fine granular morphology.

Chemical compatibility of LNF64 with 8YSZ

The XRD patterns of LNF64–8YSZ powder mixtures which were fired at 800–1200 °C in air for 5 h were determined as shown in Fig. 8. It was found that $\text{La}_2\text{Zr}_2\text{O}_7$ (JCDPS: 17-0450) was initially formed at 1000 °C with the presence of the peak at 28.5°. At 1100 and 1200 °C, the $\text{La}_2\text{Zr}_2\text{O}_7$ and NiO (JCPDS: 47-1049) phases were clearly observed and the original components disappeared. Furthermore, the shift of the LNF peaks to the lower diffraction angle indicates the volume expansion of perovskite phase, caused by the dissolution of larger Zr cations into the perovskite structure [17, 19]. Comparing the result with Chiba's group in which the chemical stability study of LNF64 to 8YSZ at 1000 °C was reported [1]. This might be due to the mixture was fired only for 2 h but 5 h firing in the study.

To simulate the SOFC configuration, the chemical compatibility was studied at the surface between LNF64

and 8YSZ. EDX spectroscopy coupled with SEM was carried out on three selected points, and the percent of metallic elements at each point were plotted in Table 2. La, Ni, and Fe could not be detected at point 1 (10 μm from the interface) and 2 (near to the interface) in the 8YSZ pellets for both firing temperatures of 900 and 1000 °C. Zr cannot be detected at point 3 (10 μm from the interface) residing in LNF64 with the firing temperature of 900 °C, but a small amount was found at 1000 °C. This result confirms the previously mentioned XRD results that the unwanted reaction between LNF64 and 8YSZ starts at 1000 °C.

Physical compatibility of LNF64 with 8YSZ electrolyte

The physical compatibility between LNF64 and 8YSZ was investigated in term of the TEC. The close TEC values of both materials are the requirement for use as the components in a SOFC.

The TEC of LNF64, obtained in this study is higher than those previously reported for the same materials, as shown in Table 3. This may involve the differences in particle sizes of LNF64 obtained from different synthesis methods. MNC method produced the smaller particle size; hence, the better packing in preparation of the TEC sample bars. The bars were sintered at 1200 °C prior to the TEC measurement, and the good packing particles resulted in a high degree of sintering and less pore volume within the TEC bar. In this case for the TEC measurement, the LNF64 particles obtained from MNC method had less pore volumes which acted as a buffer space for the expansion. Therefore, the TEC of this LNF64 bar became very large when compared with those synthesized from other methods and having larger particle sizes and hence greater porosity. The stress remaining within the bar may play a role in the high TEC as well as the effect of small particle sizes. More investigation is necessary for this assumption. Nonetheless, the TEC of LNF64 synthesized in this study is in the same range with that of 8YSZ, indicating their good physical compatibility.

Electrical conductivity

To investigate the influence of the synthesis methods on the internal resistance of the cells, the Nyquist plots of LNF64 prepared using four methods at 700 °C were compared in Fig. 9. The size of the semicircle corresponds to the specific interfacial resistance between the cathode and electrolyte, indicating its greater values of LNF64 prepared by unmodified citrate methods than those prepared from modified citrate methods. As discussed in “[Synthesis method of LNF64](#)” section, the modified citrate methods yield smaller particle size powder which led to a better

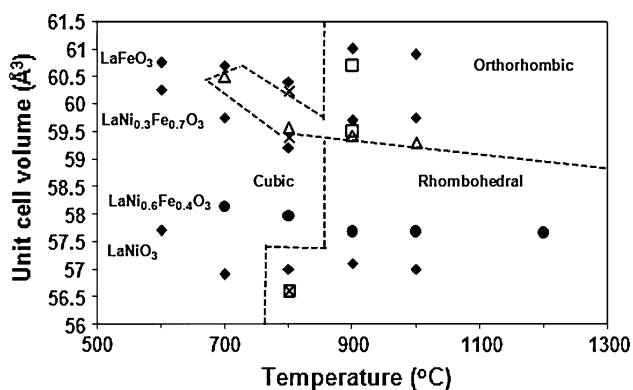


Fig. 6 Unit cell volumes as a function of calcinations temperature for LNF37 (open triangle) and LNF64 (filled circle) in the study. In comparison with Bontempi et al. [14] (filled diamond), Falcon et al. [18] (open square), and Provendier et al. [15, 16] (times)

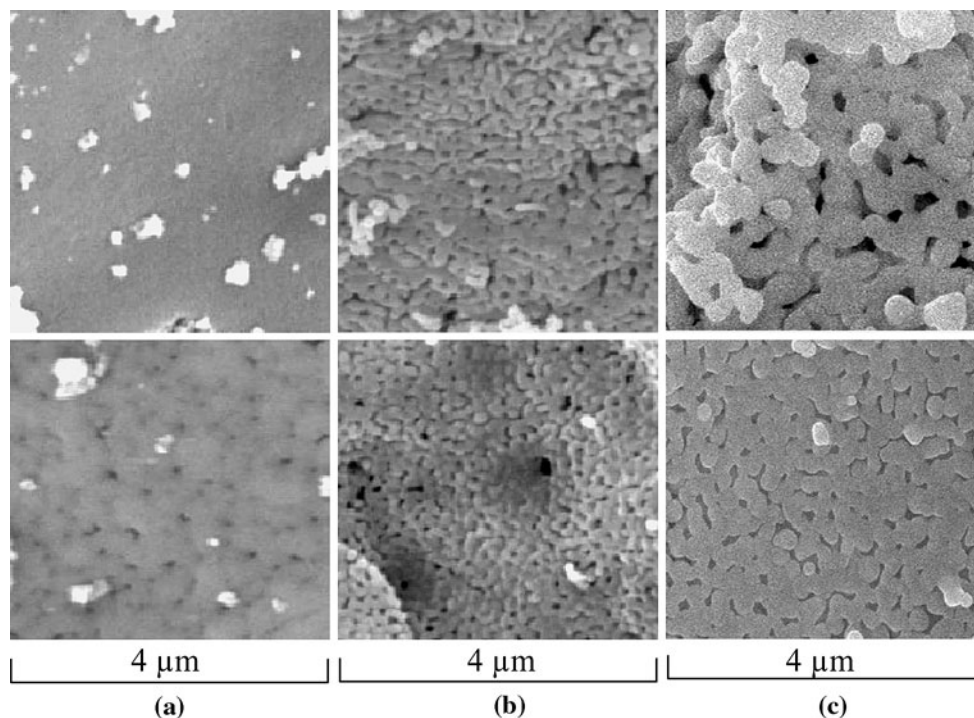


Fig. 7 SEM micrographs of LNF37 (*upper*) and LNF64 (*lower*) synthesized by WMC at various calcination temperatures; (a) 700, (b) 900, and (c) 1000°C

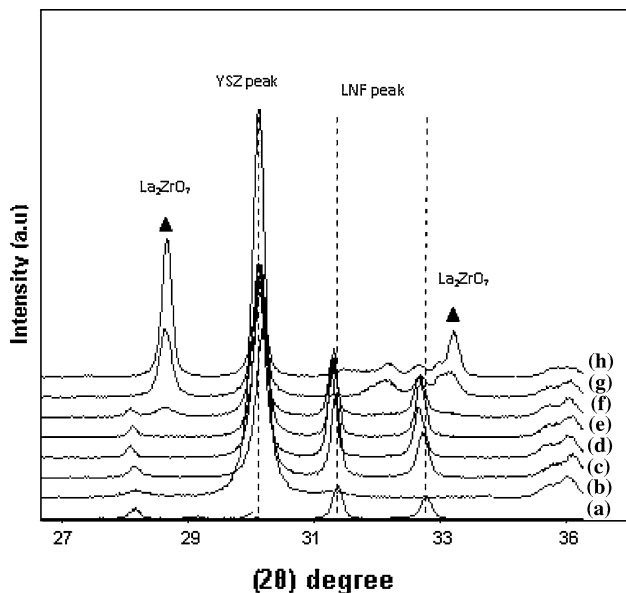


Fig. 8 XRD patterns between a 2θ range of 25° – 36° of (a) LNF64, (b) 8YSZ, and the powder mixtures between LNF64 and 8YSZ: (c) at room temperature, (d) after calcinations for 5 h at 800, (e) 900, (f) 1000, (g) 1100, and (h) 1200 °C

particle packing and higher degree of sintering. This may cause greater area of triple phase boundary which is one reason for smaller resistances. However, the consideration should include more studies to avoid the technical conflict with at least other two factors, i.e., the triple phase

Table 2 Zr, La, Ni, and Fe elemental percentage of the cross-sectioned LNF64 (MNC method)/8YSZ pellet after calcination at 900 and 1000 °C for 5 h

	Calcination at 900 °C			Calcination at 1000 °C		
	Point 1	Point 2	Point 3	Point 1	Point 2	Point 3
Zr	100	100	0	99.64	99.75	4.37
La	0	0	70.51	0.1	0.12	22.9
Ni	0	0	18.36	0.06	0.1	63.5
Fe	0	0	11.13	0.2	0.03	9.23

Table 3 The thermal expansion coefficients of LNF64 samples and 8YSZ at 800 °C

Materials	Synthesis method(s)	TEC ($\times 10^{-6} \text{ K}^{-1}$)
LNF64 [in this study]	MNC	13.2
LNF64 [2]	Citrate gel and urea	11.8
LNF64 [1]	Solid state reaction	11.4
$\text{La}_{0.8}\text{Sr}_{0.2}\text{Fe}_{0.9}\text{Co}_{0.1}\text{O}_3$ [20]	Solid state reaction	13.9
$\text{Pr}_{0.8}\text{Sr}_{0.2}\text{Fe}_{0.8}\text{Co}_{0.2}\text{O}_3$ [20]	Solid state reaction	13.2
8YSZ [21]	–	10.5

boundary length and packing patterns. Nevertheless, if the packing is too dense, the resistance will increase. As previously reported by Basu et al. [10], the results showed that the higher density and the better conductivity LNF were obtained from smaller particle size powder.

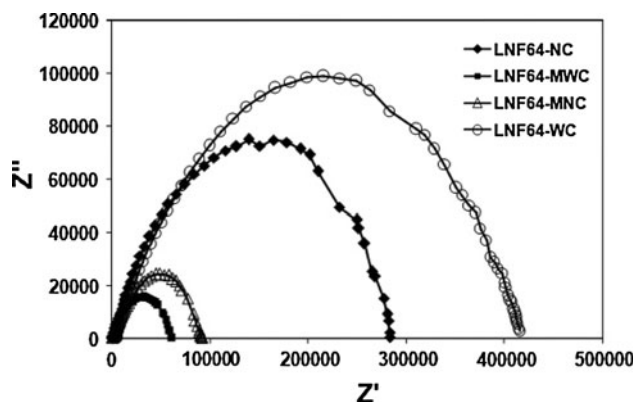


Fig. 9 Nyquist plots of LNF64 cathodes prepared using four methods at 700 °C

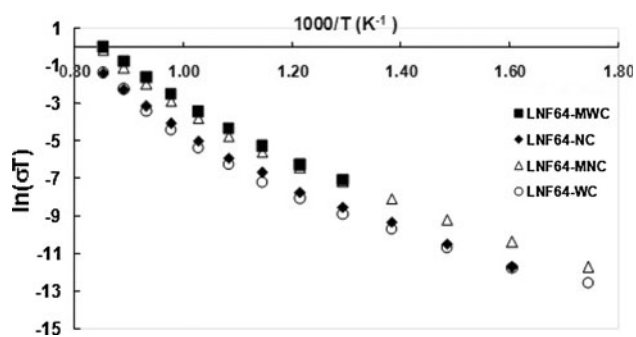


Fig. 10 Arrhenius plots of the conductivity of LNF64 prepared using four methods

The electrical conductivity (σ) of LNF64 cathodes can be calculated using the Arrhenius equation [3]. Plots of $\ln(\sigma T)$ as a function of the reciprocal of absolute temperature were shown in Fig. 10. The straight lines are obtained with the correlation coefficient $R^2 \geq 0.99$. This means that the conduction can be governed by the small polaron mechanism [1]. All lines contain two slopes, indicating that at least two mechanisms govern the conductivity at the temperature between 300 and 950 °C.

Conclusions

LNF perovskite powders have been successfully synthesized by four combustion methods. With the merit of a homogeneous reactant mixture and a relatively low calcination temperature, a single phase is obtained for all ratios of nickel to iron. The different particle size is obtained depending on the amount of fuel in the reaction mixture of

each method. The LNF perovskite with higher Fe content shows better thermal stability. The chemical and physical compatibility with 8YSZ is comparable to the LNF synthesized from the conventional methods. Synthesis technique has an effect on the cathode electrical performance of LNF64. Among the four synthesis methods, the MWC method was found to be the most promising one for fuel cell applications.

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