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Thermomechanical Analysis of Various Solid-oxide-fuel-cell Components Using Simple Analog Micrometer Measurements

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Abstract

The solid oxide fuel cell (SOFC) is a device that can convert the energy stored in gaseous chemicals such as hydrogen into electricity. Because SOFCs are operated at high temperatures, their structures must be stable and durable. The thermomechanical compatibility of SOFC components is the main issue, as negligence in regard to material compatibility leads to cell destruction. In this study, we investigated the thermal expansion coefficient (TEC) of SOFC components, which characterizes their thermomechanical properties. We measured the TEC value of various electrolyte and electrode materials [La_{0.933}Si₄O₂₆ (LSO), Ce_{0.9}Gd_{0.1}O_{1.92} (CGO), La_{0.8}Sr_{0.2}Ga_{0.8}Mg_{0.2}O_{2.55} (LSGM), LSO-CGO, LSO-LSGM, and La_{0.7}Ca_{0.3}MnO₃ (LCM)] using an analog micrometer at temperatures between 298 K and 1073 K. The obtained TEC values matched well with the theoretical references, with errors between 1.80% and 8.00% for LSO, CGO, LSGM, and LCM. The TEC of composite SOFC materials, LSO-LSGM and LSO-CGO, were $10.29 \times 10^{-6} \text{ K}^{-1}$ and $10.10 \times 10^{-6} \text{ K}^{-1}$, respectively. Given the slight difference in their TEC values, these electrolytes would thermomechanically match an LCM cathode.

Keywords: SOFC electrolytes, SOFC electrode components, TEC, analog micrometer, thermomechanical compatibility

Introduction

The solid oxide fuel cell (SOFC) is a device that can convert gaseous hydrogen into electricity. SOFCs operate in temperatures ranging from 873 K to 1473 K (600 °C–1200 °C). Because SOFCs can be constructed using a multiple-cell stack design, they are a promising energy source for high-temperature-operation systems and have the potential to produce up to 10 MW of electricity [1]. To withstand such high operation temperatures, SOFCs must be built to ensure thermomechanical and physical durability. An SOFC contains three main components: an electrolyte, anode, and cathode. To maintain good performance, these three components must be compatible. The thermal expansion coefficient (TEC) is an important parameter used to characterize the thermomechanical compatibility of an SOFC. All SOFC components must have similar TEC values. Negligence regarding the mismatch of TECs will lead to performance degradation from delamination, cracks, and vigorous cell destruction [2]. The basic features of the SOFC components, i.e., their chemical properties and structure of the materials, affect their TEC values [3].

Based on fundamental principles for determining expansion coefficients, dilatometric analysis is usually performed using a dilatometer [4-6]. This instrument provides the best accuracy by measuring the *in-situ* expansion as the temperature increases. However, the simultaneous measurement of all the materials leads to a very costly analysis. Letilly *et al.* [7] analyzed the TEC of the BIT07 electrolyte using X-ray diffraction (XRD) analysis at room temperature, whereby each sample was sintered at different temperatures, with the underlying assumption that the chemical expansion could transform the TEC value. Blum [8] conducted TEC measurements using a standard F-design. Based on the fact that material expansion occurs simultaneously in all directions, this author combined the basic principles of linear expansion that can be observed with room temperature measurements after sintering at a certain temperature, which is similar to the assumptions made by Letilly *et al.* [7]. The TEC measurements by Blum [8] were simply made using an analogous micrometer.

As a common anode material, NiO was used as the anode in a comparison study with other SOFC component materials. In that study, the main electrolyte

selected was $\text{La}_{0.33}\text{Si}_4\text{O}_{26}$ (LSO), which was further processed using the solid-state method to produce composite electrolytes with other electrolyte materials, $\text{Ce}_{0.9}\text{Gd}_{0.1}\text{O}_{1.92}$ (CGO) and $\text{La}_{0.8}\text{Sr}_{0.2}\text{Ga}_{0.8}\text{Mg}_{0.2}\text{O}_{2.55}$ (LSGM) [9]. LSO and CGO are considered to be intermediate-temperature SOFC electrolytes, whereas LSGM is a well-known good ionic conductor electrolyte in the low-to-mid temperature range [10]. To monitor the TEC values in low- and mid-range operation temperatures, the electrolytes were treated as variables. The cathode, $\text{La}_{0.7}\text{Ca}_{0.3}\text{MnO}_3$ (LCM), was synthesized using a solid-state method. This cathode was selected because it exhibits high electronic conductivity. Theoretically, all the possible combinations were predicted to be good SOFC single-stack constructions. To determine the thermomechanical compatibility of all the components in practical terms, the experimental TEC values must be measured.

The main purpose of this study was to construct a SOFC prototype and explore its TEC values. To study the TEC behavior, both the electrolyte and electrode components were observed. TEC is traditionally measured using a dilatometer [11,12]. In this study, we used a new and simpler method to monitor the TEC value of the SOFC components. This method is based on dilatometric analysis but does not involve the use a dilatometer. Instead, we used a manual micrometer over the experimental temperature range (298 K to 1073 K).

Methods

CeO_2 (99.9%), Gd_2O_3 (99.9%), MnO_2 (99.9%), CaCO_3 (99.9%), and NaOH (99.9%) were obtained from Merck Singapore. LSGM (99.99%), La_2O_3 (99.99%), $\text{Na}_2\text{SiO}_3 \cdot 5\text{H}_2\text{O}$ ($\leq 95\%$), and La_2O_3 (99.99%) were obtained from Sigma-Aldrich. The desired materials were all produced using the methods reported in our previous study [13]. XRD analysis was performed using a Rigaku MiniFlex600 X-Ray Diffractometer (γA system,

$\text{Cu K}\alpha$ radiation, $\lambda = 0.15418 \text{ nm}$, $T = 298 \text{ K}$, $v = 0.02^\circ$ in the 2θ range 20° – 80°). Data fitting was conducted using *PANalytical X'Pert Highscore Plus*© software and the ICSD/ *International Crystallographic Structure Database*. Sample pellets (0.4–0.6 g) were uniaxially pressed to a diameter of $\sim 1 \text{ cm}$ using a *Graseby Specac* and then sintered at 1473 K to obtain similar initial densification conditions. The dimensions of the sample pellets were then measured using an analog micrometer at 298 K to obtain the initial dimensions. Thereafter, the sample pellets were heated at a rate of 5 K min^{-1} to the desired temperature, which was maintained for 2 h. The pellet dimensions were measured every 100 K up to 1073 K. The measured dimensions of the pellets were then applied in the TEC calculations using Equation 1.

Results and Discussion

Figures 1 and 2 present the XRD patterns of the SOFC components that were successfully prepared in this study. All the patterns were analyzed using the Rietveld refinement technique to obtain their lattice parameters, reliability profiles (R_p), reliability weight profiles (R_{wp}), and goodness of fit. The XRD patterns conformed to the ICSD standards 98-015-5624 (LSO), 98-002-8795 (CGO), 98-009-8170 (LSGM), and 98-008-8389 (LCM). These materials were then used for TEC measurements. Table 1 lists the refinement parameters of the samples.

TEC is one of the most important parameters for evaluating the thermomechanical compatibility of SOFC components. In this study, we calculated the TEC values as follows:

$$TEC = \left(\frac{\Delta L}{L_0} \right) / (T - T_{initial}), \quad (1)$$

where ΔL is the change in length, L_0 is the initial length, $T_{initial}$ is the initial temperature, and T is the final temperature.

Table 1. TEC Values of Various SOFC Components

Sample	Structure	Space Group	a/Å	b/ Å	c/ Å	R _{expected}	R _p	R _{wp}	Gof
LSO	Hexagonal	<i>P 63/ m</i>	9.7271(8)	9.7271(8)	7.1863(6)	18.4538	20.9223	27.0724	2.1522
CGO	Cubic	<i>F m -3 m</i>	5.4128(7)	5.4128(7)	5.4128(7)	16.9718	12.0553	17.1487	1.0210
LSGM	Cubic	<i>P m -3 m</i>	3.9123(4)	3.9123(4)	3.9123(4)	7.2910	10.1499	13.5389	3.4482
LSO-CGO	Hexagonal-Cubic	-	-	-	-	5.5358	8.6783	10.9325	3.9000
LSO-LSGM	Hexagonal-Cubic	-	-	-	-	8.6988	11.0689	15.0148	2.9793
LCM	Orthorhombic	<i>P n m a</i>	5.4640(1)	5.7270(5)	5.4781(2)	15.6077	12.8506	16.7095	1.1462

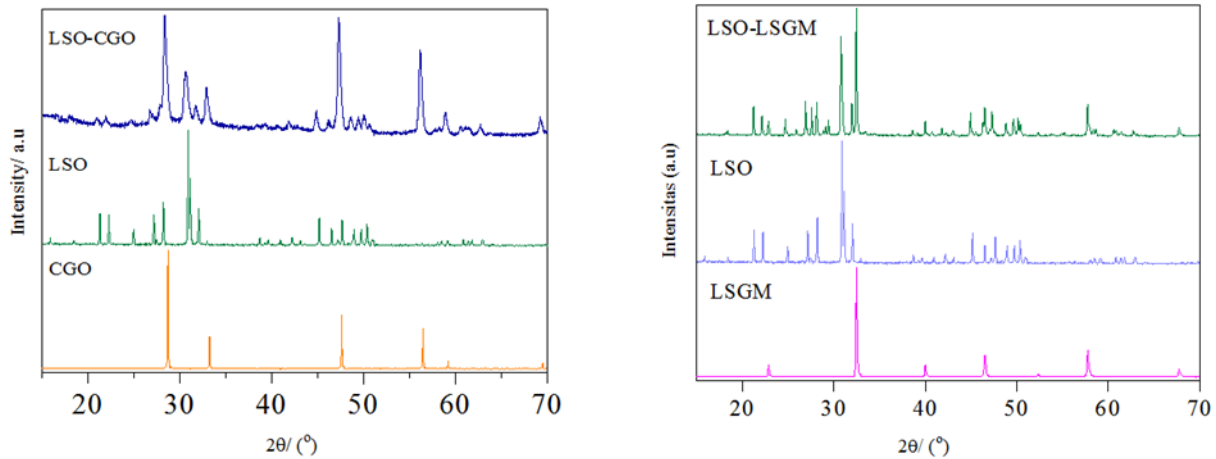


Figure 1. XRD Patterns of LSO, CGO, and LSO-CGO. LSO were Synthesized Hydrothermally. CGO and LSO-CGO were Prepared by Solid-state Synthesis (Left). XRD Patterns of LSO, LSGM, and LSO-CGO. LSO-LSGM was Prepared by Solid-state Synthesis (Right)

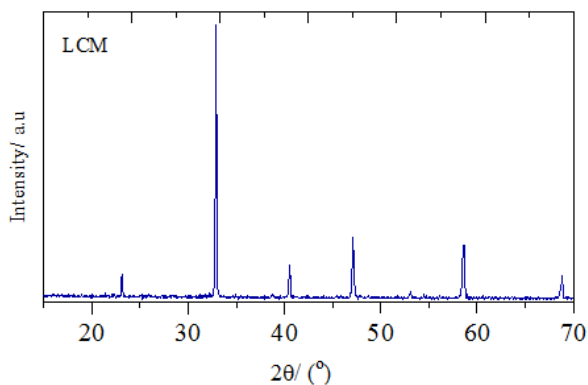


Figure 2. XRD Pattern of the LCM Cathode Prepared by Solid-state Synthesis

Table 2 presents the TEC values obtained in this study along with the values reported in the literature (theoretical values). From the reported and experimental values, we can quantify the differences by calculating the relative error percentage (Rel. error) using the following equation:

$$Rel. error = \left| \frac{TEC_{practical} - TEC_{theoretical}}{TEC_{theoretical}} \right| \times 100\%. \quad (2)$$

Except for LSO, for which a significant statistical error was found, the relative errors were less than 5%. To study the TEC behavior further, we examined the changes in the cell dimensions and volumes that occurred within the test range of 298 K to 1073 K. Figures 3 to 6 show plots of the results for each component. Each figure shows a trend of increasing length with increases in temperature [8]. The positive gradient value emphasizes this trend, however, the gradient of each component was slightly different due to the respective properties of the materials.

Table 2. TEC Values of the Studied SOFC Component Materials

Components	References	Theoretical/ $\times 10^{-6}K^{-1}$	This Study/ $\times 10^{-6}K^{-1}$
LSGM	[14]	11.5	11.77
LSO	[15]	9.88	10.67
CGO	[16]	12–12.7	(not studied)
LSO-CGO	-	(not reported)	10.10
LSO-LSGM	-	(not reported)	10.29
LCM	[17]	10.5	10.30
NiO	[18]	13.9	(not studied)

Table 3. Relative Error of TEC Values for Various SOFC Components

SOFC Components	Relative error/%
LSGM	2.35
LSO	8.00
CGO	-
LSO-CGO	-
LSO-LSGM	-
LCM	1.92

In Figure 3, we can also observe a gap between points that may have a similar order as those of LSO-CGO. With increases in temperature, the sample lengths increased linearly for LSO-LSGM and LCM. However, for LCM, we found that the linear length increased dramatically between 873 K and 1072 K, which is similar to the behavior of NiO-CGO, although that increase began at 773 K. This behavior significantly affected the TEC value. To examine the shift, we measured these changes carefully (Table 3).

Table 4. Expansion of SOFC Component Dimensions with Increasing Temperature

Temperature (T)/ K	Linear dimension (A/t)/ cm				
	LSO-CGO	LSO	LCM	LSO-LSGM	LSGM
298	76.1551	43.2363	42.9239	40.8494	37.8690
373	76.1551	43.2363	42.9700	40.9314	38.0215
473	76.3958	43.4198	42.9732	40.9574	38.1361
573	76.7752	43.5608	42.9766	40.9475	38.5408
673	76.8512	43.6897	42.9802	40.9798	38.5792
773	77.0033	43.7329	42.9841	40.9798	38.5792
873	77.0794	43.7437	42.9881	41.0207	38.5621
973	77.0794	43.7979	43.0379	41.0369	38.6005
1073	77.1187	43.8089	43.1294	41.1187	38.6220

(T ₂ -T ₁)	ΔL/ cm				
	LSO-CGO	LSO	LCM	LSO-LSGM	LSGM
373-298	0.0000	0.0000	0.04607	0.0820	0.1525
473-373	0.2407	0.1835	0.0032	0.0160	0.1146
573-473	0.3794	0.1404	0.0034	0.0000	0.4046
673-573	0.0760	0.1295	0.0036	0.0323	0.0384
773-673	0.2282	0.0432	0.0038	0.0000	0.0000
873-773	0.0761	0.0109	0.0040	0.0409	-0.0170
973-873	0.0000	0.0541	0.0499	0.0163	0.0384
1073-973	0.0392	0.0111	0.0914	0.0818	0.0215
Average	0.1300	0.0716	0.0257	0.0337	0.0941

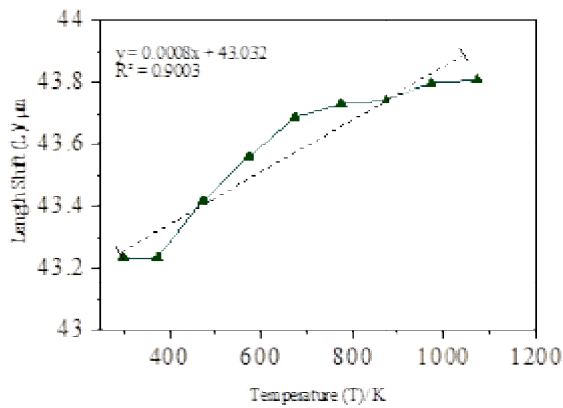


Figure 3. Expansion of LSO

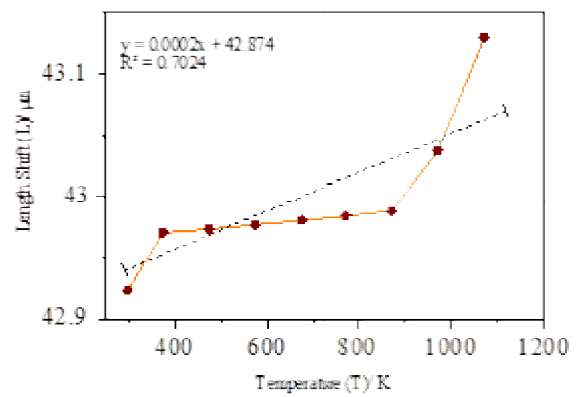


Figure 4. Expansion of LCM

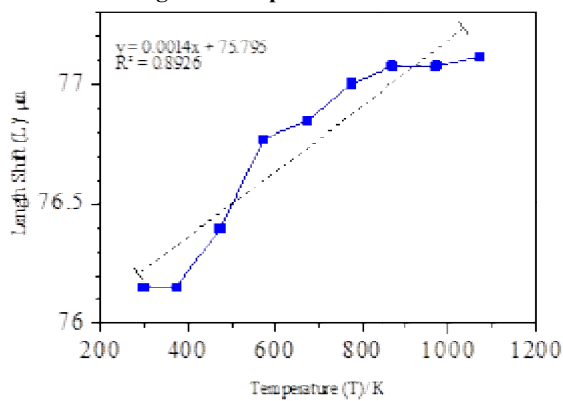


Figure 5. Expansion of LSO-CGO

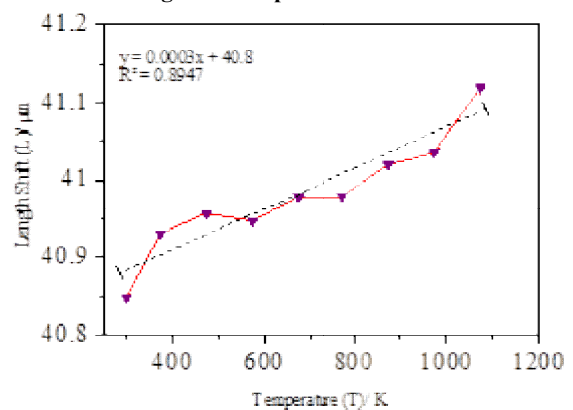


Figure 6. Expansion of LSO-LSGM

The positive gap shown in Table 4 indicates that the value of each point increased with increasing temperature. A zero value indicates that no meaningful change occurred as the temperature increased, whereas a negative value indicates a slight decrease in linear length as the temperature increased. Negative changes have yet to be reported and may have been caused by the *late ratio* of the linear length obtained from the calculation. A *late ratio* phenomenon was observed in this study due to dilatation of the material in every direction. A *late ratio* occurs when the ratio between the areas of a cylinder to its thickness at higher temperature is lower than the ratio at a lower temperature. The occurrence of this phenomenon suggests that linear expansion, as obtained from the ratio, did not occur at certain temperature, whereas volume expansion did occur. Since the TEC values obtained in this experiment are linear expansion coefficients, the area of the cylinder had to be divided by its thickness. As such, a negative value indicates a *late ratio*, which means that no linear expansion occurred at certain temperature between 873–773 K. Further study to learn more about this phenomenon is required to better understand how the late ratio behaves.

Figure 7 shows the variation in $\Delta L/L_0$ for each sample. LSGM experienced a sudden shift at 800 K. LSO-CGO and LSO also underwent a sudden shift at temperatures

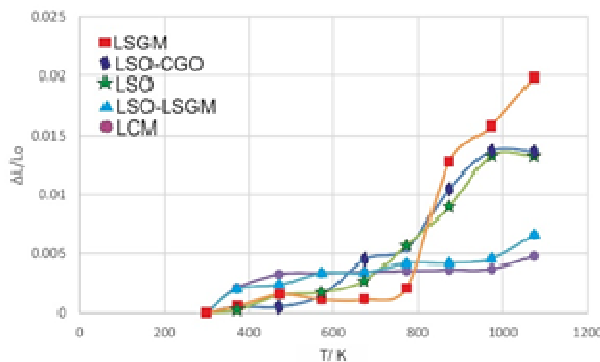


Figure 7. Expansion Ratio of Each Material

above 800 K. The LSO-CGO composite showed a slight change in its curve above that of its original matrix material, LSO. On the other hand, the electrolyte composite LSO-LSGM experienced a much smaller change than its original material, LSO, or even LSGM. This difference verifies the conclusions that could be drawn based on the TEC values, as shown in Figure 8.

Figure 8 shows the TEC values at every temperature point of all the above materials. Three materials (LSGM, LSO-CGO, and LSO) showed a negative gradient or decreasing TEC value with increases in temperature. This implies a tendency that increases in temperature could cause infinite expansion of the material until it reaches a point of maximum expansion in its linear length before becoming permanently deformed. At that time, a positive gradient implies a lower rate of linear length expansion to a finite point near and above 800 K. Until this point, the average TEC would reflect the overall TEC behavior, although the TEC might vary at different temperatures, as shown in Table 3. The TEC also might differ in some temperature ranges based on the materials used, with expansion occurring until a certain high temperature, after which deformation takes over. However, in this study, the temperature was set to 1073 K as a mid-range SOFC operational temperature.

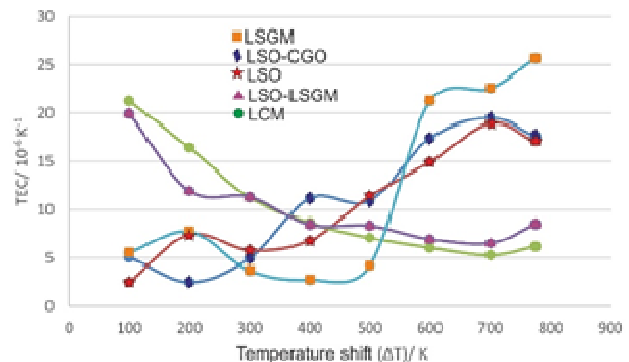


Figure 8. TEC Shifts of Various SOFC Materials

Table 5. TEC Shifts with Increases in Temperature

ΔT	TEC Value/ $10^{-6}K^{-1}$				
	LSO-CGO	LSO	LCM	LSO-LSGM	LSGM
775	16.3268	17.090	6.1758	8.5061	25.6579
700	18.0761	18.921	5.3042	6.5489	22.6524
600	15.8203	15.001	6.0634	6.9874	21.3837
500	9.0200	11.507	7.1168	8.3849	4.2904
400	8.7802	6.8968	8.6854	8.5040	2.8282
300	5.0492	5.8649	11.2844	11.3387	3.7709
200	2.5768	7.5402	16.4596	12.0008	7.8976
100	5.1536	2.5629	21.2991	20.0217	5.6653
AVG	10.1004	10.6730	10.2986	10.2866	11.7683

The TEC values observed in Table 5 also reveal that the presence of LSGM and CGO would lead to an increasing TEC value for LSO. From this finding, we can also estimate the effect of other reinforcement materials in the compound matrix. From all the obtained TEC values, we can conclude that the LSO-LSGM composite electrolyte might be the best electrolyte for an LCM cathode, as its TEC is only slightly different at approximately $1.2 \times 10^{-8} \text{ K}^{-1}$ (negligible difference of 0.012). However, the TEC value of the cathode must be slightly higher than that of the corresponding electrolyte. Hence, the electrolyte would experience compression during cell-cooling at the fabrication temperature. A great mismatch in TEC values will lead to cell destruction due to buckling [17].

Conclusion

In this study, the TEC behaviors of various materials were studied via dilatometric analysis using an analog micrometer in the temperature range of 298 K to 1073 K. The observed behaviors confirmed the characteristics and compatibilities of these materials as SOFC components. The measured TEC values compared well with the theoretical references, with the relative errors ranging from 1.80% to 8.00% for LSO, CGO, LSGM, and LCM. The TEC values of the composite SOFC materials, LSO-LSGM and LSO-CGO, were $10.29 \times 10^{-6} \text{ K}^{-1}$ and $10.10 \times 10^{-6} \text{ K}^{-1}$, respectively, which indicate their suitability for use in an LCM cathode (TEC $10.30 \times 10^{-6} \text{ K}^{-1}$). We found the most thermomechanically compatible material for an LCM cathode, with respect to the TEC difference, to be the LSO-LSGM electrolyte.

Acknowledgments

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