

SAMARIUM-DOPED CERIA/YTTRIA-STABILIZED ZIRCONIA
COMPOSITE PREPARED BY SOLID STATE REACTION*Fitria Rahmawati^{1, *}, Anang Pandan Respati¹, Mudjijono¹, Dani G. Syarif²*<https://doi.org/10.23939/chcht12.04.434>

Abstract. A research on the preparation of composite samarium-doped ceria (SDC) with yttrium-stabilized zirconia (YSZ) has been conducted at the SDC:YSZ ratio of 0:1; 1:9; 1:1; 9:1 and 1:0. This research aims to investigate the crystal structure and the ionic conductivity of the prepared materials. XRD analysis equipped with Le Bail refinement was used to analyze the crystal structure, space group, cell parameters, and cell volume. Meanwhile, the ionic conductivity was determined by impedance measurement. The result shows that the composites of SDC-YSZ were crystallized in two phases of cubic SDC and cubic YSZ without the presence of secondary phases. It indicates that the mixing and the heating at 1523 K did not change the crystal structure, even though it changed the cell parameters. Composite SDC-YSZ 91 shows a highest ionic conductivity at 873 K without the presence of electrode-bulk interface conductivity. It is a good indication of YSZ role regarding the inhibition of cerium ions auto-reduction.

Keywords: SDC-YSZ composite, electrolyte, solid oxide fuel cell, ionic conductivity.

1. Introduction

Since industrial revolution started 120 years ago, the global warming effect occurred, due to high carbon dioxide emission. This was mainly caused by combustion engine to produce energy from fossil fuel. Developing technology to produce clean and renewable energy is urgently required. A fuel cell is a promising alternative method. A fuel cell is an electrochemical cell that converts chemical energy to electrical energy through an electrochemical reaction [1]. Fuel cell shows high efficiency (50–70 %) and does not release carbon dioxide gas when use hydrogen as fuel [2]. Solid oxide fuel cells

(SOFCs) have attracted some researchers due to the potential application for electrical power production with high efficiency and low pollution when compared to classical power plants [3, 4]. The high operational temperature of SOFCs provides high efficiency of conversion to produce electric power. Also, flexibility in fuel choices, such as hydrocarbon, bioethanol, biogas, *etc.* can be used as fuel without preliminary gas conversion. However, the high operational temperature may cause the degradation of material stability and high operational cost [1]. Recently, researches on SOFCs focus on the decreasing of operational temperature from 1273 to 773–1073 K [5]. However, decreasing of operational temperature allows increasing ohmic loss in the electrolyte and polarization loss on electrode [6]. Therefore, a high conductivity of the electrolyte is required to reduce the ohmic loss and increase the fuel cell performance.

Cerium dioxide, CeO₂, is a promising candidate material for fuel cell electrolyte due to it can accommodate a high oxygen deficiency by substitution of lower valent elements on cation sublattice that leads to providing high oxygen ion conductivities [7, 8]. Samarium doped ceria (SDC) is the stabilized ceria with highest ionic conductivity [9]. However, cerium oxide is not stable for electrolyte due to the possibility of Ce⁴⁺ to reduce into Ce³⁺ that promotes electronic conduction when used as electrolyte material [10] under low oxygen partial pressure (1·10⁻⁴ Pa) and high temperature (>873 K) [5]. This electronic conduction may cause cell performance degradation due to electronic leakage currents between the anode and cathode [5] or referred

as a short circuit. Dual phases ceria based electrolytes such as doped-ceria that mixed with salts (sulfates, halides or hydrate) [11, 12], Nd-doped ceria with (La_{0.95}Sr_{0.05})_xGa_{0.9}Mg_{0.1}O_{3-δ} [13] or carbonate salts [14] that have proven effectiveness for application in intermediate-low temperature SOFCs (ILT-SOFCs) [15, 16]. A well-qualified including phase stability and high ionic conductivity is also not found in a single-phase electrolyte at low temperature. Improving ionic conductivity by varying grain boundary conditions is an alternative [17]. Mishima *et al.* [18] conducted research on the preparation

¹ Research Group of Solid State Chemistry & Catalysis, Chemistry Department, Sebelas Maret University, Jl.Ir. Sutami 36 A Kentingan, Surakarta 57126, Indonesia

² Ceramics Lab., National Atomic Energy Agency, Badan Tenaga Atom Nasional (BATAN),

Jl. Taman Sari Bandung, Indonesia

* fitria@mipa.uns.ac.id

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of SDC-YSZ at various weight ratio and investigated its performance in a single cell with LSM as cathode and Ni-YSZ as cathode. It was found that the open circuit voltage (OCV) of a fuel cell with biphasic electrolyte, SDC dispersed in YSZ matrix, was significantly higher than single SDC electrolyte. Other researches on YSZ-SDC production have also been conducted with a glycine nitrate method [19]. However, the study of its crystal structure, electrical properties, and energetics of ionic migration of the SDC-YSZ mixture were not conducted. Therefore, in this research, the SDC-YSZ composites were prepared at various weight ratio to investigate their crystal structure, cell parameters, conductivity and also their theoretical ionic conductivity based on their composition. The Arrhenius plot of conductivity values could analyze the activation energy of ionic migration. The study on the crystal structure was conducted to investigate the structural change due to mixing process.

2. Experimental

Samarium doped ceria, $\text{Ce}_{0.8}\text{Sm}_{0.2}\text{O}_{1.9}$ was prepared through sol-gel method [20]. All chemicals used were of pro analysis grade. A solution was prepared by dissolving 7.04 g of cerium nitrate hexahydrate, $\text{Ce}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ (99%, 434.22 $\text{g}\cdot\text{mol}^{-1}$, Aldrich USA) and 1.47 g of samarium nitrate hexahydrate, $\text{Sm}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ (99.9%, Aldrich USA) in 100 ml of deionized water. Citric acid was used as chelating agent, and polyethylene glycol/PEG (950–1050 $\text{g}\cdot\text{mol}^{-1}$, Merck Germany) was used as the gel-forming agent. The solution was stirred at 323 K until forming a transparent gel, which then was heated at 973 K for 3 h. The prepared powder was analyzed by X-ray diffraction equipped with Le Bail refinement to study its crystal structure and the change of structure after the addition of YSZ at various ratio. The commercial 8YSZ procured from TOSO (Japan) was used. The composition was 8 mol % Y_2O_3 in ZrO_2 . X-ray diffraction analysis was also conducted to the 8YSZ.

The composite electrolytes were prepared *via* solid state reaction with the ratio of mixed components SDC:YSZ 0:1; 1:9; 1:1; 9:1, and 1:0 w/w. The mixture was crushed for 2 h in a mortar. The mixture powder was pressed by a hydraulic press to form pellets and then sintered at 1523 K for 5 h. The choice of 1523 K sintering temperature was based on the researches where SDC/YSZ layer was prepared on NiO-YSZ anode substrate and sintering was conducted on the prepared SDC/YSZ layer at 1673 K for 2 h [21]. It was found that the inter-diffusion of doped ceria elements into YSZ layer occurred. The ratio of thickness to the area (l/A) of the sintered pellets is $0.15 \pm 0.04 \text{ cm}^{-1}$. The electrical conductivity was determined by conducting impedance measurement

at 20 Hz – 5 MHz of frequencies (LCR meter GW Instek LCR-8105G) at 573–873 K under air atmosphere. The silver paste (SPI Supplies, USA) electrode was coated on both sides of the composite pellet. The impedance data were precessed by ZView software (embedded in a Corrtest Electrochemical Station). The conductivity was then calculated by Eq. (1).

$$s = \frac{l}{RA} \quad (1)$$

where s is conductivity, $\text{S}\cdot\text{cm}^{-1}$; R is resistance, Ω ; l is the thickness of pellet, cm; A is the area of active electrode, cm^2 .

The Arrhenius plot of conductivity *versus* temperature was conducted to analyze the activation energy of ionic conduction and estimates vacancies number based on its pre-exponential factor. The Arrhenius equation is described by Eq. (2).

$$s = s_0 \exp^{-E_a/kT} \quad (2)$$

where s_0 is the pre-exponential factor; E_a is the activation energy, eV; k is Boltzmann constant, $1.38 \cdot 10^{-23} \text{ m}^2 \cdot \text{kg} \cdot \text{s}^{-2} \cdot \text{K}^{-1}$; T is temperature, K.

3. Results and Discussion

To study the crystal structure of the prepared materials, their diffraction patterns were compared to standard diffraction of related crystal that was found from Inorganic Crystal Structure Database, ICSD. The diffraction pattern of the prepared SDC as described in Fig. 1 shows that the peaks are in agreement with the standard diffraction of SDC, ICSD #28792. Le Bail refinement shows that SDC was crystallized in cubic with space group of $Fm\bar{3}m$ and cell parameter 5.42524(2) Å and cell volume 159.68(2) Å³. The Le Bail plot is depicted in Fig. 1b, and the cell parameters are listed in Table 1.

The diffraction pattern of YSZ is depicted in Fig. 2a, in which the peaks are in agreement with YSZ standard diffraction data ICSD #75312. Le Bail refinement on the diffraction data shows that YSZ was crystallized in tetragonal structure with a space group of $P42/nmcS$. The cell parameters are listed in Table 1, and the Le Bail plot is depicted in Fig. 2b. XRD patterns of the prepared composite at various weight ratios are depicted in Fig. 3. The XRD pattern consists of some characteristic peaks of SDC and YSZ without the availability of new peaks. It indicates that there was no solid reaction occurred during the heating process. The Le Bail refinement also shows parameter increasing only for few cells, *i.e.*, 0.00676 Å when 10 wt % of SDC was added to YSZ. The increasing is less than the diameter of Y^{3+} (radius: 1.04 Å) or Zr^{4+} (radius 0.86 Å). It means that Y^{3+} or Zr^{4+} did not dope into SDC structure and substitute samarium ions or cerium ions. A slight change in the cell parameters is possible because of

interaction between ions. The interaction influences the bond length of compound in the crystal. It indicates that sintered temperature of 1523 K is still suitable for SDC-YSZ composite without any concerns regarding third phases formation. Higher temperature might cause third phases formation as it was found by Mishima *et al.* [18] at 1673 K between SDC and YSZ. The interaction produced fluorite phase of $ZrO_2-CeO_2-Y_2O_3-Sm_2O_3$. A chemical reaction was found to occur at the lower temperature, *i.e.* 1473 K, for gadolinium-doped ceria with YSZ due to diffusion of Ce into YSZ [22]. If the particle size of YSZ and doped ceria are nanosized (<100 nm), the chemical interaction could occur even at lower temperature, *i.e.* 1173 K [23]. Therefore, this research prefers to sinter the composite at 1523 K to prevent third phases production, and it was proven that the third phase does not exist in the sintered composite. The particle size of the composite is also around 400 nm as described by SEM image in Fig. 4. Therefore, the reactivity is less than the nanosize particle (<100 nm) such as studied for nano YSZ and nano doped ceria [22].

The Le Bail refinement shows that the addition of YSZ did not change the crystal structure of SDC.

However, it changed the cell parameters ($a = b = c$) from 5.42524(2) Å to 5.432(1) Å, when it was mixed with 90 wt % of YSZ. Meanwhile, the cell volume changes from 159.68(2) Å³ to 160.24(7) Å³. It indicates that the interaction between atomic ions of YSZ and SDC affected the ionic bonding and expanded the bond length between atomic ions in the crystal.

Morphological analysis of the prepared composite shows that the diameters of 8YSZ granules are less than 0.4 µm. The density of SDC:YSZ (1:1), SDC:YSZ (1:9) and SDC:YSZ (9:1) are 3.10; 2.84; and 3.90 g·cm⁻³, respectively. Meanwhile, the density of SDC and YSZ are 6.05 and 5.303 g·cm⁻³, respectively. It indicates that the composites are more difficult to be sintered. It probably requires sintering temperature at above 1523 K. However, the cerium-based oxide might be decomposed at such high temperature, as Kungas *et al.* did [24] by firing the layered composite of SDC-YSZ at 1473 K. The morphological images are depicted in Fig. 4. The SEM image shows that the temperature of 1523 K did not sinter well the composite, as the grains are in a freely spherical form. Elemental analysis by EDX as shown in Fig. 5, confirms that the SDC-YSZ composite contains some elements as designed.

Table 1

The cell parameters of SDC, YSZ and SDC-YSZ composite

Cell parameters	SDC	YSZ	SDC:YSZ composite (1:9)		SDC:YSZ composite (1:1)		SDC:YSZ composite (9:1)	
	SDC cubic $Fm3m$	YSZ cubic $Fm3m$	SDC cubic $Fm3m$	YSZ cubic $Fm3m$	SDC cubic $Fm3m$	YSZ cubic $Fm3m$	SDC cubic $Fm3m$	YSZ cubic $Fm3m$
a (Å)	5.42524(2)	5.1421(3)	5.432(1)	5.1389(5)	5.4325(7)	5.1400(6)	5.433(0)	5.137(2)
b (Å)	5.423524 (2)	5.1421(3)	5.432(1)	5.1389(5)	5.4325(7)	5.1400(6)	5.433(0)	5.137(2)
c (Å)	5.423524(2)	5.1421(3)	5.432(1)	5.1389(5)	5.4325(7)	5.1400(6)	5.433(0)	5.137(2)
V (Å ³)	159.68(2)	135.97(1)	160.24(7)	135.71(2)	160.33(3)	135.79(3)	160.37(0)	135.54(8)
Rp (%)	5.47	3.72	3.49		3.44		4.11	
Rwp (%)	4.78	2.47	3.24		3.34		4.34	

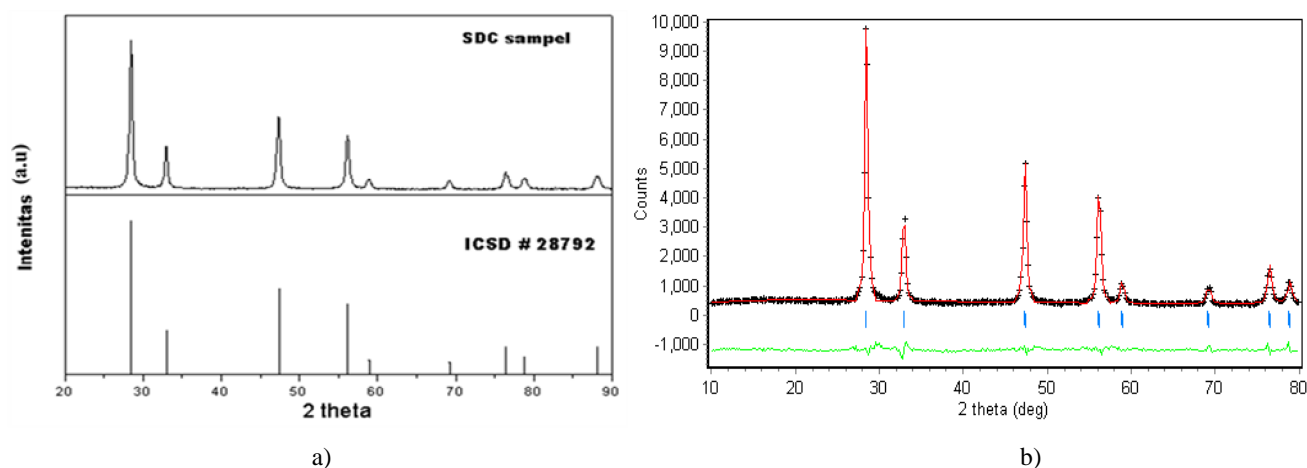


Fig. 1. XRD pattern of the prepared SDC and its comparison to standard SDC pattern ICSD #28792 (a) and the Le Bail plot of SDC diffraction data (b)

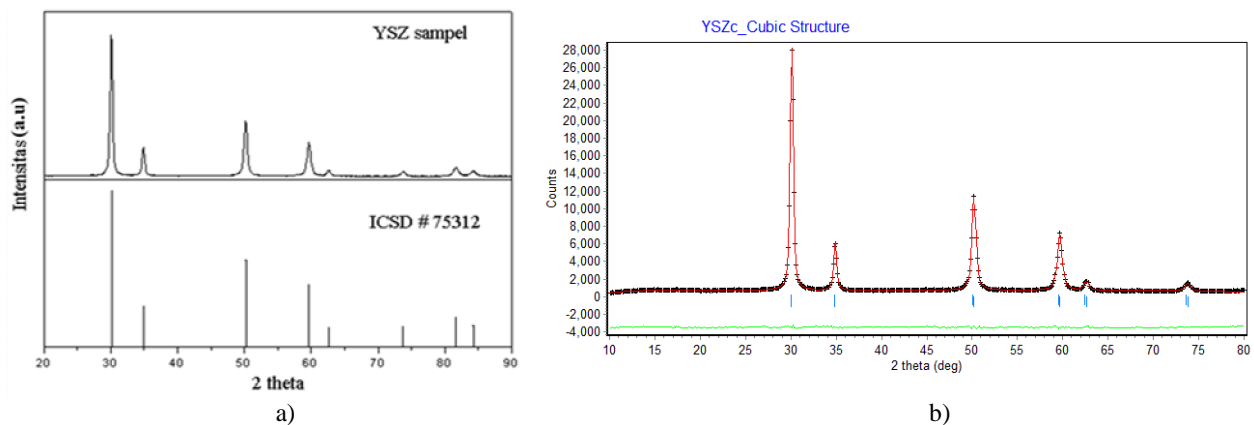


Fig. 2. XRD pattern of the YSZ and its comparison to standard YSZ pattern ICSD# 75312 (a) and the Le Bail plot of YSZ diffraction data (b)

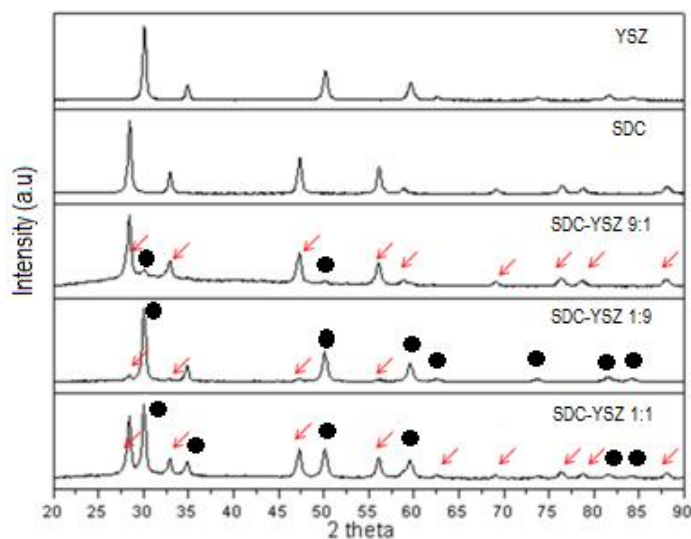


Fig. 3. XRD patterns of SDC-YSZ composite at various composition and its comparison to single SDC and single YSZ. The arrows refer to SDC peaks and circles – to YSZ peaks

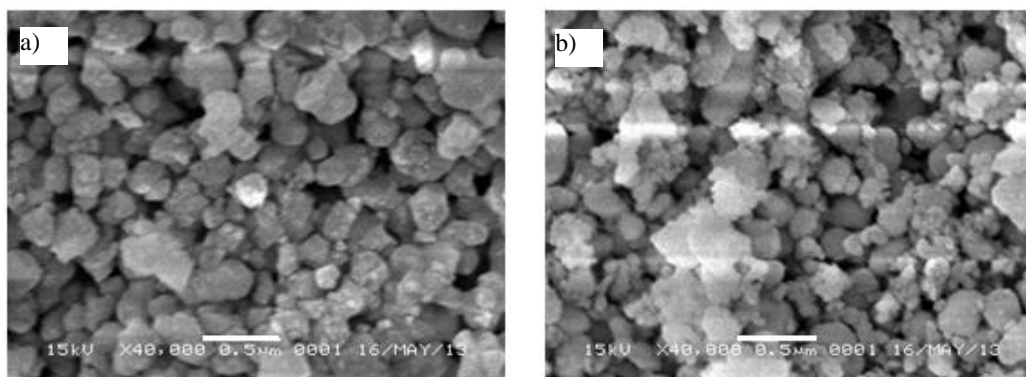


Fig. 4. SEM images of SDC-8YSZ (a) and 8YSZ (b)

Impedance data was fitted with R-C network model. The fitting produces resistance value can be converted into conductivity by applying Eq. (1). The impedance plots at 773 K temperature and its R-C network of the prepared materials is depicted in Fig. 6. The model consists of two R-C networks attributed to grain and grain boundary resistance which are overlapped. Capacitance value indicates grain or bulk resistance at $C \sim 0.1$ pF and grain boundary resistance at $C \sim 14$ nF [25]. The conductivity values are listed in Table 2. SDC has higher ionic conductivity than YSZ and SY91 at 773 K. However, at 873 K the conductivity provided by SDC is only electrode-electrolyte conduction at capacitance of $5.94 \cdot 10^{-4}$ F. Capacitance at ~ 0.1 μ F is attributed to electrode-bulk interface resistance [25]. The electrode resistance present at low frequency contains information of electronic leakage of the material [26] as the silver layer and platinum wire act as blocking electrode for oxygen ions because oxygen ions can not penetrate the dense silver and platinum.

As the impedance measurement was conducted under air atmosphere, the ions that migrate inside the composite electrolyte are oxygen ions produced by the reduction of O_2 to O^{2-} . The conductivity values are listed in Table 2. The result shows that SDC has the highest ionic conductivity at 573, 673 and 773 K. However, at 873 K only electrode-bulk interface resistance is provided. It indicates that at 873 K SDC is not appropriate to be used as electrolyte, due to electronic migration since electrode-bulk interface may cause a short circuit that will drop the fuel cell performance soon. On the other hand,

YSZ shows an ionic conductivity of $6.0 \cdot 10^{-4}$ S \cdot cm $^{-1}$ at 873 K. Meanwhile, SY91 is found to have higher ionic conductivity at 873 K, *i.e.*, $1.166 \cdot 10^{-3}$ S \cdot cm $^{-1}$ without any electrode-bulk interface resistance was detected. This indicates that the SY91 is more appropriate as electrolyte at 873 K than YSZ and SDC itself because YSZ can inhibit the SDC auto-oxidation. The ionic conductivity value is also higher than YSZ-SDC at similar ratio of 9:1 that was produced by glycine-nitrate process. The ionic conductivity is $2.49 \cdot 10^{-4}$ S \cdot cm $^{-1}$ [19] at 873 K. Meanwhile, the Arrhenius plot provides conductivity of $8.2 \cdot 10^{-3}$ S \cdot cm $^{-1}$ at 973 K and the activation energy of 1.09 eV [19].

Arrhenius plots (Fig. 7) at various temperature shows relation between temperature and ionic conductivity. Linear regression of each curve confirmed slope value, which correlates to the activation energy, E_a , as depicted in Eq. (2). The values of E_a and the pre-exponential factors are listed in Table 3. The E_a value of SDC is 0.79 eV, indicating the easiness of ions to migrate inside the SDC structure. However, the pre-exponential factor is only 3.48. The pre-exponential value might indicate the number of charge carriers [27]. However, it is also affected by some other factor, such as cell parameter, as it can be seen in Eq. (3).

$$m_i = \frac{va^2q}{4kT} \exp(-E_a/kT) \quad (3)$$

where v is the frequency, Hz; a is the cell parameter; T is the temperature, K; μ_i is the ionic mobility, m 2 \cdot s $^{-1}$ \cdot V $^{-1}$ [28].

Table 2

The electrical conductivity extracted from impedance fitting, the electrode-electrolyte conductivity and the calculated ionic conductivity of the prepared materials at various temperatures

Materials	T , K	Ionic conductivity, S \cdot cm $^{-1}$	Electrode-electrolyte conductivity, S \cdot cm $^{-1}$
SDC-YSZ11	573	$2.447 \cdot 10^{-7}$	–
	673	$3.501 \cdot 10^{-6}$	–
	773	$9.962 \cdot 10^{-6}$	–
	873	$5.862 \cdot 10^{-4}$	–
SDC-YSZ19	573	$9.506 \cdot 10^{-9}$	–
	673	$1.127 \cdot 10^{-6}$	–
	773	$1.739 \cdot 10^{-5}$	–
	873	$1.004 \cdot 10^{-4}$	–
SDC-YSZ91	573	$2.750 \cdot 10^{-7}$	–
	673	$6.803 \cdot 10^{-6}$	–
	773	$1.340 \cdot 10^{-4}$	–
	873	$1.166 \cdot 10^{-3}$	–
SDC	573	$3.209 \cdot 10^{-6}$	–
	673	$4.140 \cdot 10^{-5}$	–
	773	$2.020 \cdot 10^{-4}$	–
	873	–	$1.670 \cdot 10^{-5}$
YSZ	573	$2.440 \cdot 10^{-7}$	–
	673	$1.080 \cdot 10^{-5}$	–
	773	$1.770 \cdot 10^{-4}$	–
	873	$6.000 \cdot 10^{-4}$	–

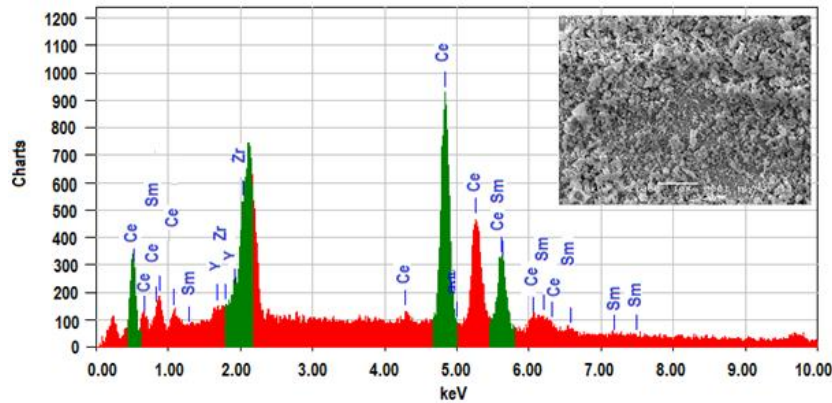


Fig. 5. EDX analysis of SDC-YSZ composite (9:1)

Table 3

The activation energy of ionic migration, activation energy and the pre-exponential factor

Materials	E_a , eV	Pre-exp factor	Linear regression coefficient R
SDC	0.79	3.48	0.995
YSZ	1.37	12.24	0.990
SDC-YSZ 1:9	1.34	8.91	0.986
SDC-YSZ 1:1	1.02	5.17	0.863
SDC-YSZ 9:1	1.38	12.61	0.950

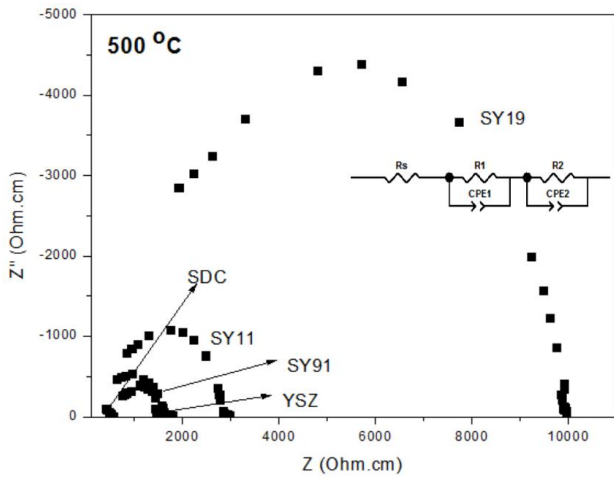


Fig. 6. The impedance plots of the prepared materials at 773 K and its resistor-capacitor network model. CPE is constant phase element

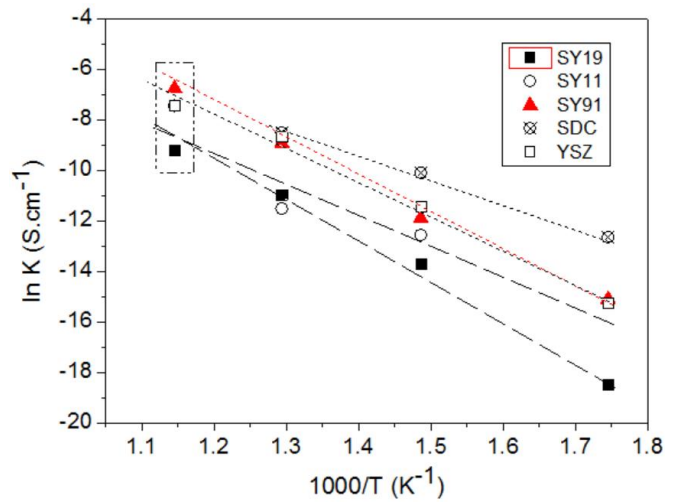


Fig. 7. Arrhenius plot of conductivity at various temperatures

4. Conclusions

The addition of YSZ into SDC does not change the crystal structure of SDC. However, it increases the cell parameters and the cell volume. Composite SDC-YSZ (9:1) shows the highest ionic conductivity at 873 K, i.e., $1.166 \cdot 10^{-3} \text{ S}\cdot\text{cm}^{-1}$ without any electrode-bulk interface conduction. It indicates that the presence of YSZ eliminated the electrode-electrolyte conduction at 873 K.

Acknowledgements

This research is part of Hibah Penelitian Unggulan UNS 2017, which was funded by Sebelas Maret University, contract No. 623/UN27.21/PP/2017. This research was also supported by the Ministry of Research, Technology and High Education, Republic of Indonesia. Authors express gratitude for the support.

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Received: August 11, 2017 / Revised: September 11, 2017 /

Accepted: January 19, 2018

ОДЕРЖАННЯ ЗА РЕАКЦІЮ У ТВЕРДОМУ СТАНІ КОМПЗИТУ ПРОМОТОВАНОГО САМАРІЄМ ОКСИД ЦЕРІЮ/ДІОКСИД ЦИРКОНІУ, СТАБІЛІЗОВАНОГО ОКСИДОМ ІТРІУ

Анотація. Одержано композити з різним співвідношенням промотованого самарієм оксиду церію (SDC) з діоксидом цирконію, стабілізованого оксидом ітрію (YSZ), SDC:YSZ = 0:1; 1:9; 1:1; 9:1 та 1:0. Досліджено структуру кристалів та йонну провідність одержаних композитів. За допомогою рентгенодифракційного аналізу з використанням методу Ле Бейля досліджено кристалічну структуру, просторові групи, параметри та об'єм комірок. Йонну провідність визначено методом імпедансу. Показано, що композити SDC-YSZ кристалізувались у двох фазах кубічного SDC і кубічного YSZ за відсутності вторинних фаз. Це вказує на те, що змішування та нагрівання за 1523 K не змінюють кристалічну структуру, навіть при зміні параметрів комірки. Композит SDC-YSZ 9:1 показує найвищу йонну провідність за 873 K. Показана роль YSZ щодо гальмування автовідновлення йонів церію.

Ключові слова: композит SDC-YSZ, електроліт, твердооксидний паливний елемент, йонна провідність.