[JKSA] Submission Acknowledgement

Eksternal | Kotak Masuk ×

Adi Darmawan <adidarmawan@live.undip.ac.id>
kepada saya

Min, 7 Agu 2022, 15.08

Inggris > Indonesia | Terjemahkan pesan | Nonaktifkan untuk: Inggris ×

Dear Ph.D adi Subardi

Thank you for submitting the manuscript, "Enhanced SOFC Cathode Performance Through Surface Modification NdBa0.5Sr0.5Co2O5+δ Nanoparticles" to Jurnal Kimia Sains dan Aplikasi. With the online journal management system that we are using, you will be able to track its progress through the editorial process by logging in to the journal web site:

Manuscript URL: https://ejournal.undip.ac.id/index.php/ksa/author/submission/48147
Username: subardi

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[JKSA] Editor Decision - your submission needs to be revised

Eksternal | Kotak Masuk ×

Adi Darmawan <adidarmawan@live.undip.ac.id>
kepada saya

Rab, 12 Okt 2022, 13.53

Inggris > Indonesia | Terjemahkan pesan | Nonaktifkan untuk: Inggris ×

Dear Ph.D adi Subardi

We have reached a decision regarding your submission to Jurnal Kimia Sains dan Aplikasi, "Enhanced SOFC Cathode Performance Through Surface Modification NdBa0.5Sr0.5Co2O5+δ Nanoparticles".

Our decision is that you need to revise this manuscript according to the feedback from the review.

How to prepare a revised manuscript.
(1) Carefully read reviewer(s)' comments and make appropriate revisions.
(2) Revised parts should be highlighted by changing the background color or the color of the words. The revised manuscript is in .docx format. Make sure that the revised manuscripts are cleared from the reviewer comments (none of the reviewers' comments are in the revised manuscript)

(2) Revised parts should be highlighted by changing the background color or the color of the words. The revised manuscript is in .docx format. Make sure that the revised manuscripts are cleared from the reviewer comments (none of the reviewers' comments are in the revised manuscript)

* How to prepare a response letter (a reply letter to the reviewer's question)*
(1) Copy all the comments given by the reviewers.
(2) Respond each comment, question, or suggestion one by one. The answers to the reviewer questions is in docx format. An example of the answers to the reviewer questions is attached.
(3) Indicate changes you make in the manuscript.

* How to upload your revised manuscript. *
(1) The revised manuscript and the response letter should be put together by compressing it into a zip file
(2) Login JKSA through your OJS account
(3) Find your active manuscript listed under "review" and click it
(4) Click "In Review" listed under "Status"
(5) Upload your file through "Upload Author Version" under "Editor Decision"

Please revise this manuscript no more than 3 (three) weeks from now. If within a month you do not revise this manuscript, then your manuscript will be treat as a new submission.

Regards

Adi Darmawan
Chemistry Department, Faculty of Sciences and Mathematics, Diponegoro University
Jl. Prof. Soedarto, SH., Tembalang, Semarang
Phone +6282221219817
adidarmawan@live.undip.ac.id

Editor comment:
Please cite articles published by JKSA

Reviewer A:

This manuscript should be minor revised before considered for publication. There are a few grammatical and a lot of punctuation mistakes in the manuscript which need to be corrected. Besides, the following correction should be incorporated: -

Introduction
• In text citation and references need correction according to the journal requirements.
• For the introduction, Describe more on the environmental issues together with treatment possibility and methods
• Further highlight novelty in last paragraph and the work carried out in this paper.

Experimental
• Repetitive punctuation error (please choose the '°C' carefully throughout the whole manuscript)
• Take note of unit spacing and citation spacing issue

Results and Discussion
• Authors may include the activation energy for the electrical conductivity analysis since the NBSC showed both metallic and semiconductor behaviour and explain a bit more on these behaviour.
• Please refer to the reference style on merging the in text citation styles.
• 3.2(2nd paragraph). NBSC+0.5 the same as NBSC+0.5M SDC? If the materials name was abbreviated, please specify first.

Kindly improve to include in more concise and significant results. Author should include some present challenges and possible routes to improve them. Describe them in more details.

Reviewer B:
Overall, the manuscript presents a good work. However, it requires some amendment and justifications. Please strictly recheck the English. To further improve the quality and content of this manuscript please refer to the specific comments as follows:

Title:
Please add 'of' after Modification.

Abstract:
It seems OK but it can be further improved.

- English needs to be revised.
- Paragraph 1, line 1: "SOFCs are the most…" 🖾 Solid oxide fuel cells (SOFCs) are the most…
- Paragraph 2, lines 5 – 8: The sentence seems incomplete. Please rewrite.
- There is no connection between paragraph 2 and paragraph 3. It is suggested to include some explanations on TEC in paragraph 2 before proceeding with TEC problem in the first sentence of paragraph 3.
- Research gap or novelty of this work is unclear. Please justify.

2. Experimental
It seems OK but it can be further improved.
- English needs to be revised.
- Section 2.1, paragraph 1, line 1: NBC or NBSC? Please also recheck in Paragraph 2, line 2 and throughout the manuscript.
- Section 2.1, paragraph 1, line 4: "The Ce(NO3)3.6H2O and SmCe(NO3)3.6H2O were.." 🖾 Please recheck. Ce and SmCe??
- Section 2.1: Is there any reason why 0.5 M? Please justify.
- Section 2.2: Is there any reason why stability testing was carried out for 96 h at 600 °C? Why not for 100 h? Please justify.
- The AC impedance measurement was conducted in what condition or environment? Gas flow rate? Please justify.

3. Results and Discussion
They seem OK but they can be further improved.
- English needs to be revised.
- Section 3.1: at 302 °C, 552 – 800 °C, 611 – 663 °C. At what interval did you measure the electrical conductivity from 300 – 800 °C? 1 °C (310, 302,

303.… 800)? 50 °C (300, 350, 400 …)? It is quite doubtful. Please justify.

- You did mention in the introduction that NBSC is a mixed ionic and electronic conductor (MIEC). From Figure 1, could you please describe which part is ionic conduction and which part is electronic conduction. You may refer to this article: Journal of Sol-Gel Science and Technology (2018) 86:617–630.
- Section 3.2, paragraph 1, line 6: Please write properly. 0.5 M not 05. Please recheck throughout the manuscript.
- Section 3.2, paragraph 2, lines 3 – 4: "The Rp value decline was primarily due to the additional SDC|NBSC+0.5 phase limit." How can you confirm that it is already the phase limit since you did not vary the amount of SDC. You only employed 0.5 M SDC. Please justify.
- Section 3..2, paragraph 2, lines 4 – 6: "Gas-phase molecules can easily migrate into the SDC|NBSC+0.5 interlayer mainly to the produced nano-sized SDC particles on the very porous NBSC surface cathode." What is the size of SDC particles? What is the porosity value of the very porous NBSC surface cathode?
- Section 3.2: "The cathode delamination of the electrolyte can be one of the possible causes of the increased Rp at the beginning of rapid degradation." Any SEM image to show the microstructure of the symmetrical cell after 2 h and 96 h stability testing to prove the delamination?
- Figure 6: Please also include SEM image at interface of SDC|NBSC-0.5 SDC symmetrical cell.

4. Conclusions

- It seems too general. Please rewrite.
- English needs to be revised.

References

They seem OK.

Jurnal Kimia Sains dan Aplikasi

Reviewer A:

This manuscript should be minor revised before considered for publication. There are a few grammatical and a lot of punctuation mistakes in the manuscript which need to be corrected. Besides, the following correction should be incorporated: -

Introduction

• In text citation and references need correction according to the journal requirements.

Reply: Thanks for the suggestion, we have revised this section

• For the introduction, Describe more on the environmental issues together with treatment possibility and methods

Reply:

Despite the fact that there are still plenty of fossil fuels available, important technological advancements have been made, and demand on ecological environmental preservation is increasing [1]. Fuel cells (FC) and hybrid electric vehicles play a major role in decreasing carbon dioxide pollutions. By using the H_2 generated by this process, FC vehicles (FCVs) can reduce CO_2 emissions in this sector to 80% compared to traditional vehicles [2]. In a global effort to overcome the dangers of climate change, cogeneration systems, especially fuel cells, are becoming massive due to of their excellent energy efficiency [3].

• Further highlight novelty in last paragraph and the work carried out in this paper.

Reply: Thank you and we have added highlight novelty as follows

Novelty this work is the cathode design using double perovskite oxide doped with 0.5 M electrolyte material.

Experimental

• Repetitive punctuation error (please choose the '°C' carefully throughout the whole manuscript)

Reply:

Thank you for the suggestion, it has been revised

• Take note of unit spacing and citation spacing issue

Reply:

Thank you for the suggestion, it has been revised

Results and Discussion

• Authors may include the activation energy for the electrical conductivity analysis since the NBSC showed both metallic and semiconductor behaviour and explain a bit more on these behaviour.

Reply:Thanks for the confirmation, the energy activation value for the NBSC cathode has been added.

As previously reported that the activation energy (Ea) of the NBSC cathode polarization resistance from the Ln(R) vs. 1000/T is 102.5 kJ mol^{-1}

• Please refer to the reference style on merging the text citation styles.

Reply: Thank you for the suggestion.

• 3.2(2nd paragraph). NBSC+0.5 the same as NBSC+0.5M SDC? If the materials name was abbreviated, please specify first.

Reply: Thank you, the abbreviation used is LBSC+0.5 M SDC and has been written the same throughout the article.

• Did all the Nyquist plot was fitted? If yes, please include in the caption and please include the circuit used for the fitting.
Reply: Yes, we have added the circuit used for the fitting as follows:

The resistance of the evaluation cell was demonstrated using an analogous circuit of the impedance curve and was fitted using Z-View based on the following series circuit R_1 (R_2-CPE_1) R_3-CPE_2). The R is same as to Ohmic resistance (R_Ω), and the resistance of polarization is characterized by two resistances (R_2+R_3). A constant phase element (CPE) symbolizes a non - ideal capacitor, such as the double layer at a nonplanar TPB, and the n parameter correlated with the CPE equivalent to a real capacitor, where n=1.

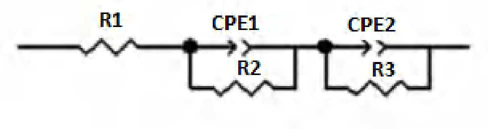
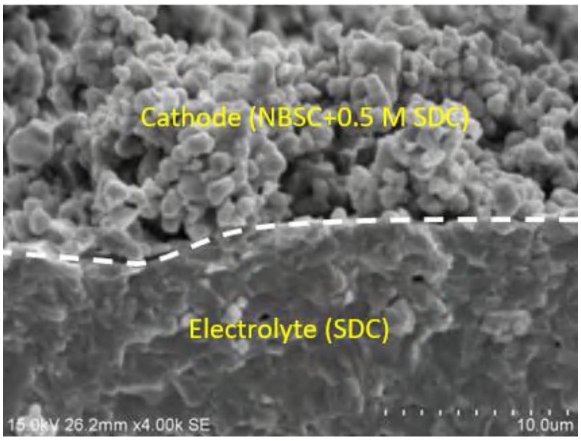


Figure 4. (a)-(c) Nyquist diagram of impedance spectroscopic symmetric cell NBSC+0.5 M SDC|SDC|NBSC+0.5 M SDC in the OPP range between 0.112–0.019 atm at various temperatures and (d) Equivalent circuit used to fit the impedance spectra

• Figure 6.
Reply: It has revised

• Please elaborate further on the 'adhesion of the two layers (cathode and anode) looks strong'. Please include the cross section area if author wish to discuss on the adhesion of two components. Author may improve the image by highlighting which are what.
Reply: Thanks for the suggestion, the cross-section area for the two layers (cathode and electrolyte) has been added. In the half cell sample, the two layers that are connected are the cathode and the anode. Meanwhile, the cathode layer is not connected to the anode.

The microstructure of the NBSC+0.5 M SDC cathode surface and cross-sectional microstructure of the two layers (cathode and electrolyte) are presented in Figure 6.



(b)

Figure 6. SEM images: (a) The cathode surface and (b) Cross-sectional microstructure of the two layers (cathode and electrolyte).

• There are agglomerations occurred. Please explain further. The NBSC images was analysed after the EIS analysis or before? If after, some reaction that lead to the agglomeration.
Reply: Thank you for the suggestion. The explaination has been added as follow:

> After testing, the surface of the cathode sample experienced agglomeration. The microstructure growth, such as grain expansion or particle coarsening, is critical since a practical SOFC operates at high temperatures (at T = 800-1000 °C) [33]. With increasing temperature and current density, as well as the duration of the test, the microstructure gets coarser and denser [34].

• Porosity of the NBSC?
Reply: Yes, the requirements for a SOFC cathode are physically high porosity. Good morphology promotes rapid oxygen diffusion, minimizes polarization resistance, and improves current collection.

• Is the caption correct? NBSC or NBSC+0.5M SDC?
Reply: The microstructure sample is a cathode that has been doped with 0.5 M SDC, namely NBSC+0.5 M SDC

• The SEM image explanation does not seem relatable to the EIS analysis. Please re-arrange the flow of the characterizations for the SEM if needed.
Reply: Thank you for the suggestion, it has been added [3.3. last paragraph].

> The limit for good performance solid oxide fuel cell (SOFC) value is Rp <0.15 $\Omega.cm^2$). Testing symmetrical cells in the OPP range of 0.214–0.0027 atm obtained relatively small RP values between 0.030 $\Omega.cm^2$–0.039 $\Omega.cm^2$ at 800°C. The surface morphology of the NBSC+0.5 M SDC sample corresponds to the physical characteristics of the SOFC cathode

Conclusion

Kindly improve to include in more concise and significant results. Author should include some present challenges and possible routes to improve them. Describe them in more details.
Reply: Thank you for the suggestion, it has been revised.

Reviewer B:
Overall, the manuscript presents a good work. However, it requires some amendment and justifications. Please strictly recheck the English. To further improve the quality and content of this manuscript please refer to the specific comments as follows:

Title:
Please add 'of' after Modification.
Reply: Thank you for the suggestion, It has been added.

Abstract:
It seems OK but it can be further improved.
- English needs to be revised.
  Reply: Thanks for the advice, I tried to revise the english

- The electrical conductivity was measured at what temperature? (Please include in the text)

  Reply: Thank you, the electrical conductivity test temperature has been added

> Figure 1 shows the relationship between temperature and the NBSC cathode electrical conductivity at $P(O_2)$ = 0.03 atm and 0.0032 atm in the range of 300°C and 800°C.

- Please also include important finding(s) on long term stability measurement.

Reply: Thank you for the suggestion, the long-term stability measurement findings have been added.

> The NBSC + 0.5 M SDC cathode sample has better long-term stability than NBSC, with a lower Rp value of 2.35.

Keywords:
They seem OK.

1. Introduction
It seems OK but it can be further improved.

- English needs to be revised.

  Reply: Thank you for the advice, We tried to revise the english.

- Paragraph 1, line 1: “SOFCs are the most…” ◊ Solid oxide fuel cells (SOFCs) are the most…

Reply: Thank you for the advice, It has been revised.

> Solid oxide fuel cells (SOFCs) are the most environmentally friendly electrical energy generator and effective electrochemical energy converter.

- Paragraph 2, lines 5 – 8: The sentence seems incomplete. Please rewrite.

Reply: Thank you for the suggestion, It has been revised.

> Cobalt-based mixed ionic-electronic conductors such as $LnBa_{0.5}Sr_{0.5}Co_{1.5}Fe_{0.5}O_{5+\delta}$ [8], $Pr_{0.5}Y_{0.5}BaCo_2O_{5+\delta}$ [9], $SmBaCo_{2-x}Ni_xO_{5+\delta}$ [10], $PrBaCo_{2-x}Mn_xO_{5+\delta}$, [11], $NdBa_{0.25}Sr_{0.75}CoCuO_{5+\delta}$, [12], $YBa_{0.5}Sr_{0.5}Co_{2-x}Fe_xO_{5+\delta}$, [13], $SmBa_{1-x}Sr_xCo_2O_{5+\delta}$ δ [14], $GdBa_{0.5}Sr_{0.5}Co_{1.5}Fe_{0.5}O_{5+\delta}$ [15], $YBa_{0.7}Sr_{0.3}Co_2O_{5+\delta}$ [16] have been published as promising materials for IT-SOFC cathodes.

- There is no connection between paragraph 2 and paragraph 3. It is suggested to include some explanations on TEC in paragraph 2 before proceeding with TEC problem in the first sentence of paragraph 3.

Reply: Thank you for the advice, It has been added.

> The coefficient of thermal expansion (TEC) of SOFC components (cathode, electrolyte anode, and interconnection material) must be similar (almost the same) in order to obtain efficient operation.

- Research gap or novelty of this work is unclear. Please justify.

Reply: Thank you for the confirmation. Regarding the novelty has been added [The last paragrap of Introduction]

Novelty in this work is the cathode design using double perovskite oxide doped with 0.5 M electrolyte material.

2. Experimental

It seems OK but it can be further improved.

- English needs to be revised.

Reply: Thank you for the advice, We tried to revise the english.

- Section 2.1, paragraph 1, line 1: NBC or NBSC? Please also recheck in Paragraph 2, line 2 and throughout the manuscript.

Reply: Thank you for the correction, NBSC has been used and has been revised throughout the manuscript

- Section 2.1, paragraph 1, line 4: "The Ce(NO3)3.6H2O and SmCe(NO3)3.6H2O were.." ◊ Please recheck. Ce and SmCe??

Reply: Thank you for the correction, The correct materiel is Sm not SmCe, it has been revised.

The $Ce(NO_3)_3.6H_2O$ and $Sm(NO_3)_3.6H_2O$ were used to synthesize the electrolyte powder SDC as a precursors material.

- Section 2.1: Is there any reason why 0.5 M? Please justify.

Reply: Thank you, the determination of the doping level of 0.5 M SDC is based on previous research, described as follows:

Considerations regarding the doping content of 0.5 M SDC refer to the results of our previous research, the title of the article **"Electrochemical and thermal properties of SmBa0.5Sr0.5Co2O cathode impregnated with $Ce_{0.8}Sm_{0.2}O_{1.9}$ nanoparticles for intermediate temperature solid oxide fuel cells, International Journal of Hydrogen Energy, 42 (2017) 24338-24346".** There are three different samples with doping levels of 0.13 M SDC, 0.39 M SDC, and 0.65 M SDC. The highest SOFC performance (The highest peak power density) was obtained in a sample with a doping of 0.65 M SDC of 823 $mWcm^{-2}$. Until now, the optimization of SDC doping contents is still being developed at SOFC cathodes. Related to this, doping with 0.5 M SDC content in NBSC alloys is part of an effort to optimize SDC doping levels to obtain maximum SOFC performance.

- Section 2.2: Is there any reason why stability testing was carried out for 96 h at 600 °C? Why not for 100 h? Please justify.

Reply: Thank you for your confirmation. Long-term testing was carried out for four days for 96 hours.

- The AC impedance measurement was conducted in what condition or environment? Gas flow rate? Please justify.

Reply: Thank you for the suggestion and it has been added.

The symmetric cell test was also carried out under various atmospheric pressure $P(O_2)$ = 0.112 atm–0.019 atm at a temperature between 600°C and 800°C.

3. Results and Discussion

They seem OK but they can be further improved.

- English needs to be revised.

Reply: Thank you for the advice, We tried to revise the english.

- Section 3.1: at 302 °C, 552 – 800 °C, 611 – 663 °C. At what interval did you measure the electrical conductivity from 300 – 800 °C? 1 °C (310, 302, 303…. 800)? 50 °C (300, 350, 400 …)? It is quite doubtful. Please justify.

Reply: Conductivity testing is carried out at temperatures from 300 to 800°C without interval or starting at 300, 301, 3002........800°C.

- You did mention in the introduction that NBSC is a mixed ionic and electronic conductor (MIEC). From Figure 1, could you please describe which part is ionic conduction and which part is electronic conduction. You may refer to this article: Journal of Sol-Gel Science and Technology (2018) 86:617–630.

Reply: Thank you for the suggestion, regarding the MIEC discussion has been added [3.1]

> Cobalt-containing perovskites are widely investigated because of their high electronic and oxide-ionic conductivity values. The conductivity value of this material exceeds perovskite oxide with other 3d transition metal ions [30]. Measurement of the NBSC cathode shows a relatively high electrical conductivity value above 100 S cm^{-1}, indicating that it is a *p*-type electronic conductor. The total value of the conductivity of the NBSC sample is a combination of the electronic ions conductivity and oxides caused by the existence of electron-hole pairs and oxygen vacancies. The ionic conductivity of the perovskite-type oxide material is significantly lower than the electronic conductivity. Therefore, it can be concluded that electronic conductivity dominates the conductivity value [31].

- Section 3.2, paragraph 1, line 6: Please write properly. 0.5 M not 05. Please recheck throughout the manuscript.

Reply: Thank you for the suggestion, It has been revised.

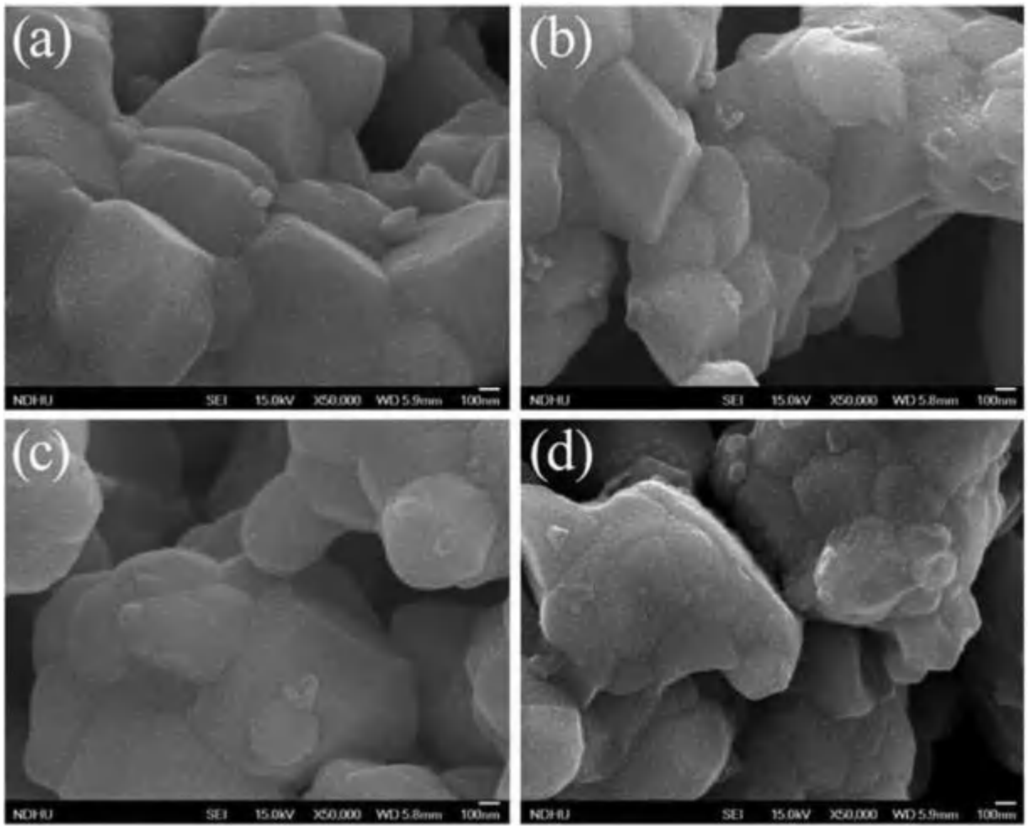
- Section 3.2, paragraph 2, lines 3 – 4: "The Rp value decline was primarily due to the additional SDC|NBSC+0.5 phase limit." How can you confirm that it is already the phase limit since you did not vary the amount of SDC. You only employed 0.5 M SDC. Please justify.

Reply: Thank you for your confirmation. Related to this discussion can be explained as follows. This sample consists of 2 types, namely NBSC [without SDC doping] and NBSC with 0.5 M SDC doping or NBSC+0.5 M SDC. So this study compared NBSC samples without SDC doping and NBSC with 0.5 M SDC doping. The doping impact of 0.5 M SDC into the NBSC alloy is quite significant to the value of Rp.

- Section 3..2, paragraph 2, lines 4 – 6: "Gas-phase molecules can easily migrate into the SDC|NBSC+0.5 interlayer mainly to the produced nano-sized SDC particles on the very porous NBSC surface cathode." What is the size of SDC particles? What is the porosity value of the very porous NBSC surface cathode?

Reply: Thank you for your confirmation. Related to this discussion can be explained as follows.
As previously reported by our team, SDC infiltration towards the $SmBa_{0.5}Sr_{0.5}Co_2O_{5+\delta}$ cathode shows SDC grains in the range of 10–35 nm as seen in the SEM micrographs below. SDC granules are evenly distributed homogeneously on the surface of the porous cathode resulting in good interconnection and improved electrochemical properties. The size of the porosity on the cathode surface is in the range of 0.5-1 µm. ***[Ref: Adi Subardi & Yen-Pei Fu, Electrochemical and thermal properties of*** $SmBa_{0.5}Sr_{0.5}Co_2O_{5+\delta}$ ***cathode impregnated with*** $Ce_{0.8}Sm_{0.2}O_{1.9}O_{5+\delta}$ ***nanoparticles for***

intermediatetemperature solid oxide fuel cells, International Journal of Hydrogen Energy, 42, (2027) 24338-24346].



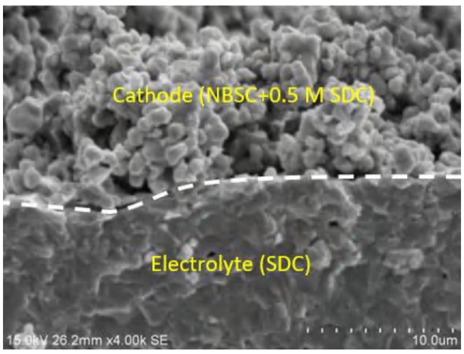
- Section 3.2: "The cathode delamination of the electrolyte can be one of the possible causes of the increased Rp at the beginning of rapid degradation." Any SEM image to show the microstructure of the symmetrical cell after 2 h and 96 h stability testing to prove the delamination?

Reply: Thank you for the confirmation. In general, the cathode delamination of the electrolyte occurs after testing the stability of the cell. Figure 6 (a), SEM shows the symmetrical cell microstructure after 96 hours of stability testing. Unfortunately, we do not have SEM images of the microstructure after 2 hours of testing.

- Figure 6: Please also include SEM image at interface of SDC|NBSC-0.5 SDC symmetrical cell.

Reply: Thank you for the suggestion, It has been revised.

The microstructure of the NBSC+0.5 M SDC cathode surface and cross-sectional microstructure of the two layers (cathode and electrolyte) are presented in Figure 6.



(b)

Figure 6. SEM images: (a) The cathode surface and (b) Cross-sectional microstructure of the two layers (cathode and electrolyte).

- English needs to be revised.

Reply: Thank you for the suggestion, It has been revised.

References

They seem OK.

____

[JKSA] [ID-48147] - Editor Decision - your submission is accepted Eksternal Kotak Masuk ×

Adi Darmawan <adidarmawan@live.undip.ac.id> Sel, 29 Nov 2022, 16.56

kepada saya

Inggris > Indonesia Terjemahkan pesan Nonaktifkan untuk: Inggris ×

Dear Ph.D adi Subardi

We have reached a decision regarding your submission to Jurnal Kimia Sains dan Aplikasi, "Enhanced SOFC Cathode Performance Through Surface Modification NdBa0.5Sr0.5Co2O5+δ Nanoparticles".

Our decision is to accept your submission. Congratulations

We are pleased to inform you that Jurnal Kimia Sains dan Aplikasi would like to publish your manuscript in our next issue. Before this manuscript proceeds to the layouting process, please pay attention to the following:

- Send a signed Copyright transfer agreement. You can download the Copyright transfer agreement in the Copyright notice section. Please fill in handwriting and sign and then scan and email us (email: jksa@live.undip.ac.id).
- Send a graphical abstract

Enhanced SOFC Cathode Performance Through Surface Modification of $NdBa_{0.5}Sr_{0.5}Co_2O_{5+\delta}$ Nanoparticles

Adi Subardi
Department of Mechanical Eng., Institut Teknologi Nasional Yogyakarta
Daerah Istimewa Yogyakarta 55281, Indonesia

Abstract
The cathode materials fabrication with outstanding performance and stability at intermediate temperatures of 600°C–800°C is required for the prospective mass production of solid oxide fuel cells (SOFCs). Infiltration is a potential method because it has proven successful in fabrication and cell performance enhancement. This study mainly focuses on the electrical conductivity and long-term reliability of cathode symmetric cells $NdBa_{0.5}Sr_{0.5}Co_2O_{5+\delta}$ (NBSC) fabricated by traditional solid-state reaction techniques. The electrical conductivity value of the cathode is in the range of 174–278 $S.cm^{-1}$. Impedance analysis showed that the infiltration of 0.5M SDC on the NBSC cathode surface dramatically reduced the polarization resistance (Rp) between layers (cathode-electrolyte) from 3.32 $\Omega.cm^2$ to 1.82 $\Omega.cm^2$ at 600°C or decreased by 45 % compared to NBSC cathode without 0.5M SDC infiltration. The enhanced stability of NBSC cathode specimens with 0.5M SDC infiltration (NBSC+0.5 M SDC) under SOFC operating conditions proves that samples with infiltration extend their lifetime. Compared to the NBSC cathode, the NBSC + 0.5 M SDC cathode has better long-term stability with a lower RP value of 2.35 $\Omega.cm^2$. In the OPP range of 0.214-0.0027 atm at 800°C, the relatively tiny Rp value of the symmetrical cell is between 0.030 $\Omega.cm^2$ and 0.039 $\Omega.cm^2$, below the 0.15 $\Omega.cm^2$ suitable performance limit for solid oxide fuel cells

Keywords: SOFC; Cathode; Electrical conductivity; Infiltration; Long-term performance stability

1. Introduction

Although plenty of fossil fuels are still available, critical technological advancements have been made, and the demand for ecological and environmental preservation is increasing [1]. Fuel cells (FC) and hybrid electric vehicles play a significant role in decreasing carbon dioxide pollution. By using the H_2 generated by this process, FC vehicles (FCVs) can reduce CO_2 emissions in this sector to 80% compared to traditional vehicles [2]. In a global effort to overcome the dangers of climate change, cogeneration systems, especially fuel cells, are becoming massive due to of their excellent energy efficiency [3].

Solid oxide fuel cells (SOFCs) are the most environmentally friendly electrical energy generator and effective electrochemical energy converter. The current weakness of SOFCs is the high operating temperature of 800°C–1000°C, which significantly affects cost, reliability, and application. The focus of SOFC development is efforts to reduce the working temperature of SOFC between 600°C to 800°C. Unfortunately, the low working temperature causes the SOFC electrode kinetics to decrease. Two essential strategies can be used to develop SOFCs today, namely (a) reducing ohmic resistance; (b) reducing the polarization resistance of the cathode [4, 5]. Cathode development continues to be carried out to improve electrode and cathode optimization as a factor that determines the overall SOFC performance [6].

Materials that provide ionic and electronic conduction (mixed ionic-electronic conductors, MIEC) can minimize polarization resistance [7]. Most oxide compositions with a perovskite structure show mixed conductivity in the medium n temperature range [8]. Cobalt-containing double perovskite-based cathodes exhibited excellent electrochemical activity and compatibility with SDC doping electrolytes [9, 10]. Cobalt-based mixed ionic-electronic conductors such as $LnBa_{0.5}Sr_{0.5}Co_{1.5}Fe_{0.5}O_{5+\delta}$ [11], $Pr_{0.5}Y_{0.5}BaCo_2O_{5+\delta}$ [12], $SmBaCo_{2-x}Ni_xO_{5+\delta}$ [13], $PrBaCo_{2-x}Mn_xO_{5+\delta}$, [14], $NdBa_{0.25}Sr_{0.75}CoCuO_{5+\delta}$, [15], $YBa_{0.5}Sr_{0.5}Co_{2-x}Fe_xO_{5+\delta}$, [16], $SmBa_{1-x}Sr_xCo_2O_{5+\delta}$ [17], $GdBa_{0.5}Sr_{0.5}Co_{1.5}Fe_{0.5}O_{5+\delta}$ [18], $YBa_{0.7}Sr_{0.3}Co_2O_{5+\delta}$ [19], and $YBaCo_2O_{5+\delta}$ [20] have been published as promising materials for IT-SOFC cathodes. The coefficient of thermal expansion (TEC) of SOFC components (cathode, electrolyte anode, and interconnection material) must be similar in order to obtain efficient operation.

One effective method to reduce the thermal expansion coefficient (TEC) and improve electrochemical performance is through the infiltration/impregnation method of electrolyte material into a porous cathode to obtain a composite cathode. Using composite cathodes in SOFC devices reduces TEC and expands the cathode layer's three-phase boundary zone (TPB) [21]. In this work, catalytically active $Ce_{0.8}Sm_{0.2}O_{1.9}$ (SDC) nanoparticles were infiltrated into the NBSC cathode to improve the catalytic activity of the SDC electrolyte-based electrode. The cathode properties of the NBSC were evaluated for electrical conductivity and electrochemical impedance spectroscopy during long-term performance testing. Novelty in this article is the cathode design using double perovskite oxide doped with 0.5 M electrolyte material. This work suggested that a double perovskite-based cathode material is appropriate for IT-SOFC applications.

2. Experimental

2.1. Preparation of cathode materials and electrolytes

The $NdBa_{0.5}Sr_{0.5}Co_2O_{5+\delta}$ (NBSC) cathode was fabricated using the solid-state reaction method. The precursors use high-grade materials Nd_2O_3, $SrCO_3$, $BaCO_3$, and CoO. The milled material was dried, crushed into a powder using a grinder, and then calcined for four hours at 1100°C in the air. The $Ce(NO_3)_3.6H_2O$ and $Sm(NO_3)_3.6H_2O$ were used to synthesize the electrolyte powder SDC as a precursor material. The precursor material was dissolved in distilled water, then added to the ammonia solution with this stoichiometric ratio. After the mixture's pH was adjusted to 9.5 to 10, coprecipitation powder was heated for two hours at 600 °C in the air. The reference [22] provides detailed instructions for SDC fabrication.

The infiltration technique procedures are detailed in reference [23, 24]. For fabrication of the required SDC nanoparticles in the NBSC porous cathode, symmetric cells impregnated with 0.5M SDC, hereinafter abbreviated as NBSC+0.5M SDC were heated at 900°C for 2 hours. The $Ce(NO_3)_3.6H_2O$ and $Sm(NO_3)_3.6H_2O$ were dissolved in deionized water to create an aqueous nitrate solution of the $Ce_{0.8}Sm_{0.2}(NO_3)$ precursor. The porous cathode was coated on either side with 0.5M SDC nanoparticle liquid using the microliter injector.

Table 1. Composition and Abbreviation of $NdBa_{0.5}Sr_{0.5}Co_2O_{5+\delta}$ Based Specimens

Composition	Abbreviation
$NdBa_{0.5}Sr_{0.5}Co_2O_{5+\delta}$	NBSC
$NdBa_{0.5}Sr_{0.5}Co_2O_{5+\delta}$+0.5 M SDC	NBSC+0.5 M SDC

2.2. Specimen Testing

The NBSC pellets measuring 5 x 5 x 10 mm^3 were sintered at 1200°C for 1 hour and were used as electrical conductivity test specimens. Electrical conductivity was investigated by the four-probe DC method, and the voltage response was recorded using a Keithley 2420 Source Meter at a temperature between 300°C and 800°C by applying a continuous current to the two current wires. For electrochemical testing of symmetrical cell samples, the NBSC-based cathode was utilized as the working electrode (WE). The Ag counter electrode (CE) was placed on the opposite side of the sintered SDC disk, and the reference electrode (RE) was fabricated 3–4 mm from the WE. Screen-printing was used to coat the NBSC-based cathodes on both sides of the SDC electrolyte, and they were further sintered in the air at 1000°C for four hours.

Cell stability testing was carried out for 96 hours without stopping at 600°C. The symmetric cell test was also carried out under various atmospheric pressure $P(O_2)$ = 0.112 atm–0.019 atm at a temperature between 600°C and 800°C. The applied frequency ranges from 100 kHz to 0.1 Hz with an AC amplitude signal of 10 mV. The AC impedance measurement calculated the current density value (io). The following formula adapted from the Bulter–Volmer formula was used to calculate the value of io, and the total cathode Rp was calculated from the intersection with the axis of the impedance loop [25].

$$io = \frac{RTv}{nFR_p}$$

Where F is Faraday's factor (F = 96500 C mol^{-1}), R is the ideal gas constant (R = 8,31 J mol^{-1} K^{-1}), The total electrons number transferred in the reaction is given by the notation n, and the number of times the rate-determining step begins in a whole reaction event is given by the notation m. For oxygen reduction reactions, n and m are commonly used to be 4 and 1.14, respectively. The comprehensive calculating process refers to reference [26].

3. Results and Discussion

3.1. NBSC Cathode Electrical Conductivity

Figure 1 shows the relationship between temperature and the NBSC cathode electrical conductivity at $P(O_2)$ = 0.03 atm and 0.0032 atm at 300°C to 800°C. It can be seen that at $P(O_2)$ = 0.03 atm (red line), at a temperature of 302°C it has reached the highest electrical conductivity value of 278 $S.cm^{-1}$. Defects in the Co-O-Co bond lattice cause the release of oxygen atoms from the lattice and the reduction of Co^{4+} to Co^{3+} or Co^{3+} to Co^{2+} to cause a significant drop in electrical conductivity beginning at 330°C. At 440°C the rate of decrease in conductivity slows down and forms a linear line from 552°C to 800°C. At $P(O_2)$ = 0.0032 (blue line), the reduction in electrical conductivity starts from the initial heating of 300°C to 603°C, forming a linear line.

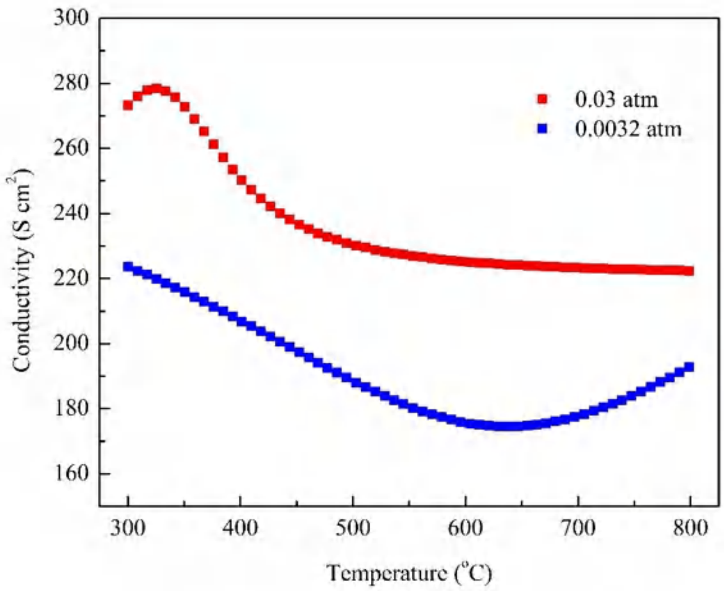


Figure 1. NBSC cathode conductivity at $P(O_2)$ = 0.03 atm and $P(O_2)$ = 0.0032 atm with respect to temperature

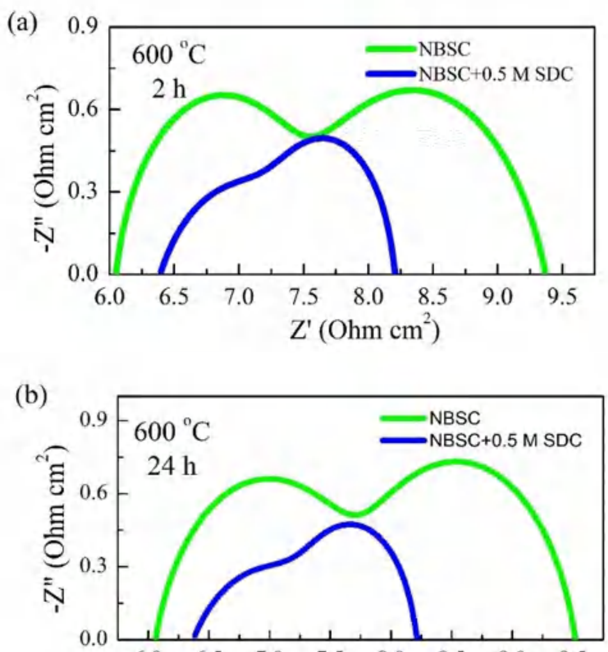
At 611oC–663oC, conductivity began to move slowly, increasing significantly up to 800oC. NBSC cathode conductivity values are 174-278 S.cm-1 and meet the requirements of SOFC cathode material [27]. The metallic conductivity properties of NBSCs can be related to several factors [19, 28, 29]: (1) energy bands overlap between Co-3d and O-2p; (2) the presence of Co^{4+} ions from the thermally affected Co^{3+} charge disproportion; and (3) loss of oxygen from the lattice at higher temperatures.

Cobalt-containing perovskites are widely investigated because of their high electronic and oxide-ionic conductivity values. The conductivity value of this material exceeds perovskite oxide with other 3d transition metal ions [30]. Measurement of the NBSC cathode shows a relatively high electrical conductivity value above 100 S cm^{-1}, indicating that it is a *p*-type electronic conductor. The total value of the conductivity of the NBSC sample is a combination of the electronic ions conductivity and oxides caused by the existence of electron-hole pairs and oxygen vacancies. The ionic conductivity of the perovskite-type oxide material is significantly lower than the electronic conductivity. Therefore, it can be concluded that electronic conductivity dominates the conductivity value [31]. As previously reported that the activation energy (Ea) of the NBSC cathode polarization resistance from the Ln(R) vs. 1000/T is 102.5 kJ mol^{-1} [32].

3.2. Symmetrical Cell Long-term Test

The specimens were evaluated using AC impedance spectroscopy under conditions of an open circuit. The symmetrical cell performance was examined to assess the NBSC long-term stability, and the polarization resistance (Rp) value was measured versus time in stationary air at 600 °C. Figure 2(a)-(e) shows NBSC cathodes with infiltration and without SDC infiltration and the Rp value as a function of time under stationary air as an oxidant in the 2 to 96 hours. The Rp value of NBSC+0.5 M SDC cathode decreased significantly from 3.32 Ω.cm^2 to 1.82 Ω.cm^2 or decreased by 45% compared to NBSC cathode without infiltration.

The Rp value of NBSC+0.5 M SDC cathode in this study is still better than that achieved by cathodes $GdBa_{0.5}Sr_{0.5}Co_{1.5}Fe_{0.5}O_{5+\delta}$ [18] and $YBa_{0.7}Sr_{0.3}Co_2O_{5+\delta}$ [19], which are 2.33 Ω.cm^2, and 3.29 Ω.cm^2, respectively. The Rp value decline was primarily due to the additional SDC|NBSC+0.5 M SDC phase limit. Gas-phase molecules can easily migrate into the SDC|NBSC+0.5 M SDC interlayer, mainly to the produced nano-sized SDC particles on the very porous NBSC surface cathode. The oxygen reduction reaction (ORR) activity in electrochemical sites is greatly increased under these conditions. The ORR is on the surface area of the NBSC cathode, simultaneously hitting the electrolyte and air. These newly formed SDC nanoparticles were deposited on the NBSC porous framework [24].



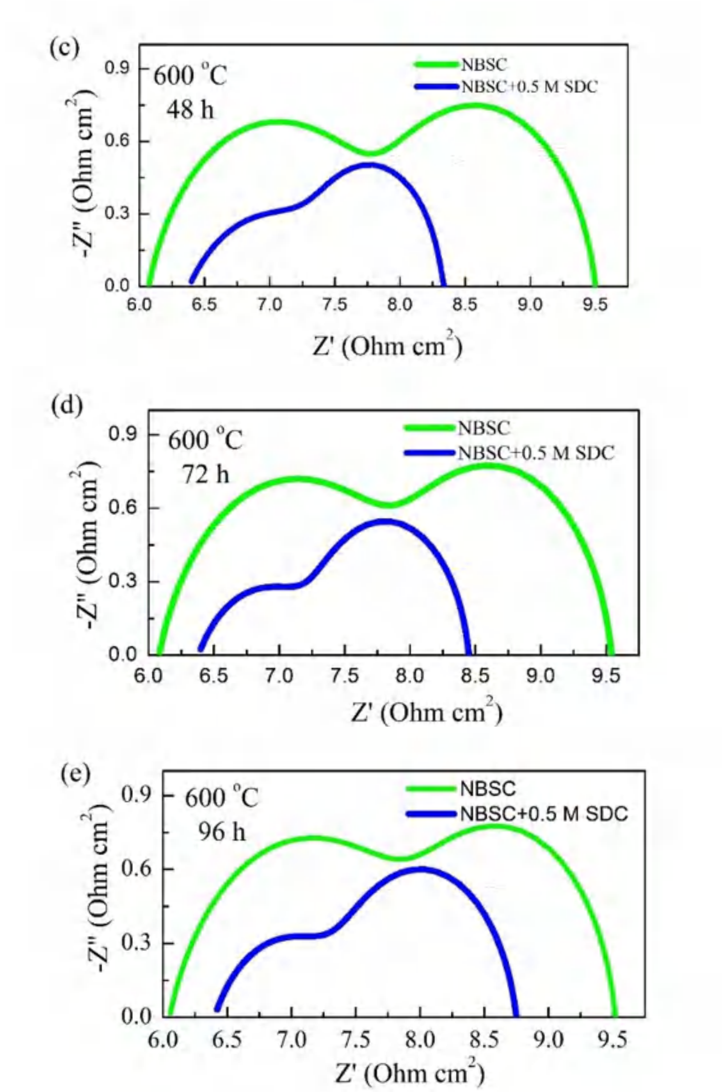


Figure 2. Long-term test: (a) Nyquist diagram of symmetrical cell spectroscopic impedance NBSC|SDC|NBSC and (b) NBSC+0.5 M SDC |SDC|NBSC+0.5 M SDC in the temperature range 2–96 hours at 600^{o}C

The Rp value of the NBSC+0.5 M SDC cathode specimen increases gradually over time, whose value goes up from 1.82 Ω.cm^2 in the initial 2 hours to 2.35 Ω.cm^2 on a long-term test of 96 h at 600°C as shown in Table 2. A slight improvement in the cathodic Rp, and the rising rate was about 0.55% h^{-1} from the initial two h to 96 h. The NBSC+0.5 M SDC cathode sample has better long-term stability than NBSC, with a lower Rp value of 2.35 Ω.cm^2. Figure 3 shows the polarization resistance rate between the NBSC cathode and NBSC+0.5 M SDC.

Table 2. Rp Value of symmetrical cells for NBSC and NBSC+0.5 measured during 96 hours at 600°C

Time (h)	Rp ($\Omega.cm^2$)	
	NBSC	NBSC+0.5 M SDC
2	3.32	1.82
12	3.39	1.86
24	3.46	1.85
36	3.40	1.92
48	3.43	1.96
60	3.40	2.05
72	3.46	2.07
84	3.43	2.26
96	3.47	2.35

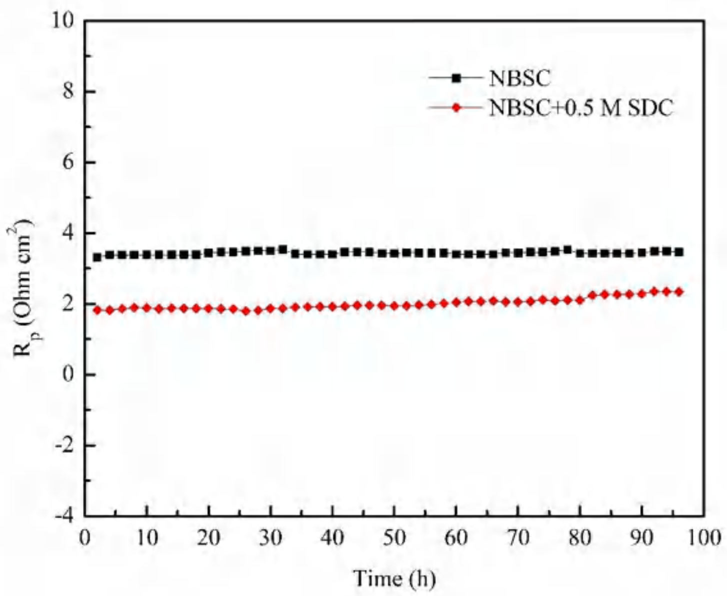
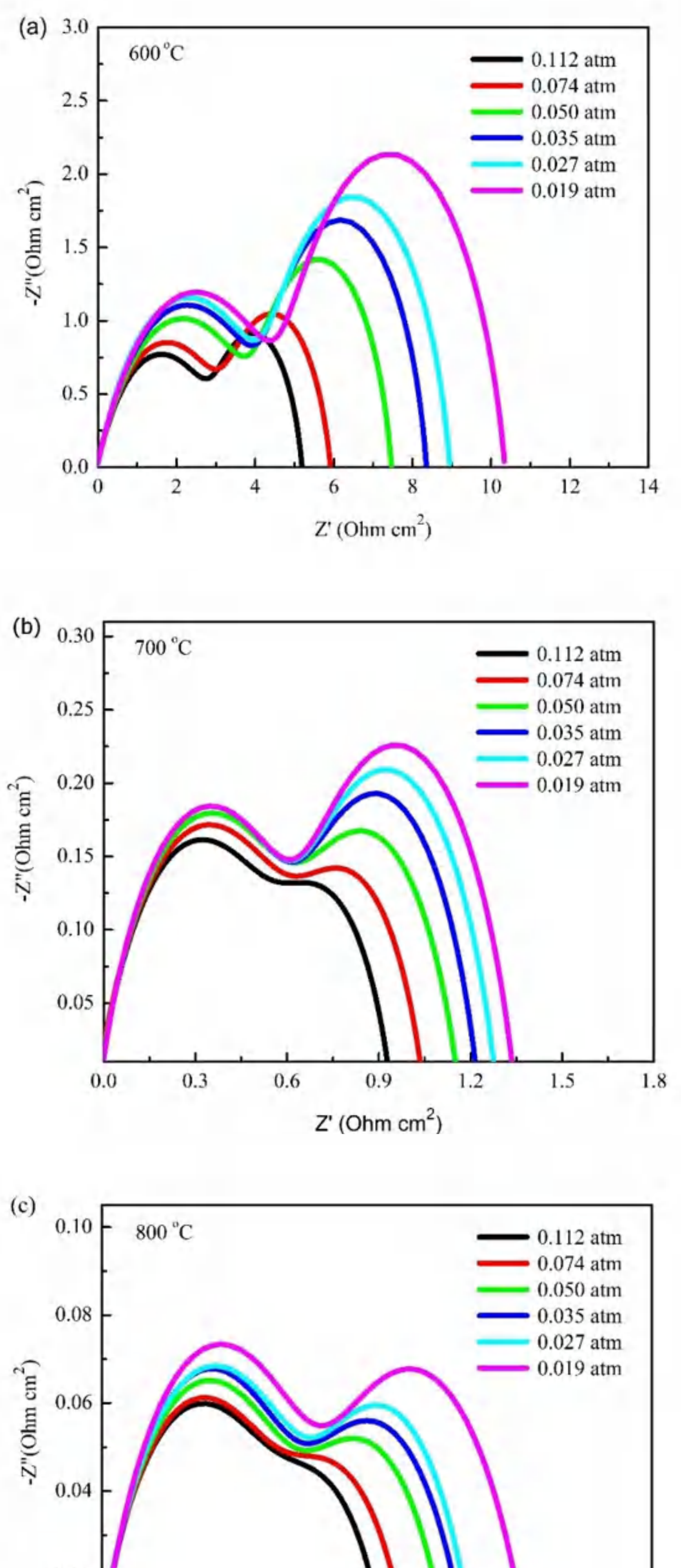


Figure 3. Polarization resistance (Rp) curve for long-term symmetric cell testing: (a) NBSC|SDC|NBSC, and (b) NBSC+0.5 M SDC|SDC|NBSC+0.5 M SDC.

The cathode delamination of the electrolyte can be one of the possible causes of the increased Rp at the beginning of rapid degradation. Delamination between layers affects a drop in the activity site of the ORR, which causes an improvement in polarization resistance. According to previous studies, the difference between NBSC (TEC = 25.2 ppm K^{-1}) and SDC (TEC = 12.4 ppm K^{-1}) is approximately 12.8 ppm K^{-1} [22]. After testing, the surface of the cathode sample experienced agglomeration. The microstructure growth, such as grain expansion or particle coarsening, is critical since a practical SOFC operates at high temperatures (at T = 800-1000 °C) [33]. With increasing temperature and current density, as well as the duration of the test, the microstructure gets coarser and denser [34].

3.3. Symmetric cell measurement in various OPP

To further investigate the oxygen reduction reaction process of the symmetrical specimen NBSC+0.5 M SDC|SDC|NBSC+0.5 M SDC. The electrochemical impedance spectroscopy (EIS) was tested at different partial pressures of oxygen (OPP) according to previously published papers [22]. The Rp values are based on the Nyquist diagram of NBSC+0.5 M SDC cathode impedance spectroscopy on SDC electrolytes with various OPP at various temperatures, as shown in Figure 4. The polarization resistance values increase with decreasing OPP at 600°C, 700°C, and 800°C, respectively. At a temperature of 600°C, the value of the polarization resistance (Rp) increases from 3.69 Ω.cm^2 (0.112 atm) to 6.05 Ω.cm^2 (0.019 atm). At 700°C the pattern is the same, the Rp value increases from 0.75 Ω.cm^2 to 1.17 Ω.cm^2 at 0.112 atm and 0.019 atm, respectively. Also, at 800°C the increase in polarization resistance continues from 0.030 Ω.cm^2 to 0.039 Ω.cm^2 at OPP = 0.112 atm and 0.019 atm, respectively.



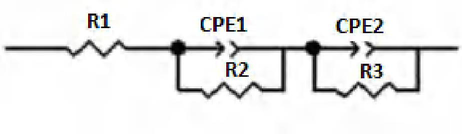


Figure 4. (a)-(c) Nyquist diagram of impedance spectroscopic symmetric cell NBSC+0.5 M SDC|SDC|NBSC+0.5 M SDC in the OPP range between 0.112–0.019 atm at various temperatures and (d) Equivalent circuit used to fit the impedance spectra

The resistance of the evaluation cell was demonstrated using an analogous circuit of the impedance curve and was fitted using Z-View based on the following series circuit R_1 (R_2-CPE_1) R_3-CPE_2). The R is the same as Ohmic resistance (RΩ), and the resistance of polarization is characterized by two resistances (R_2+R_3). A constant phase element (CPE) symbolizes a non - ideal capacitor, such as the double layer at a nonplanar TPB, and the n parameter correlated with the CPE equivalent to a real capacitor, where n=1.

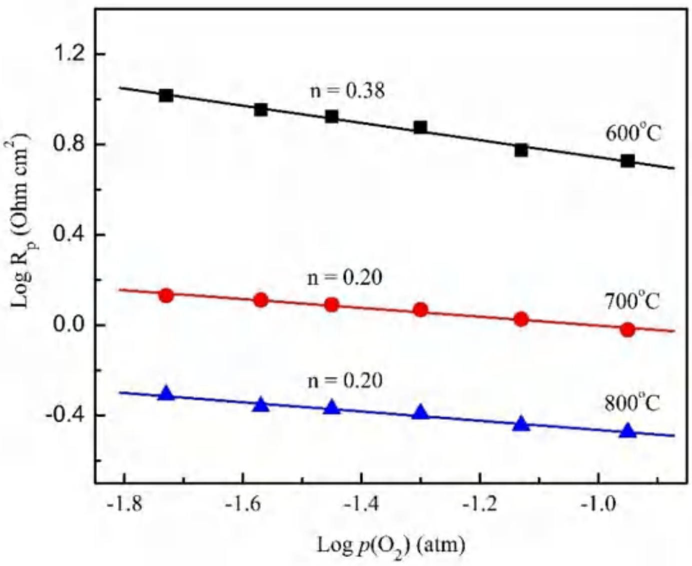


Figure 5. Polarization resistance between layers as a function of oxygen partial pressure (OPP) symmetrical cell NBSC+0.5 M SDC|SDC|NBSC+0.5 M SDC at various temperatures

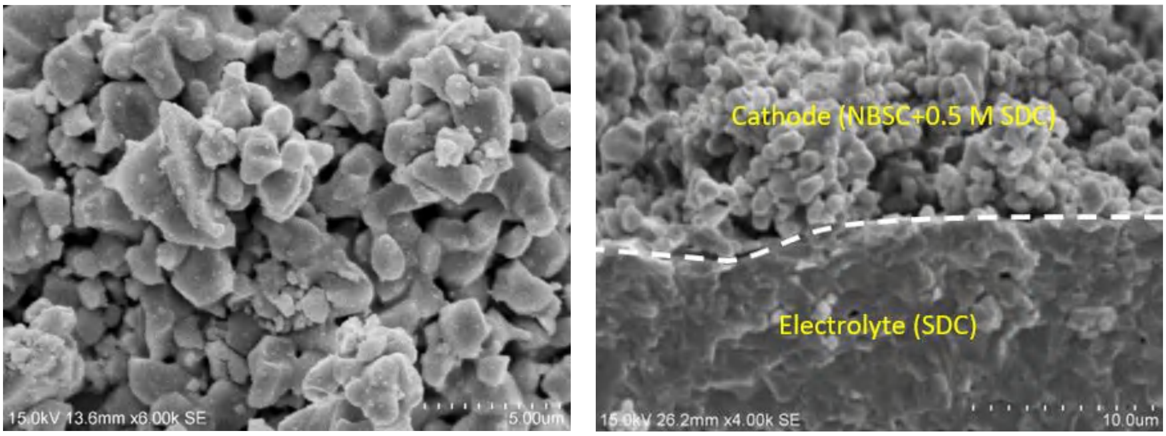
Table 3. The Rp value of symmetrical cells in the OPP range of 0.214–0.0027 atm at various temperatures

OPP (atm)	600°C	700°C	800°C

	Rp (Ω.cm^2)	Rp (Ω.cm^2)	Rp (Ω.cm^2)
0.112	3.69	0.75	0.030
0.074	3.99	0.82	0.032
0.050	4.54	0.91	0.033
0.035	5.06	1.02	0.035
0.027	5.61	1.08	0.038
0.019	6.05	1.17	0.039

The log (Rp) as a function of log P(O_2) cathode NBSC+0.5 M SDC measured at various temperatures is illustrated in Figure 5. It is evident that as P(O_2) decreases, the value of Rp rises due to a reduction in mobile interstitial oxygen at lower P(O_2). The Rp value of symmetrical cells in the OPP range of 0.214 atm–0.0027 atm at various temperatures, as shown in Table 3. The value ranges between n = 0.20 and n = 0.38, depending on the slope of the curve. The result shows that the primary ORR process is dominated by the charge transfer process (charge transfer processes) TPB and/or site 2PB cathode NBSC+0.5 to the electrolyte in the temperature range 600°C–800°C [35]. The transfer of electrons and oxygen is closely related to the cathode structure, which impacts fuel cell performance, including reaction kinetics and charge and mass transfer preprocessing.

The microstructure of the NBSC+0.5 M SDC cathode surface and the cross-sectional microstructure of the two layers (cathode and electrolyte) are presented in Figure 6. The adhesion of the two layers (cathode and electrolyte) looks strong. The grain size was evenly distributed in the range of 1–2 µm, and the microstructure of the NBSC+0.5 M SDC cathode was porous. A good SOFC cell morphology includes (1) a porous cathode microstructure, (2) interlayer (electrolyte-cathode) connectivity, and (3) a dense electrolyte layer. Good morphology promotes rapid oxygen diffusion, minimizes polarization resistance, and improves current collection. The surface morphology of the NBSC+0.5 M SDC sample corresponds to the physical characteristics of the SOFC cathode. The limit for reasonable performance solid oxide fuel cell (SOFC) value is Rp <0.15 Ω.cm2). The surface morphology of the NBSC+0.5 M SDC sample corresponds to the physical characteristics of the SOFC cathode.



(a) (b)

Figure 6. SEM images: (a) The cathode surface and (b) Cross-sectional microstructure of the two layers (cathode and electrolyte).

4. Conclusion

This research analyzes the electrical conductivity, infiltration impact, and stability of the cathode operating at an intermediate temperature of 600°C–800°C. The NBSC cathode conductivity values are in the range that meets the requirements of SOFC cathode materials. The surface morphology of the NBSC+0.5 M SDC sample matches the physical properties of the SOFC cathode. The relatively small Rp value of the symmetrical cell is between 0.030 $\Omega.cm^2$ and 0.039 $\Omega.cm^2$ in the OPP range 0.214–0.0027 atm at 800°C, below 0.15 $\Omega.cm^2$ the reasonable performance limit for solid oxide fuel cells (SOFC). The infiltration of 0.5 M SDC on the NBSC porous cathode significantly reduced the interlayer's polarization resistance value by almost half compared to that of the NBSC cathode without 0.5 M SDC infiltration. The NBSC+0.5 M SDC cathode specimen demonstrated stability under SOFC operating conditions and increased operational life compared to non-infiltrated cathodes.

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