

INVESTIGATIONS OF Ni-SDC CARBONATE (Ni-SDCC) AS COMPOSITE  
ANODE FOR LOW TEMPERATURE SOLID OXIDE FUEL CELLS

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## ABSTRACT

Systematic research regarding NiO-SDC carbonate (NiO-SDCC) as composite anode is limited despite great chemical compatibility and cell performance achieved with other low temperature solid oxide fuel cell (LTSOFC) components. This study focuses to investigate the correlation of powder composition and calcination temperature on the chemical compatibility, morphologies, thermal and electrochemical performance of NiO-SDCC composite anode. NiO-SDCC composite powders with the weight ratios of 50:50 (NC55), 60:40 (NC64), and 70:30 (NC73) were achieved using high-energy ball-milling. All powders were calcined at 600–800 °C, pelletized and sintered at 600 °C. Characterisation include the crystalline phases, thermogravimetry, thermal expansion coefficient (TEC), hardness and morphologies were conducted. Electrochemical impedance spectroscopy (EIS) was conducted under in-situ reduction process. The powder and pellet morphologies, thermal expansion, and hardness were mostly affected by the calcination temperature as compared to powder composition. NC55 was selected for anode reduction process in hydrogen due for the least TEC values as compared to NC64 and NC73. The Ni-SDCC exhibited porosity of 36-40% after reduction process. The lowest area specific resistance of  $5.3 \Omega\text{cm}^2$  was achieved with sample calcined at 800 °C. In this study, unexpected mechanical failure has been observed after EIS measurements. Therefore, chemical reactions and anode failure mechanisms were successfully proposed in this study. This mechanism is a new finding that has not been reported in previous studies and must be given appropriate attention. In conclusion, this study significantly contributes to the development of Ni-SDCC as LTSOFC composite anode. Further enhancement on this material is required on improved durability for LTSOFC application.

## ABSTRAK

Penyelidikan yang sistematik berkenaan dengan komposit anod NiO-SDC karbonat (NiO-SDCC) masih terhad walaupun keserasian kimia dan prestasi sel tunggal yang unggul telah dicapai dengan komponen sel fuel oksida pepejal bersuhu rendah (*low temperature solid oxide fuel cell*, LTSOFC) yang lain. Kajian ini menumpu kepada perkaitan di antara komposisi serbuk dan suhu kalsin dengan mengkaji keserasian kimia, morfologi, terma dan prestasi elektrokimia bagi komposit anod NiO-SDCC. Serbuk komposit NiO-SDCC telah dihasilkan dengan menggunakan teknik pengisaran bebola bertenaga tinggi pada nisbah peratus berat NiO terhadap SDCC, 50:50 (NC55), 60:40 (NC64), dan 70:30 (NC73). Kesemua serbuk dikalsin pada suhu 600–800°C, dipellet dan disinter pada 600 °C. Pencirian melibatkan struktur hablur, termogravimetri, pekali pengembangan terma (TEC), kekerasan, dan morfologi telah dikaji. Spektroskopi impedans elektrokimia telah dijalankan untuk sel simetri dalam proses penurunan *in-situ*. Faktor kalsin lebih banyak menpengaruhi morfologi serbuk dan pelet, sifat pengembangan terma dan kekerasan berbanding komposisi serbuk. NC55 dipilih untuk kajian penurunan anod dalam persekitaran hidrogen kerana ia memberikan nilai TEC yang paling rendah berbanding NC64 dan NC73. Ni-SDCC telah dihasilkan dengan keliangan sebanyak 36-40% selepas proses penurunan anod. Rintangan luas spesifik yang paling rendah iaitu  $5.3 \Omega\text{cm}^2$  telah berjaya dicapai bagi sampel yang dikalsin pada suhu 800 °C. Dalam kajian ini, perhatian luar jangka berkenaan kegagalan mekanikal pada sel simetri selepas pengukuran EIS telah dikenalpasti. Sehubungan itu, kajian ini telah berjaya mencadangkan tindak balas kimia dan mekanisme kegagalan anod. Mekanisme ini merupakan penemuan baru yang belum lagi dilaporkan oleh kajian lepas dan perlu diberi perhatian sewajarnya. Kesimpulannya, kajian ini memberikan sumbangan yang penting dalam perkembangan Ni-SDCC sebagai komposit anod LSOFC. Kajian lanjutan untuk bahan ini amat diperlukan untuk meningkatkan ketahanan bagi aplikasi LSOFC.

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## LIST OF SYMBOLS AND ABBREVIATION

|                 |   |   |
|-----------------|---|---|
| AFC             | - | Alkaline fuel cell                          |
| ASR             | - | Area specific resistance                    |
| BSCF            | - | Barium strontium cobalt ferrite             |
| C               | - | Carbon                                      |
| CO <sub>2</sub> | - | Carbon dioxide                              |
| Ce              | - | Cerium                                      |
| DOE             | - | Department of energy                        |
| EDS             | - | Energy dispersive spectroscopy              |
| EIS             | - | Electrochemical impedance spectroscopy      |
| FESEM           | - | Field emission scanning electron microscopy |
| GDC             | - | Gadolinium-doped ceria                      |
| h               | - | hour  |
| H <sub>2</sub>  | - | Hydrogen                                    |
| HT              | - | High temperature                            |
| HV              | - | Hardness Vickers                            |
| IT              | - | Intermediate temperature                    |
| Li              | - | Lithium                                     |
| LSCF            | - | Lanthanum strontium cobalt ferrite          |
| LT              | - | Low temperature                             |
| NiO             | - | Nickel Oxide                                |
| Ni              | - | Nickel                                      |
| MCF             | - | Molten carbonate fuel cell                  |
| Na              | - | Natrium                                     |
| O <sub>2</sub>  | - | Oxygen                                      |
| OCV             | - | Open circuit voltage                        |
| R <sub>O</sub>  | - | Ohmic resistance                            |
| R <sub>p</sub>  | - | Polaristion resistance                      |

|       |   |                                    |
|-------|---|------------------------------------|
| PEMFC | - | Proton exchange membrane fuel cell |
| PAFC  | - | Phosphoric acid fuel cell          |
| SOFC  | - | Solid oxide fuel cell              |
| SDC   | - | Samarium-doped ceria               |
| SDCC  | - | Samarium-doped ceria carbonate     |
| Sm    | - | Samarium                           |
| TEC   | - | Thermal expansion coefficient      |
| TPB   | - | Triple phase boundary              |
| XRD   | - | X-ray diffraction                  |
| YSZ   | - | Yttria stabilised zirconia         |



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# **CHAPTER 1**

## **INTRODUCTION**

### **1.1 Background of study**

Over the past 30 years, rapid industrialisation, escalation in population and urbanisation had stepped up the global energy demand. According to the forecasted data from U.S International Energy Administration (IEA), an increase of 28% of the global energy demand is expected from 2015 to 2040, which is about 1.2% annually (EIA, 2017). Currently, the primary source of electricity is still dominated by technologies that are relying on the non-renewable fossil fuel combustion such as coal, petroleum, and natural gas. This impacts greatly on the global climate due to the anthropogenic increase of green-house emission. With the diminishing fossil fuel reserves, much research is on-going for decades attempting to bring alternative sources of energy to the next impetus. Fuel cells can be integrated as a part of global power generation sources that would cater to the increasing demand of the global energy (Abdalla *et al.*, 2018; Kreuer, 2013; Zhang *et al.*, 2010).

Fuel cells are stationary electrochemical devices that generate renewable energy from the conversion of chemical energy in a form of fuel (hydrogen, H<sub>2</sub>) and oxygen (O<sub>2</sub>) into usable electricity. There are a few types of fuel cell which are classified after the electrolyte type and operating temperature. This includes the polymer electrolyte membrane fuel cell (PEMFC), phosphoric acid fuel cell (PAFC), alkaline fuel cell (AFC), molten carbonate fuel cell (MCFC) and solid oxide fuel cell (SOFC). Among them, SOFC has received extensive attention for power generation since no precious metal catalysts are used (Jiang, 2012; Kan & Thangadurai, 2014). Table 1.1 shows the description of several fuel cell types.

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