

Influence of Silver Addition on the Morphological and Thermal Characteristics of Nickel Oxide-Samarium Doped Ceria Carbonate (NiO-SDCC) Composite Anode

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Abstract: Addition of silver (Ag) as an electro-catalyst has been widely investigated to enhance the cathode performance for intermediate-to-low temperature solid oxide fuel cells. Ag is seldom incorporated into composite anode materials, especially for low temperature application. Therefore, this study aimed to investigate the effects of a small amount of Ag on the microstructure and thermal behaviour of nickel oxide-samarium-doped ceria carbonate (NiO-SDCC) composite pellets. A high-speed ball milling technique was employed to prepare the NiO-SDCC composite anode powder. Subsequently, a small amount of Ag (1, 3, and 5 wt.%) was added into NiO-SDCC composite powder via ball milling. The pellets were manually pressed and sintered at 600 °C. Characterisation of the composite anodes included X-ray diffraction, Fourier transform infrared spectroscopy (FTIR), energy-dispersive spectroscopy, scanning electron microscopy, dilatometry and porosity measurement. NiO-SDCC maintained good chemical compatibility regardless of Ag loading. FTIR analysis also verified the presence of carbonates, suggesting that Ag did not influence the carbonate bonding in all NiO-SDCC. The porosity of all composite anodes was maintained within the satisfactory level for good anode performance (20%–40%). The thermal expansion of the composite samples matched well with the SDCC electrolyte. This finding indicated that the addition of small Ag loading into NiO-SDCC was within the acceptable range, demonstrating promising potential as low-temperature solid oxide fuel cell composite anode.

Keywords: Nickel oxide (NiO), samarium doped-ceria carbonate (SDCC), silver, composite anode, electrocatalyst

1. Introduction

In the past decade, research efforts have focused on developing high-performance composite anodes by mixing nickel oxide (NiO) as anode material with ceria-based electrolytes, such as nickel oxide samarium-doped ceria (NiO-SDC) and nickel oxide gadolinium-doped ceria. However, the cell performance of composite anodes is limited at the intermediate temperature region of 600°C–800 °C [1–3]. At reduced temperature <600 °C, cell stability is affected due to the partial stability of cerium (Ce⁴⁺ to Ce³⁺). To overcome this issue, high-performance composite electrolytes, such as samarium-doped ceria carbonates (SDCCs), were developed and gained much popularity. Thus, the research and development of low-temperature solid oxide fuel cells (LT-SOFCs; 400°C–600 °C) greatly accelerated. SDCC is developed by the addition of binary carbonate (Li/Na)₂CO₃ into SDC, yielding good electrochemical performance and preventing the partial stability of cerium faced by pristine ceria-based electrolytes. A combination of NiO-SDCC may provide promising performance, and considerable systematic research on this topic is in progress. Moreover, the potential of NiO-SDCC composite anode has been explored with promising cell performance at low temperature (400°C–600 °C) [4,5].

Polarisation resistance is one of the main challenges for reducing operating temperature. To reduce polarisation resistance, incorporating highly catalytic noble metal, such as platinum (Pt) and palladium (Pd), into electrodes is one possible strategy to improve the triple phase boundary (TPB) length [6,7]. These noble metals are usually expensive for cell commercialization. Alternatively, silver (Ag) is an inexpensive candidate with high electrocatalytic activity, high oxygen reduction rate, low electrical and contact resistance, good mechanical properties and stable in both oxidising and reduction atmospheres [8,9]. The addition of Ag into cathode components has been studied extensively for intermediate-temperature solid oxide fuel cells, resulting in promising cell performance by a significant reduction in cathode polarisation resistance as compared to Ag-free cathodes [10,11].

Aside from attaining good electronic conductivity and low polarisation resistance, an anode component requires good microstructure, adequate chemical compatibility and thermomechanical stability that are equally important for effective cell performance. To date, the effects of Ag addition into LT-SOFC cathodes has been found to influence the thermal expansion and cell microstructure, thereby affecting the cell performance [12–13]. A wide range the amount of Ag has been reported to enhance the properties of cathode. For example Ag loading ranging

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from 0-3 wt.%, 0.1-10 wt.% and 0-5 wt.% were investigated by Lee et al.[14], Wang et al.[15], and Sun et al.[16], respectively. Notably, this information is still scarce on anode components, especially on NiO–SDCC. Therefore, understanding the effects of Ag on the properties (physical, chemical and thermal) of NiO–SDCC is interesting. In this study, different Ag loadings (0,1,3,5 wt.%) were used to investigate the influence of Ag in terms of the chemical, physical and thermal properties of NiO–SDCC.

2. Materials and Method

2.1 Boundary conditions

SDCC electrolyte, which was prepared via ball milling, consisted of 20 wt.% binary carbonates (67 mol.% Li_2CO_3 :33 mol.% Na_2CO_3) Sigma–Aldrich, USA) and 80 wt.% SDC (Kceracell, South Korea). The mixture was milled for 24 h at 150 rpm in ethanol. Subsequently, the mixture was dried for 24 h in an oven at 100 °C to vaporise ethanol. The dried aggregate was then refined using an agate mortar before being used to prepare composite anode.

A nano-sized NiO (<100 nm) (Kceracell, South Korea) was mixed with the above-mentioned SDCC electrolyte powders at the weight ratio of 60:40 wt.% to prepare the composite anode powder. High-energy ball milling (Fristch, Germany) was employed to prepare the composite anode powders at a speed of 550 rpm for 2 h. The slurry was then dried for 24 h at 100 °C and refined using agate mortar. The powder was placed in a closed-lid alumina cup and assigned for calcination at 700 °C for 1 h in air using a furnace (PLF130, Protherm, Turkey). The powder was then refined again using an agate mortar to reduce agglomeration.

Ag powders (300-700 nm, Alfa Aesar, USA) were systematically added at 0,1, 3, and 5 wt.% into NiO–SDCC powder by low-speed milling of 100 rpm for 1 h. The composite powders were uniaxially pressed using a manual hydraulic press (Bench Top 3851, Carver, USA) into anode pellets (~0.90 mm thickness, 13 mm diameter) and rods (6 mm thickness, ~20 mm length). The anode samples were sintered in air at 600 °C at a heating rate of 2 °C/min for 1 h.

2.2 Sample characterisation

X-ray diffraction, (XRD; D8 Advance-Bruker, Germany) was used within the range of 2θ from 20–80° and Cu $K\alpha$ as the radiation source. Fourier transform infrared spectroscopy (FTIR; Spectrum 100-Perkin Elmer, USA) analysis was performed on the basis of the attenuated total reflectance technique over the range of 4000-550 cm^{-1} . Morphologies of anode pellets and elemental analysis were obtained using field-emission scanning electron microscopy equipped with electron-dispersive spectroscopy (FESEM-EDS; JSM 6380-Jeol, Japan). Carbon tape was used as conductive adhesive on top of metallic conducting stub for both fractured cross-section of anode pellets and powders to ensure sample

stability during imaging process. Apart from that, a thin layer of gold coating was sputtered on the sample to improve the surface conductivity prior to imaging (JFC 1600-Jeol, Japan). Image J software (1.51k) was used to estimate the grain size of the anode pellet from the FESEM micrograph. Dilatometry measurements (L75H-Linseis, Germany) were conducted within 30–600 °C in accordance with ASTM E228-11 to investigate the thermal expansion coefficient (TEC) of the anode. The porosity of the anode pellets was determined using Archimedes' principle (Density Determination Kit, XP-Mettler Toledo, USA). The anode pellets were immersed in ethanol.

3. Results and Discussion

Fig. 1 shows the XRD diffractogram for sintered NiO–SDCC composite anode pellets incorporated with different Ag loadings. Only three cubic crystalline phases were identified, namely, NiO (JCPDS No. 47-1049), SDC (JCPDS No: 75-0158) and Ag (JCPDS No: 04-0783). The intensity of the Ag peaks also increased with the increase in Ag loading. Meanwhile, the carbonate peaks were undetected within the sensitivity of XRD, suggesting that the carbonates were present in the amorphous state. Similar findings were reported by previous studies [1,10]. Moreover, the XRD results showed no formation of secondary phases, signifying good chemical compatibility and stability were achieved between Ag and the composite anodes. Formation of secondary phases can be detrimental to the structural stability of the composite anode. Therefore, this condition must be avoided to ensure that the intended function of the composite anode is maintained [18].

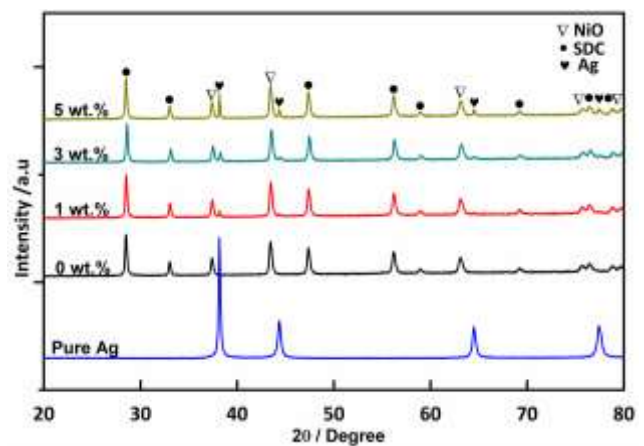


Fig. 1 X-ray diffractogram of NiO-SDCC composite anode pellets with various Ag loadings

Fig. 2 shows the crystallite size of NiO, SDC and Ag that estimated using Scherrer formula as in Equation (1).

$$D = \frac{0.9\lambda}{\beta \cos\theta} \quad (1)$$

where, D is the crystallite size, λ is the wavelength of the X-ray, β is the full width half maximum of the

diffracted peak, and θ is the angle of diffraction [19]. Since same sintering temperature of 600 °C is applied, the addition of Ag exhibited negligible variations on the crystallite size of NiO and SDC. Meanwhile, the crystallite size of SDC is slightly larger compared to NiO because of the carbonate coating on SDC particle. This trend is also consistent with previous studies reported by Jarot et al. [17] and Ma et al. [20]. The larger crystallite size of Ag as compared to NiO and SDC might be resulted from larger particle size of starting material (300 to 700 nm)

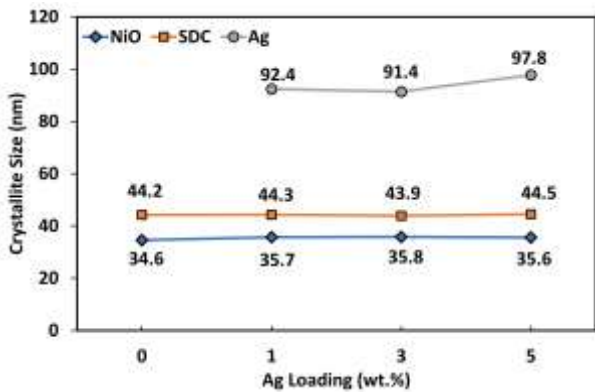


Fig. 2 Crystallite size of NiO-SDCC with various Ag loadings

Fig. 3 shows the EDS spectra for NiO-SDCC with Ag loading of 5 wt.%. The EDS results verified that no other elements were detected other than the main elements of the composite anodes, namely, nickel (Ni), samarium (Sm), ceria (Ce), sodium (Na), and silver (Ag), carbon (C) and oxygen (O). In addition, lithium (Li) was undetected in the EDS spectra due to its light atomic mass.

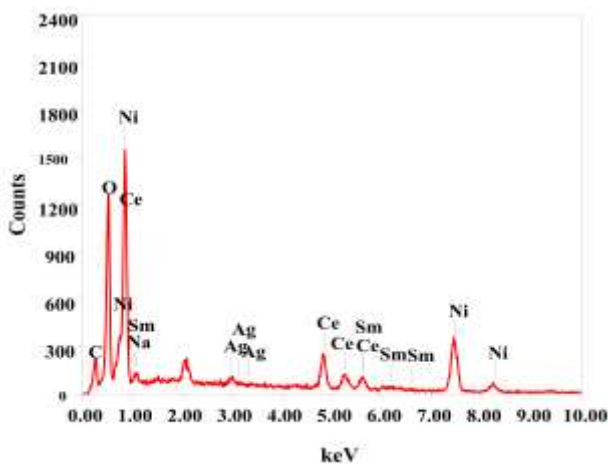


Fig. 3 EDS spectra of NiO-SDCC composite anode pellets with 5 wt.% Ag loading

Fig. 4 shows the FTIR spectra for NiO-SDCC with the addition of various Ag loadings. The presence of carbonate bonding existed within the peak range of 1433–1429 cm^{-1} and 881 cm^{-1} , which resembles to the stretching and the in-plane bending vibration modes of

CO_3^{2-} , respectively [21]. It is also observed that, these carbonate bonding was clearly detected in all composite anodes at increasing Ag loading. Furthermore, the existence of C and O elements were clearly observed in the EDS spectra as shown in Fig. 2. The presence of carbonates in the composite anodes are imperative for providing the fast multi-ionic conductions of H^+ and O^{2-} ; this phenomenon may improve the electrochemical performance at low operating temperature ($<600\text{ }^\circ\text{C}$) [22].

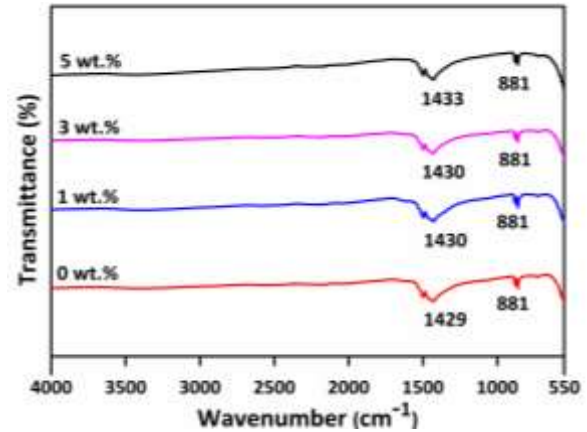


Fig. 4 FTIR spectra of NiO-SDCC composite anode powders with various Ag loadings

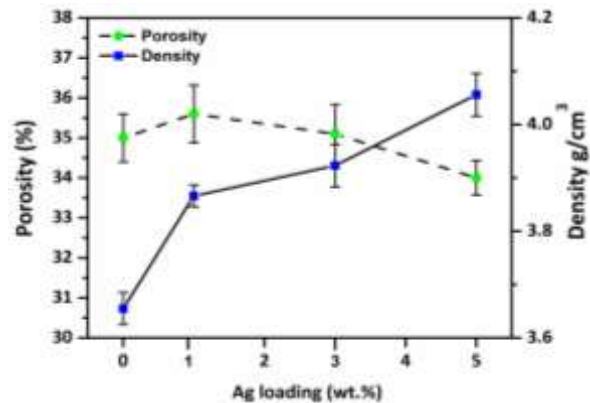


Fig. 5 Porosity and density of NiO-SDCC composite anodes with various Ag loadings

Fig. 5 shows the porosity and density results of NiO-SDCC composite anode pellets under various Ag loadings. A slight increase of 1% in porosity was observed with the addition of Ag loading of up to 1 wt.%. Meanwhile, further additional Ag loading of beyond 1 wt.% caused a minor decrease of 1.5% in porosity level. A reasonable explanation for such decrease in porosity might be due to Ag agglomeration, which increased the anode densification. This finding was supported by the slight increment in density from $3.66\text{ g}/\text{cm}^3$ to $4.06\text{ g}/\text{cm}^3$ (Fig. 5) when Ag loading increased from 0 wt.% to 5 wt.%. This phenomenon has been briefly outlined by Ding et al.[9], in which Ag metal may lead to the formation of Ag agglomerates. Therefore, selection of an appropriate Ag loading is important because high Ag loading may form agglomerates that block the pores, causing gas transport

restriction that is detrimental for the TPB length and electrochemical performance. A similar observation was reported by Li *et al.* [23] on $\text{La}_{1.6}\text{Sr}_{0.4}\text{NiO}_4\text{-Ag}$, in which an increase in Ag content $> 5 \text{ vol.}\%$ decreases the TPB, leading to an increase in polarisation resistance.

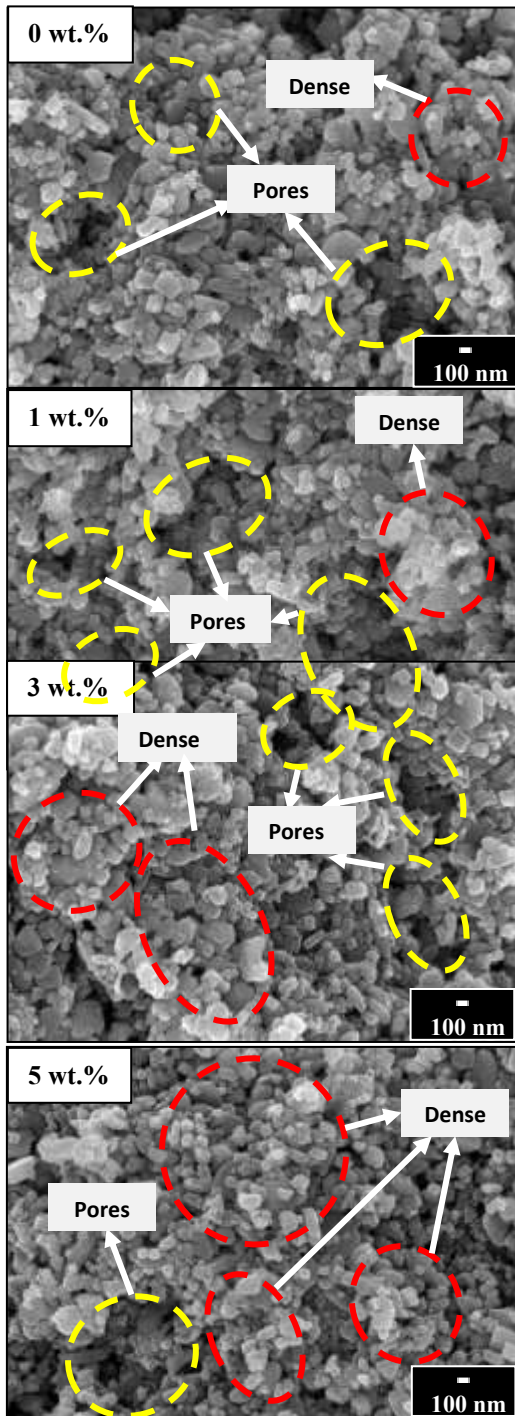


Fig. 6 FESEM cross section of NiO-SDCC composite anode pellets with various Ag loading

Fig. 6 shows the FESEM micrograph of the NiO-SDCC anode pellets obtained from the cross-sectional view. No prominent microstructural changes, such as the increment of grain size, were observed when

the Ag loading increased. The grains of the NiO-SDCC also remained in nano-submicron size. The grain size of the anode sample measured from Image J software were $134.1 \pm 4.1 \text{ nm}$, $134.9 \pm 5.3 \text{ nm}$, $135.5 \pm 5.0 \text{ nm}$ and $135.6 \text{ nm} \pm 2.8 \text{ nm}$ for Ag loading of 0, 1, 3, and 5 wt.%, respectively. The insignificant changes on the grain size is also in good agreement with the crystallite size as presented earlier. Nano-structured composite anodes are preferable in the current SOFC research due to the potential to provide good effective TPB length for enhanced electrochemical performance [14–15,20]. However, the increase in Ag loading caused slightly reduction on pores and created dense part (as depicted Fig. 6), which might be caused by Ag agglomeration as discussed earlier. This result was also in good agreement with the porosity and density trend as presented previously in Fig. 5. In brief, all composite anodes were still within the range of porosity requirement of 20%–40% for effective anodic electrochemical performance [26]. The Ag loading used in this study was also within the suitable range, without significant porosity reduction ($\sim 1.5\%$). It is noteworthy that, reduction in hydrogen will convert NiO-SDCC into ceramic-metal phase (Ni-SDCC). In general, this process will generate a high porosity level and microstructural re-distribution due to the transformation of NiO into Ni [27]. Thus, the influence of Ag needs to be further investigated in the future to evaluate the effects of Ag on the properties of reduced composite anode for LT-SOFC application.

The TEC obtained within the range of 30–600 °C is shown in Table 1. In SOFC application, achieving a small thermal expansion difference with respect to the electrolyte is crucial to ensure microcracks and delamination issues can be avoided during cell fabrication or operation at elevated temperatures. Mechanical failure due to thermal stresses can lead to gas crossover and cell deactivation [28]. In the present study, a slight increment in the TEC values of NiO-SDCC was observed as the Ag loading increased. Previous studies by Chen *et al.* [29] revealed that, the acceptable thermal expansion difference of the anode component with respect to the electrolyte is limited within the range of 15%–20% for maintaining good thermo-mechanical compatibility. On the bases of this criterion, all composite anodes exhibited thermal expansion difference of 1.64%–4.10% with respect to the TEC value of SDCC. In brief the TEC of all the anodes matched appropriately with the SDCC electrolyte.

Table 1 TEC values of NiO-SDCC with various Ag loadings

Sample	Thermal expansion coefficient (10^{-6} K^{-1}) (30-600 °C)	Thermal expansion difference (%)
SDCC	12.20	-
0 wt.%	12.40	1.64
1 wt.%	12.40	1.64
3 wt.%	12.70	4.10
5 wt.%	12.60	3.28

4. Summary

In this work, Ag with different loadings (0–5 wt.%) was incorporated into NiO–SDCC composite anodes. The chemical, physical and thermal behaviour of the composite anodes have been evaluated. XRD results revealed that all composite anodes shown excellent chemical compatibility without formation of secondary phases. The addition of Ag also did not interrupt the carbonate vibrational bonding as evidenced in FTIR. Besides that, all composite anodes revealed an adequate amount of porosity (20%–40%) for good anodic performance. A small thermal expansion difference was obtained for all composite anodes (1.64%–4.10%), thereby signifying good thermal matching with the SDCC electrolyte. In brief, this study demonstrated that the addition of small Ag loading into NiO-SDCC was within the acceptable range without compromising the important properties such as the microstructural and thermal characteristics. Hence, all sample presented in this study exhibited promising potential as composite anode for LTSOFC.

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