

## Synthesis and evaluation of NiO–Ce<sub>0.8</sub>Sm<sub>0.2</sub>O<sub>1.9</sub> nanocomposite powders for low-temperature solid oxide fuel cells

### Changsheng Ding\*, Toshiyuki Hashida

Fracture and Reliability Research Institute, Tohoku University, 6-6-11-707 Aza-Aoba, Aramaki, Aobaku, Sendai 980-8579, Japan

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#### ABSTRACT

In this study, NiO–Ce<sub>0.8</sub>Sm<sub>0.2</sub>O<sub>1.9</sub> (SDC) nanocomposite powders with NiO content of 40–60 mass% were successfully synthesized by hydroxide co-precipitation method. The effect of NiO content on crystal phase, crystallite size, average particle size and particle size distribution of the NiO–SDC nanocomposite powders was investigated. The average particle size of the NiO–SDC nanocomposite powders increased and the particle size distribution became wider with an increase in the NiO content. Anode-supported solid oxide fuel cells (SOFCs) were fabricated from the NiO–SDC nanocomposite powders was tested at 500–650 °C using humidified hydrogen as fuel. Results showed that the NiO–SDC nanocomposite powders were suitable to be applied as anode materials for low-temperature SOFCs. The anode-supported SOFC fabricated from the NiO–SDC nanocomposite powders with 50 mass% NiO demonstrated the highest electrical performance among the anode-supported SOFCs fabricated from the NiO–SDC nanocomposite powders with 50 mass%.

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#### 1. Introduction

Solid oxide fuel cells (SOFCs) are electrochemical energy conversion devices that convert chemical energy in fuels directly into electricity with high-energy conversion efficiency and low pollution emission [1]. Anode-supported SOFCs have attracted much attention in recent years due to their potential lower operating temperatures [2–4]. At low operating temperatures, the overall performance loss of anode-supported SOFCs is dominated by the polarization loss of the electrode reactions because of the ohmic loss across electrolyte minimized by reducing electrolyte thickness [5]. Thus, the development of electrode materials with high performance and stability is very important for the commercialization of anodesupported SOFCs. Such electrode materials can be achieved either by the development of new electrode materials with high ionic-electronic conductivity or by the continuous improvement of existing electrode materials, such as (La,Sr) (Co,Fe)O<sub>3</sub> cathode and Ni/Sm-doped CeO<sub>2</sub> (Ni–SDC) anode.

Ni–SDC is regarded as the suitable anode materials for low-temperature SOFCs due to the excellent electrocatalytic activity for hydrogen oxidation reaction, high ionic and electronic conductivity, and high stability and compatibility with the Sm-doped CeO<sub>2</sub> (SDC) electrolyte [6]. It has been shown that the electrocatalytic activities of nickel anodes for the hydrogen oxidation are directly related to the length of three phase boundary (TPB), the intersection of the electrolyte–electronic conductor–gas phases, and increasing the

<sup>\*</sup> Corresponding author. Tel.: +81 22 795 7524; fax: +81 22 795 4311. E-mail address: dingchsh@rift.mech.tohoku.ac.jp (C. Ding).

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TPB length may lead to the decrease of polarization loss of anodes [7–9]. Thus, the grain size and distribution of the Ni and SDC phases in the Ni–SDC anode, which affect the length of TPB, may be regarded as being critical to the performance of the Ni–SDC anodes. For Ni–SDC anodes, the large TPB length may be obtained from homogeneous and contiguous structure of pores, Ni and SDC fine grains.

Anode materials were usually prepared either by impregnating metal salts into a porous matrix [10,11] or by the mechanical mixing method [12,13], which are simple and allow for accurate chemical composition. However, it is difficult to achieve uniform distribution of Ni and SDC phases in the anode by these methods. The non-uniform distribution of Ni and SDC phases in the anode may be unbeneficial for the formation of the Ni-Ni and SDC-SDC networks, and thus result in low electrical performance. Therefore, to improve the performance of Ni-SDC anode, the NiO-SDC composite powders with uniform phase distribution need to be developed. Yin et al. [14] synthesized the NiO-SDC composite powders with average particle size of 45 nm using gel-casting technique. Kawano et al. [15] synthesized the NiO-SDC composite particles by spray pyrolysis method from the starting solution containing citric acid without preheat treatment. Chen et al. [16] reported a urea-combustion technique using nitrate and urea as raw materials to synthesize the NiO-SDC composite powders consisting of 40-150 nm particles. Liu et al. [17] synthesized the NiO-SDC composite powders by self-rising approach using polyvinyl alcohol as fuel and binder and urea as leavening agent. Each synthesis method has its advantages and disadvantages. Compared with the previous reported methods, hydroxide co-precipitation method is simple and easy to be controlled, and can produce more homogeneous and small sized powders. In this study, we applied hydroxide co-precipitation method to synthesize NiO-SDC nanocomposite powders with uniform phase distribution.

In addition, decreasing the Ni content in the Ni–SDC anode is effective to reduce the difference of the thermal expansion coefficient of electrolyte and anode, and suppress the sintering of fine Ni grains during the operation of SOFC. However, for high electrical conductivity, Ni content above 30 vol.% on the basis of a percolation model is needed for the Ni-based anodes [18]. SOFC anodes were usually prepared with the Ni content of over 50 vol.% in order to obtain good electrical performance. However, for the use of nanocomposite NiO-SDC powders, the good Ni-Ni and SDC-SDC networks may be formed in Ni-SDC anode, and thus the use of the low NiO content may be expected to give better electrochemical performance of SOFCs. Therefore, in this study, the NiO-SDC nanocomposite powders with different NiO content were synthesized by hydroxide co-precipitation method, and the electrochemical performance of anode-supported SOFCs fabricated from the NiO-SDC nanocomposite powders was evaluated and compared.

#### 2. Experimental

 $\label{eq:ce(NO_3)_3} \cdot 6H_2O \mbox{ (Kanto Chemical Co., 99.99\%), Sm(NO_3)_3} \cdot 6H_2O \mbox{ (Kanto Chemical Co., 99.95\%) and Ni(NO_3)_2} \cdot 6H_2O \mbox{ (Kanto Chemical Co., 99.95\%)}$ 

Chemical Co., 99.95%) were used as starting materials for the synthesis of NiO–Ce<sub>0.8</sub>Sm<sub>0.2</sub>O<sub>1.9</sub> (SDC) nanocomposite powders. NaOH and NH<sub>3</sub>·H<sub>2</sub>O were used as co-precipitation agents. The appropriate proportion of Ce(NO<sub>3</sub>)<sub>3</sub>·6H<sub>2</sub>O, Sm(NO<sub>3</sub>)<sub>3</sub>·6H<sub>2</sub>O and Ni(NO<sub>3</sub>)<sub>2</sub>·6H<sub>2</sub>O were dissolved into distilled water, and continuously stirred to form a homogenous solution. NH<sub>3</sub>·H<sub>2</sub>O solution was then added dropwise to the above solution to form precipitate with stirring until the pH of 7 was reached. And then, NaOH solution was added to form complete precipitate was then washed with distilled water and ethanol for several times. Finally, the precipitate was dried and calcined at 600 °C for 1 h to form NiO–SDC nanocomposite powders. The NiO content in the NiO–SDC nanocomposite powders was 40, 50 and 60 mass%, respectively.

Crystal phases of the synthesized powders were analyzed by X-ray diffraction (XRD, MX21, Mac Science, Japan) using CuK $\alpha$  radiation ( $\lambda = 1.5406$  Å). The morphology and particle size of the synthesized powders were examined by transmission electron microscopy (TEM, HF-2000, Hitachi, Japan).

In order to evaluate the synthesized NiO–SDC nanocomposite powders as anode materials for SOFCs, anode-supported SOFCs were fabricated from the NiO–SDC nanocomposite powders with different NiO content. The NiO–SDC nanocomposite powders were first pre-pressed in a steel die. SDC nanopowders (Anan Kasei Co. Ltd., Japan) were then added onto the pre-pressed green NiO–SDC substrate which was still contained in the steel die. And then, the SDC powders and NiO–SDC substrate were co-pressed to form a green bilayer and subsequently co-sintered at 1300 °C for 5 h to form dense SDC electrolyte film. Finally,  $La_{0.8}Sr_{0.2}Co_{0.8}Fe_{0.2}O_3$  (LSCF, prepared by a sol–gel process) nanopowders were screen-printed onto the SDC electrolyte films supported by the NiO–SDC anodes and sintered at 900 °C for 2 h to form complete single cells.

Electrochemical performance of the fabricated anodesupported SOFCs was evaluated using an in-house test station. Humidified H<sub>2</sub> (3 vol.% H<sub>2</sub>O) was fed to the anode chamber and oxygen was supplied to the cathode chamber. The current–voltage characteristics of the single cells were measured at 500–650 °C. The microstructure of the single cells after performance testing was examined by field-emission scanning electron microscopy (FE-SEM, S4300, Hitachi, Japan).

#### 3. Results and discussion

The XRD patterns of the synthesized powders with the NiO content of 40–60 mass% are shown in Fig. 1. All the synthesized powders consist of SDC and NiO two crystal phases without any other crystal phases, and the relative intensity of the diffraction peaks corresponding to the NiO phase increases with an increase in the NiO content. This means that the NiO–SDC composite powders have been successfully synthesized and the crystal phases of the synthesized NiO–SDC composite powders are unaffected by the NiO content. However, the resulted crystallite size and particle size of the synthesized NiO–SDC composite powders with different NiO content are different.

The crystallite sizes of NiO and SDC phases in the NiO–SDC composite powders were calculated from XRD line broadening

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