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Synthesis and Structural Properties of Zirconium Doped Strontium Neodymium Oxide

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Abstract

The purpose of this research is the synthesis and structural characterization of $SrNd_{0.65}Zr_{0.35}O_{3-\delta}$ perovskite material. The sample has been prepared by solid-state sintering method and was sintered at 1300 °C for 12 hours in an air atmosphere where rate of heating and cooling was 5 °Cmin^{-1}. The structural and various crystalline parameters of this sample were calculated by the X-ray diffraction (XRD) experiment. The XRD data was refined using FullProf suite software which is based on Rietveld method. Refinement confirmed perovskite structure with single-phase cubic perovskite (fluorite-type) structure. Space group was found as Fm-3m. The cell parameters of the material were calculated and found to be a = b = c = 5.252509 Å and α = β = γ = 90°. After Rietveld refinement, the goodness of fit (χ^2) value of the sample was in good agreement with the reference data. The structural, mechanical and chemical properties of the sample were studied by scanning electron microscopy (SEM) and differential thermal analysis (DTA). The SEM microstructures of the sample showed that the samples were very dense sintered at 1300 °C. The DTA scan was done for understanding the temperature dependent behavior. The dilatometer measurement of $SrNd_{0.65}Zr_{0.35}O_{3-\delta}$ shows a good agreement with the literature results of YSZ which is a commercial material used for the electrolyte component of SOFC. The structural, density and thermal properties demonstrate that SNZ perovskite can be used as a proton-conducting electrolyte material for the intermediate temperature solid oxide fuel cell (IT-SOFC).

Keywords

Materials science; Perovskite; Rietveld refinement; XRD; DTA; SEM; SOFC

1. Introduction

At the start of civilization, people burned wood for cooking, heating, extracting chemicals, melting metals, converting heat to mechanical power and many other things. The combustion of wood releases carbon dioxide (CO_2) into the air. Plants absorb CO_2 and convert it back to carbon, reusing it as a fuel. To reduce deforestation as well as to fulfill the energy demand, the Industrial Revolution began to meet the demand by using fossil fuels such as crude oil, coal and gas. The combustion of these fossil fuels has increased the density of CO_2 in the atmosphere. As a result, global warming has commenced [1]. Global warming also occurs when the percentage of air pollutants and greenhouse gases (GHGs) such as carbon dioxide (CO_2), nitrous oxide (N_2O), methane (CH_4), ozone (O_3), aqueous vapors (H_2O), chlorofluorocarbons (CFCs) and hydrofluorocarbons (HFCs) increase in the atmosphere. The GHGs absorb solar energy, which is reflected from the earth's surface. Generally, some solar energy is reflected back to outer space and the remainder of it is absorbed by GHGs. The amount of GHGs is increasing day by day and they can remain in the air for years to come. As a result, heat is trapped in the Earth and gradually, the planet's temperature increases. This effect is called the greenhouse effect. Electricity production from the combustion of fossil fuels contributes to over 25% of global GHG emissions. In 2018, the single largest pollutant occurred in the coal-fired power plants that emitted more than 10 Gt of CO_2 [2].

Solid oxide fuel cells (SOFC) are considered a promising alternative source of energy production for reasons like low pollution, high efficiency (up to 60%) and fuel flexibility. SOFCs operate at intermediate temperature (500-750 ℃) and are considered the most promising green energy technology for their excellent protonic conductivity, thermodynamic stability, low environmental impact, long-term durability, conversion efficiency, a wide range of applications (including accommodation and transport) and the affordability [3-5]. The fuel cell has two porous electrodes known as an anode and a cathode, separated by a very dense electrolyte [3]. In the early 1980s, Iwahara's group first reported several perovskite-type oxide materials (ABO_3), such as barium cerate ($BaCeO_3$) and strontium cerate ($SrCeO_3$), when accurately doped with rare earth elements, exhibit high proton conductivity at high temperatures. This attribute sets perovskite-type oxides mainly based on $BaCeO_3$, $BaZrO_3$, $BaTiO_3$ and $SrCeO_3$ compatible for electrolyte materials in solid oxide fuel cells (SOFCs), batteries, piezoelectric devices, steam electrolysis, thermistors, capacitors, mobile electronic devices, motor starters, separators, supercapacitors, membrane reactor, hydrogen pumps and sensors [4, 6, 7].

In this study, a new composite material of $SrNd_{0.65}Zr_{0.35}O_{3-\delta}$ (SNZ) was prepared, where zirconium was doped for neodymium in the B site. The weight percentage of Zr was varied instead of Nd to find the optimal proportion of Zr doping and in this trial, only one composition $SrNd_{0.65}Zr_{0.35}O_{3-\delta}$ was found to be a pure phase. The perovskite compound was prepared by solid-state reaction method and this conventional method was selected because of its simplicity and low cost.

A solid oxide fuel cell (SOFC) is made of three significant segments: two permeable ceramic electrodes (anode and cathode) and a solid-state electrolyte shaped by solid metal oxides. Hence, the SOFC is otherwise called "Ceramic Fuel Cell". Solid oxide fuel cells (SOFCs) are high-temperature energy units that utilize a strong ceramic, inorganic oxide as an electrolyte; for example, Yttria-stabilized zirconia (YSZ) [8]. The most common SOFC is made out of an electrolyte made of YSZ, a permeable cathode made out of doped $LaMnO_3$ and a permeable anode made of Nickel and Yttria-stabilized zirconia cermet. SOFCs are considered by numerous analysts to be the most appropriate for creating power from hydrocarbons as a result of their straightforwardness, effectiveness, and capacity to endure some level of contamination. The raised working temperature of the SOFC brings about a few outcomes, for example, slow startup, significant expense, narrow mindedness to sulfur content and is not appropriate for larger variances in load requests. At Tokyo Gas Co. Ltd., Yakabe et al., [9] built up a 3 kW SOFC at a working temperature of around 700–1000 ℃. Although SOFCs have those previously mentioned favourable circumstances, stability and cost are currently upsetting potential applications in stationary, transport and compact force age. Strength is lacking for some SOFC applications. The main challenges for the commercialization of SOFCs are high manufacturing costs and reduced cell performance at a low operating temperature. The utilization of hydrogen as the source to control SOFCs has been explored by numerous scientists [10-12]. Zr and Y co-doped perovskite was utilized as a cathode for proton-conducting solid oxide fuel cells (PC-SOFCs), which produced a pinnacle power thickness of 0.97 W cm^{-2} at 500 ℃ and 0.13 W cm^{-2} at 350 ℃. It also showed good H_2O and CO_2 tolerance [13].

Depending on the operating temperature, the SOFCs are classified into three different types: (i) Low-temperature SOFC (LT-SOFC), the fuel cell involved in this type is operated in the temperature region of (400–600 ℃), (ii) Intermediate temperature SOFC (IT-SOFC), the fuel cell is operated in the temperature span of (600-800 ℃) and (iii) High-temperature SOFC (HT-SOFC), the fuel cell involved in this type is operated in the temperature range of (800-1000 ℃). LT-SOFC and IT-SOFC have generated enormous interest because of their numerous advantages over high-temperature SOFCs, which include smaller thermal mismatch between cell components and rapid startup with less energy consumption [14].

The primary target of this work was to investigate the structural, microstructural and thermal properties of $SrNd_{0.65}Zr_{0.35}O_{3-\delta}$ compound to find their potential use in IT-SOFCs. The investigation of the microstructural properties of the $SrNd_{0.65}Zr_{0.35}O_{3-\delta}$ materials was done using Scanning electron microscopy (SEM). Then, for analyzing the physical, mechanical and chemical stabilities of the materials, Differential thermal analysis (DTA) was carried out.

2. Experimental

The $SrNd_{0.65}Zr_{0.35}O_{3-\delta}$ material was prepared by the conventional method, named solid-state reaction method. Starting with the chemicals of $SrCeO_3$, Nd_2O_3 and ZrO_2 having higher than 99.9% purities as initial materials, were used in exact stoichiometric amounts to prepare the compound. The materials were weighed individually using a precise digital microbalance and then mixed properly in an agate mortar for one hour each with a small amount of ethanol. The sample was then heated on a magnetic heater at 80 ℃ for three hours until dry. The sample was then ground for one hour with two drops of polyvinyl alcohol (PVA). The PVA was used because of its solubility in water, as it is a synthetic polymer, also for its effectiveness in film-forming and emulsifying and has an adhesive nature. After that, the pellets were made of 2.5 gm of each and were pressed under the hydraulic press with 3 tons pressure and heated inside a muffle furnace at 900 °C for 12 hours with the heating and cooling rate of 5 °Cmin^{-1} for calcinations. The calcined samples were reground and remixed in a mortar pestle for 1 hour. Then the pellets of 13 mm diameter were made from the powder (2.5 gm each) by using the hydraulic press with 3 tons pressure and then pre-sintered at 1200 °C for 12 hours inside a muffle furnace. The XRD scans were performed on the sample to verify the phase purity and reaction after heating at 1200 °C. The grinding and pelleting of the samples were repeated again and finally sintered at 1300 °C for 10 hours inside a muffle furnace in an air atmosphere with the same heating and cooling rate of 5 °Cmin^{-1}. The SEM was done to check the grain growth and density of the materials. Differential thermal analysis (DTA) was performed to investigate the thermal behavior of the materials at various temperatures.

3. Results and Discussion

3.1 X-ray Diffraction (XRD)

The phase formation, phase purity and crystalline structure of SNZ material were found at room temperature by Rigaku Ultima IV X-ray Diffractometer within the 2θ scan range from 10° to 90° with a step width of 0.01° using $CuK_{\alpha1}$ filtered radiation (λ = 1.5406Å). The Chekcell [15] program was used for indexing the material. The XRD data was analyzed by using the FullProf [16, 17] suite program. The goodness of fit (χ^2) value obtained from the least-square refinement of the structural lattice parameters agreed with the literature data [18, 19]. The XRD powder diffraction pattern of the sample was obtained after sintering the sample. Figure 1 depicts the Rietveld refinement pattern of XRD data of $SrNd_{0.65}Zr_{0.35}O_{3-\delta}$ prepared at room temperature.

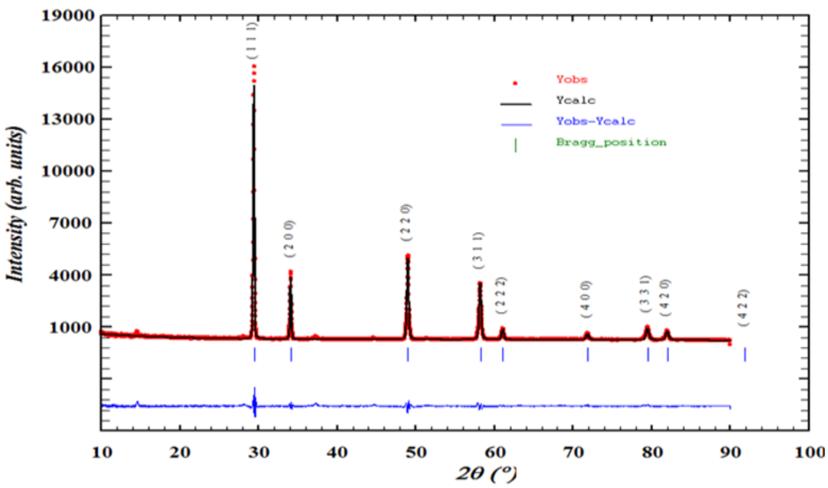

Figure 1. The Rietveld refinement XRD pattern of $SrNd_{0.65}Zr_{0.35}O_{3-\delta}$ at room temperature.

The analysis of XRD data gives the correct atomic positions, space group and cell parameters. The XRD pattern confirmed the cubic perovskite (fluorite-type) structure for the sample after Rietveld analysis and the positions of the peaks agree with the literature [18, 19]. The sample exhibits cubic perovskite (fluorite-type) symmetry with the Fm-3m space group. Figure 2 shows the polyhedral view of SNZ with the crystal structure. From the figure it is clear that Nd/Zr atom is in the center and Sr atom resides at the corner of the crystal structure. On the other hand, oxygen atom positions themselves at the edge which evidently forms the double perovskite structure.

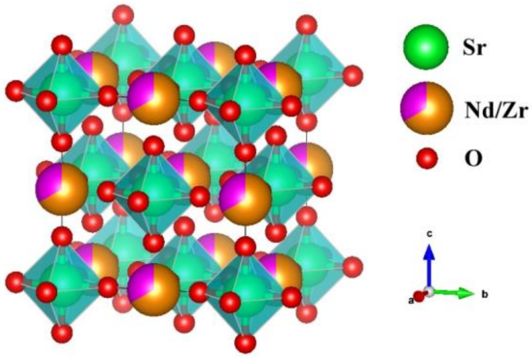

Figure 2. Polyhedral view of $SrNd_{0.65}Zr_{0.35}O_{3-\delta}$.

The X-ray powder diffraction indexed parameters of cubic $SrNd_{0.65}Zr_{0.35}O_{3-\delta}$ and the refinement parameters are listed in Tables 1 and 2 respectively.

Table 1. X-ray powder diffraction indexed parameters of cubic $SrNd_{0.65}Zr_{0.35}O_{3-\delta}$

$2\theta_{obs}$(°)	d_{obs}(Å)	$(I/I_o)_{obs}$	h	k	l	$2\theta_{cal}$(°)	d_{cal}(Å)	$\Delta 2\theta$(°)
29.452	3.0332	100	1	1	1	29.424	3.0332	-0.0005
34.132	2.6269	26.3	2	0	0	34.105	2.6268	-0.0013
49.032	1.8574	32.0	2	2	0	49.002	1.8574	0.0017
58.224	1.5840	22.2	3	1	1	58.149	1.5840	0.0009
61.081	1.5165	5.7	2	2	2	61.050	1.5166	0.0023
71.841	1.3135	4.2	4	0	0	71.817	1.3134	-0.0040
79.452	1.2053	6.2	3	3	1	79.451	1.2053	0.0007
81.948	1.1747	4.8	4	2	0	81.948	1.1747	0.0003

Table 2. The crystallographic data of the sample obtained from Rietveld analysis of the XRD data

Measurable factors	Outcomes
Unit cell parameters (Å)	a = b = c = 5.252509 and $\alpha = \beta = \gamma = 90°$
Space group	Fm-3m
Atomic fractional coordinates	Sr (0,0,0); Nd/Zr (0.5,0.5,0.5); O (0,0,0.25)
R-factors (%)	$R_p = 5.63$, $R_{wp} = 7.73$, $R_{exp} = 4.98$, $\chi^2 = 2.41$
Theoretical density (g/cm^3)	6.586

3.2 Scanning Electron Microscope (SEM)

Figure 3 shows the SEM image of SNZ and no pore or crack was found in the image for this material which was sintered at a temperature of 1300 °C in the air atmosphere. The SEM morphology of the SNZ material shows a very smooth and liberated surface. The sample surface is discovered to be profoundly grain growth, sizes and it displays a permeable thickness of protonic (H^+) conduction, which is one of the fundamental necessities of electrolyte material. The SEM revealed more than 97.5% relative density, confirmed by the Archimedes' method.

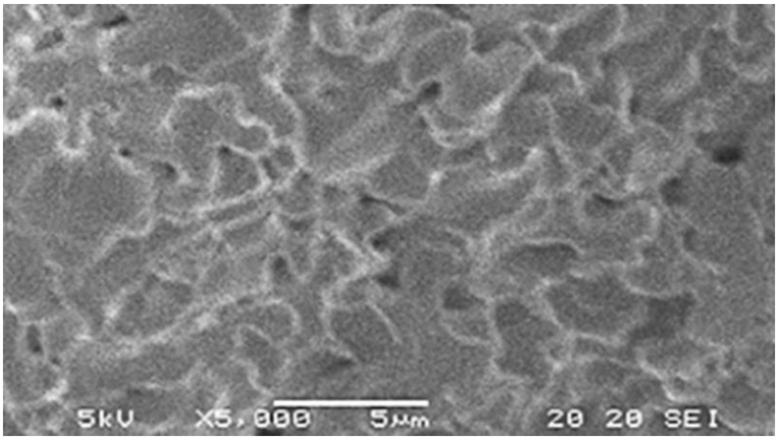

Figure 3. SEM micrograph of $SrNd_{0.65}Zr_{0.35}O_{3-\delta}$.

As appeared in Figure 3, it is seen that the revealing SNZ shows that the zirconium substitution makes the materials dense without any pore or crack. In 2013, Li et al., demonstrated the micrographs of the cracked surfaces of BCZYZ pellets in the wake of sintering at 1320 °C [20], which indicated high thickness without evident pores and splits. In 2018, Hossain et al., reported the SEM micrographs of the BCZYZ materials without any crack surface which was sintered at 1200 °C (4) without any sintering additive, which additionally demonstrated high density without pore and split. From the SEM morphology of SNZ, the average grain size is found to be 3.213 μm.

3.3 Differential Thermal Analysis (DTA)

The Figure 4 shows the DTA curve of the as-synthesized $SrNd_{0.65}Zr_{0.35}O_{3-\delta}$ sample. The thermal analysis data was taken in the air atmosphere. The DTA curve exhibits an exothermic peak at approximately 1400 °C. This peak is accompanied by a weight gain in the material, as evidenced by a simultaneous increase in the thermogravimetric analysis (TGA) curve.

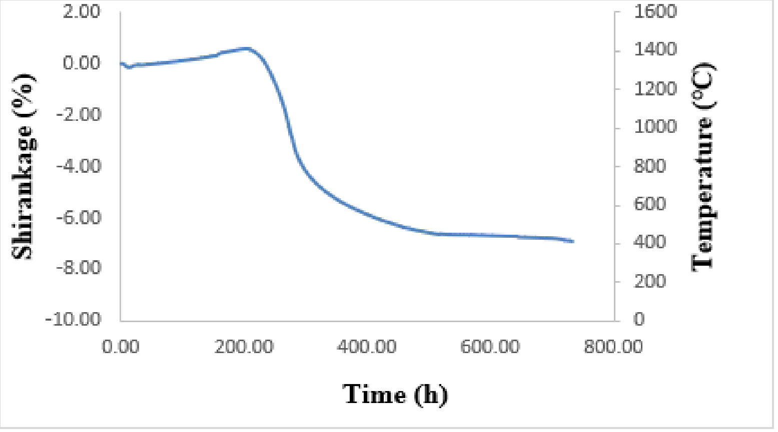

Figure 4. DTA performed in the air on $SrNd_{0.65}Zr_{0.35}O_{3-\delta}$ (heating rate 5 °Cmin^{-1}).

The exothermic peak at 1400 °C is attributed to the re-oxidation of oxygen vacancies in the perovskite structure. This process can be described by the following defect reaction in Kröger-Vink notation:

$$V_{\ddot{O}} + \frac{1}{2}\, O_2\,(g) \rightarrow\, O_O^{\times}$$

This reaction represents the filling of oxygen vacancies in the lattice by oxygen from the air, which results in a mass increase and releases heat. This re-oxidation is a common phenomenon in doped perovskite-type oxides, such as strontium zirconate, when they are synthesized under reducing conditions and subsequently heated in an oxidizing atmosphere like air. This result is consistent with the stability requirements for materials intended for use as solid oxide fuel cell (SOFC) electrolytes. The high density and thermal stability of SNZ align with IT-SOFC electrolyte requirements, as demonstrated by comparable shrinkage to YSZ (7% vs. 21%) business powder [21].

4. Conclusion

The material was successfully prepared by the solid-state reaction method and the XRD experiment reveals that the sample is in single phase. The crystalline parameters were obtained by the room temperature X-ray diffraction experiment. The Rietveld refinement XRD data indicated that the material shows cubic perovskite (fluorite-type) crystal symmetry with the space group Fm-3m. The lattice constant for SNZ was found to be 5.252509 Å. The goodness of fit χ^2, profile R factor R_P, weighted R factor R_{wp}, and expected R factor R_{exp} were additionally determined by the refinement procedure and are well consistent. Differential thermal analysis (DTA) was done to examine the stability of the materials. The outcome from DTA displays a decent concurrence with the writing results made of YSZ as a commercial material utilized for the SOFC electrolyte part. The microstructure was seen by SEM and the morphology of the revealing sample shows an exceptionally dense material without any pore or crack. The surface of the sample (SNZ) is very smooth and liberated from any split. The average grain size of the sample (SNZ) was measured as 3.213 μm.

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