Synthesize and characterization of nanocomposite anodes for low temperature solid oxide fuel cell

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Abstract

Solid oxide fuel cells have much capability to become an economical alternative energy conversion technology having appropriate materials that can be operated at comparatively low temperature in the range of 400-600 °C. The nano-scale engineering has been incorporated to improve the catalytic activity of anode materials for solid oxide fuel cells. Nanostructured $\text{Al}_{0.10}\text{Ni}_x\text{Zn}_{0.90-x}\text{O}$ oxides were prepared by solid state reaction, which were then mixed with the prepared Gadolinium doped Ceria GDC electrolyte. The crystal structure and surface morphology were characterized by XRD and SEM. The particle size was evaluated by XRD data and found in the range of 20-50 nm, which was then ensured by SEM pictures. The pellets of 13mm diameter were pressed by dry press technique and electrical conductivities (DC and AC) were determined by four probe techniques and the values have been found to be 10.84 and 4.88 S/cm, respectively at hydrogen atmosphere in the temperature range of 300-600°C. The Electrochemical Impedance Spectroscopy (EIS) analysis exhibits the pure electronic behavior at hydrogen atmosphere. The maximum power density of ANZ-GDC composite anode based solid oxide fuel cell has been achieved 705mW/cm² at 550°C.

Keywords: nanocomposite, energy conversion technology, nanostructure anode, LT- SOFC

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1. Introduction

Fossil fuels are running out rapidly, therefore, the entire world is cognizant to find alternative energy sources [1]. Fuel cell is an undoubtedly emerging alternative energy candidate, which has much more interest among the conventional energy conversion technologies in this new millennium [2]. Fuel cell consists of a dense electrolyte sandwiched by porous electrode materials. Fuel cells are classified primarily by the kinds of electrolyte they employ and the chemical reactions that take place in the cell. Among the fuel cell's family, solid oxide fuel cell (SOFC) is much famous and deliberated the most exciting potential candidate for energy conversion technology due to fuel flexibility, and high power efficiencies [3-8]. Carbon mono oxide CO can be used as a fuel in solid oxide fuel cells; even it is poison for proton exchange membrane fuel cell (PEMFC) [9]. It is reported that fuel cell has combined advantage/disadvantages due to low/high working temperature; low working temperature requires expensive metal as catalyst while cheap electrode can be used for high temperature fuel cell [10]. Recently, an SOFC is operated at intermediate temperature in the range of 600-800°C [11- 17]. Jung and Huang [18-19] reported that the electrodes used in the conventional SOFC are composites of YSZ with an electronically conductive material, usually Ni at the anode and Sr doped LaMnO_3 at cathode. The Ni-YSZ cermet electrodes and LSM cathode offer reasonably good performance as well as high electronic conductivity at high temperature of 1000°C. However, Ni-YSZ electrode/anode creates numerous problems in the working of solid oxide fuel cell at high temperature of 1000°C. For example; material degradation, maintenance of high temperature, and high cost. Of course, Ni-YSZ cermet has been widely used as anode in an SOFC due to high catalytic activity and excellent electrical conductivity, but the structural and long-term performance stability of Ni-YSZ cermet anode is a critical issue [20]. Low temperature is desired to improve the stability of the electrode/anode material, which can help to produce low cost solid oxide fuel cell. It is therefore, an immense challenge to develop new Ni free or less Ni contents electronic and ionic conductors, which may provide massive performance at comparatively low temperature (400-600°C). For example Ni free $\text{Cu}_{0.2}\text{Zn}_{0.8}\text{O}$ nanostructured

anode with doped ceria oxide electrolyte and less Ni content $\text{Cu}_{0.16}\text{Ni}_{0.27}\text{Zn}_{0.30}\text{Ce}_{0.16}\text{Gd}_{0.04}$ nanocomposite anode exhibit high performance of 1000mW/cm^2 and 570mW/cm^2 at 580°C and 550°C , respectively [21]. It can be further elaborated that doped ceria oxide (DCO), doped lanthanum gallate and doped barium cerate are good ionic conductors, they possess 0.1S/cm ionic conductivity at 600°C [19, 124-25]. It can be suggested that only a fast ionic conductor cannot play an important role for high power density until a compatible electronic conductor (electrode) doesn't contribute. Obviously, the high cost is a major barrier to widespread commercially implementation of SOFC, but the most important technical barrier currently being addressed is electrode particularly fuel electrode (anode) [26]. Therefore, the present research work was focused to develop compatible composite anode for low temperature solid oxide fuel cells (LTSOFCs), which would attempt high performance in the temperature range of $400\text{--}600^\circ\text{C}$. It is suspected that the performance of fuel cell depends on the anode microstructure strongly; this can be determined by the anode composition and fabrication conditions [27]. In our particular research work, the optimized nanostructure anode has been fabricated which enhances the SOFC performance at low temperature. The best SOFC electrodes are the composites of electrolyte materials in dual nature for electronic as well as ionic conduction. In order to make electrodes partially ionic conductors, an appropriate electrolyte material is mixed in electrode to provide high way for ions to migrate at interface of both cathode-electrolyte and electrolyte-anode or vice versa.

New five samples were prepared in order to optimize the molar percentage of Zn element with less Ni element. Aluminum (Al) molar ratio was kept constant in all compositions. The ore of bauxite prevents the material for oxidation. The aim of this study and work is to reduce the Ni contents in anode materials by replacing it with Zn element, which can work at comparatively low temperature range ($400\text{--}550^\circ\text{C}$). The reason to reduce Ni contents in anode materials is that because Ni based electrodes/anodes execute some disadvantages; for example i) poisoning in the form of sulphur, ii) carbon deposition and iii) poor oxidation stability [23]. The detailed study

and analysis of the anode material and its behavior with different Zn ratios is extensively discussed below.

2- Experimental

2.1- Powders Preparation

2.1.1- Anode Preparation

The $\text{Al}_{0.10}\text{Ni}_x\text{Zn}_{0.9-x}\text{O}$ (ANZ) anode powders were synthesized through solid state reaction where $x = 0.1, 0.2, 0.3, 0.4,$ and 0.5 in molar ratio; $\text{Al}_2\text{NO}_3 \cdot 9\text{H}_2\text{O}$ (Aldrich, USA), NiCO_3 (Aldrich, USA), and $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ (Aldrich, USA) were used as starting materials. These powders were mixed and ground in a mortar with pestle for one hour to make homogeneous. Then the ground ANZ powders were sintered in a furnace for four hours at 800°C and annealed. These prepared samples were named as 44(a), 44(b), 44(c), 44(d), and 44(e). The Gadolinium Doped Ceria (GDC) powder was prepared by solid state reaction (discussed below) was mixed in this prepared ANZ anode material with a weight ratio of 20wt% electrolyte and 80wt% anode including a small amount about 0.1% of carbon. This 80wt%ANZ-20wt%GDC powder was named ANZ-GDC composite anode for solid oxide fuel cell.

2.1.2- Electrolyte Preparation

The appropriate molar ratio of $\text{Ce}_{0.8}\text{Gd}_{0.2}\text{O}_{1.9}$ (GDC) powder was prepared by dry method using $\text{Ce}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ and $\text{Gd}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ as the starting materials. The GDC ground powder was sintered at 800°C for four hours.

2.1.3- Asymmetrical Fuel Cell Fabrication

The fuel cell's pellets of 13mm diameter and 1mm thickness were fabricated by dry press technique making three consecutive layers of anode, electrolyte and cathode materials; 80wt % ANZ-20wt % GDC/GDC/BSCF layers were pressed under a pressure of $270\text{kg}/\text{cm}^2$. The thickness of the cells was controlled as 0.40, 0.25, and 0.35mm for anode, electrolyte, and

cathode respectively. The conventional cathode BSCF [229] was obtained prepared already and used as cathode material for performance test. The cells were sintered for 40 minutes at 600°C. In order to provide current collector, both external sides of the cell anode and cathode were painted by silver paste.

2.1.4- Conductivity Measurement

In order to measure the electrical conductivity of the anode materials, pellets of 13mm diameter and 3mm thickness were fabricated and sintered at 700°C for one hour. The silver was pasted on both sides of the pellets to provide good electrical contact. AC and DC conductivities were determined at hydrogen atmosphere by implementing Auto Versa STAT 2273 (Princeton Applied Research, USA) and KD 2531 Digital Micro-ohmmeter, China, respectively. The frequency range was varied from 0.01Hz to 1MHz. The both conductivities were calculated using formula;

$$\sigma = L/RA \quad (i)$$

Where σ is conductivity, L is the thickness of the pellet, R is the internal resistance, and A is the active area of the pellet. The active area of the pellet was taken 0.64cm².

2.1.5- Fuel Cell Performance

Fuel cell performance was obtained by providing pure hydrogen as a fuel at anode side and air as oxidant at cathode side under variable resistance load by using fuel cell testing unit L-43 China. The data of open circuit voltage (OCV) and current was recorded under each resistance load and I-V curves were drawn. Power density was also calculated from the I-V results and I-P power curves also were drawn. These curves are known as IV and IP characteristics of the solid oxide fuel cell. The H₂ gas flow rate was controlled in the range of 80 to 100ml/min at 1 atm pressure.

2.2- Material Characterization

2.2.1- X-Ray Powder Diffractometry

The XRD pattern of sintered anode was analyzed to determine the crystalline structure at room temperature by using X-Ray Powder Diffractometer (D/Max-3A Regaku) with Cu K α radiation ($\lambda = 1.5418 \text{ \AA}$), 35 kV voltage, and 30 mA current. The crystallite size (D_β) of ANZ anode materials was also calculated from line-broadening measurements on the (111) peak by applying the Scherer's formula;

$$D_\beta = 0.89\lambda/\beta\cos\theta \quad (\text{ii})$$

Where λ is the wavelength and β is the full width half maximum (FWHM).

2.2.2- Scanning Electron Microscopy (SEM)

A Philips XL-30 Scanning Electron Microscopy (SEM) was used to analyze the particle size and surface morphology of the ANZ-GDC material.

3.- Results and Discussion

3.1- Structural Studies

Figure 1 shows the crystallographic structure of the compositions $\text{Al}_{0.1}\text{Ni}_x\text{Zn}_{0.9-x}\text{O}$ where $x = 0.1, 0.2, 0.3, 0.4$ and 0.5 . The results exhibit that all the samples, which were sintered at 800°C for four hours execute crystalline nano-structure. The XRD peaks of small constant amount of Al in all samples have been completely shifted into Ni-Zn compound, because XRD patterns do not show any peak of the Al content. The ZnO and NiO phases have been observed only according to JCPDS cards no. 36-1451 and 01-1239 respectively. Average particle sizes of all $\text{Al}_{0.1}\text{Ni}_x\text{Zn}_{0.9-x}\text{O}$ compositions were calculated from the XRD patterns using Scherer's equation and shown in Table 1. The values of average particle size obtained from XRD patterns were found to be in the range of 24-51 nm, which revealed that the sintering temperature of 800°C creates good nanostructure crystallites in anode materials. However, the materials except 44(b) possesses less conductivity having the same sintering temperature of 800°C due to vary Ni and Zn contents, the

results of all the samples indicate the 70% contents of Zn compound is optimized compound. It can be depicted that the maximum conductivity of 10.84 Scm^{-1} can be achieved due to the nanostructure obtained at 800°C sintering temperature. The nanostructure created at 800°C emphasizes that the conductivity of the anode material can be enhanced. For example Vitoriano et al [11] reported that the values of polarization resistance (R_p) and ohmic resistance (R_s) for the symmetric cell using LSCFN800 electrode have been found that the very small the LSCFN800 have the smallest R_p value in pure oxygen while below or beyond this sintering temperature, the value of R_p increases.

3.2 Microstructure View (Scanning Electron Microscopy) SEM

The microstructure of the optimized composite anode $\text{Al}_{0.10}\text{Ni}_{0.20}\text{Zn}_{0.70}\text{O}$ 44(b) has been shown in figure 2. It has been found from SEM image that particles are in the range of 20-50 nm. These results of particle sizes validate indeed a good agreement with those results obtained from XRD data, which confirms that the present material holds nanostructure. The nanostructure enhances the surface to volume ratio of the material as compared to bulk material. It can be further observed from SEM photograph, that the ANZ-GDC composite anode is well homogeneous and porous. The porous structure helps to transfer the ions coming from the either side of cathode or anode during the cell reaction.

3.3 Conductivity Measurements

Both DC and AC conductivities of all samples 44(a), 44(b), 44(c), 44(d), and 44(e) were measured at hydrogen atmosphere in the temperature range of $300\text{-}600^\circ\text{C}$. Their results of measurements are shown in Figure 3(a) and 3(b), respectively, and the values are listed in Table 1. The maximum value of DC conductivity was found to be 10.84 S/cm and that for AC conductivity was 4.88 S/cm of sample 44(b) in hydrogen atmosphere at temperature 600°C . This conductivity reveals that the ANZ 44(b) material is suitable for anode material, because the DC conductivity of ANZ 44(b) material is approximately 100 times greater than that of GDC electrolyte material at hydrogen atmosphere. The conductivity of GDC electrolyte is reported 0.1 S/cm [26]. All the other samples have lower conductivities than that of 44(b).

3.4 Calculation of Activation Energy (E.)

The activation energy of all samples has been calculated at hydrogen atmosphere. Arrhenius curves have been plotted and shown in figure 4(a) and 4(b). These plots exhibit an increasing linear function of temperature, however, using the linear fit technique, the activation energies were calculated of all the materials by using formula given below;

$$\sigma = A/T \exp (-E_a/kT) \quad (iii)$$

Where σ is the conductivity, T is temperature in kelvin, A is exponential factor, and K is Boltzmann's constant. The results of activation energies of all the samples are also listed in Table 1. The results of measurements of activation energy from DC and AC conductivity at hydrogen atmosphere for sample 44(b) are shown in Figure 5(a) and 5(b), respectively. The activation energy of the sample 44(b) was found to be 2.7×10^{-2} and 9.6×10^{-2} eV from DC and AC data, respectively, which is shown in inset of figure 5(a) and 5(b). The small value of activation energy exhibits the existence of catalytic activity and capability of composite anode ANZ-GDC for transportation of H^+/O^{2-} ions when hydrogen fuel is provided [20].

3.5 AC Electrochemical Impedance Spectroscopy (EIS) Analysis

Electrochemical Impedance Spectroscopy (EIS) was employed to perceive the catalytic activity of the composite anode 80 wt.%ANZ-20 wt.% GDC at hydrogen atmosphere in the temperature range of 350-550°C. The results of measurements are shown in Figure 6, which indicate that the samples have almost same response at all temperatures except 350°C; The experimental curves of impedance spectra correspond to a pure electronic behavior at all temperatures [24]. However, the impedance spectra of 80 wt. % ANZ-20 wt. % GDC anode at low frequency intercept give high resistance due to addition of electrolyte. However, resistance becomes low at high-frequency intercept. It has been reported [26] in previous work that the composition of 20wt. % electrolyte and 80wt. % anode is the most suitable ratio to balance the electronic/ionic conductivity for the transportation of oxygen ions through an interface of either cathode-electrolyte or electrolyte-anode.

3.6 Performance Measurements

The performance of all the anode samples using GDC as electrolyte and conventional BSCF [27] as cathode were measured. It can be deduced that all the samples exhibited open circuit voltage values equal to the theoretical value of the solid oxide fuel cell. It has been found that the sample named as 44(b), having 70 % contents of Zn compound exhibits a maximum voltage and performance of 1.030 V and 705mW/cm² respectively at 550°C, whereas all other Zn percentage values yield low voltage as well as performance as compared to 44(b). However, Saroboga [30] reported a best value 426 mW/cm² at 800°C using Ni/YSZ as anode with H₂ gas fuel. The complete data of OCVs, current densities and power densities has been tabulated in Table 2. I-V and I-P curves were drawn from the data collected during cell performance. The results of measurements have also been shown in Figure 7. In this figure, unfilled emblems show the current density versus power density and solid emblems correspond to current density versus voltage.

4 CONCLUSIONS

The anode materials ANZ (different compositions) have been successfully synthesized by solid state reaction method. XRD patterns indicate that all compositions have their particle sizes in the range of 20-50 nm. It has been observed that the composition 44(b) having 70% contents of Zn compound has maximum power density of 705mW/cm² 550°C. It has also been noted that the 44(b) composition has maximum conductivity and minimum activation energy as compared to others Zn molar ratios in the same context. These Zn based anodes with fewer contents of Ni show reliable performance as well as conductivity. The purpose of this study was to find the Ni free or less Ni components composite anodes on the basis of NANOCOFC approach. However, the prepared sample has 20 mol% of NiO, which can be a precursor to produce Ni free anodes for solid oxide fuel cells. . All the samples were analyzed and tested with all aspects and it has been concluded that the sample 44(b) is highly suitable as far as conductivity and performance is concerned.

Acknowledgments

Higher Education Commission (HEC), Islamabad, Pakistan is highly acknowledged. This work is supported by HEC Pakistan through International Research Support Initiative Program (ISRIP). This work was completed in the Department of Energy Technology, Royal Institute of Technology, KTH, Stockholm, Sweden.

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Figure Captions

- Figure 1: XRD Patterns of Different Composition of $\text{Al}_{0.1}\text{Ni}_x\text{Zn}_{0.9-x}$, ($x = 0.1 - 0.5$)
- Figure 2: Scanning Electron Microscopic Image of Composite $\text{Al}_{0.1}\text{Ni}_{0.2}\text{Zn}_{0.7}$
- Figure 3(a): DC Conductivities of $\text{Al}_{0.1}\text{Ni}_x\text{Zn}_{0.9-x}$ Anode at Hydrogen Atmosphere
- Figure 3(b): AC Conductivities of $\text{Al}_{0.1}\text{Ni}_x\text{Zn}_{0.9-x}$ Anode at Hydrogen Atmosphere
- Figure 4(a): Arrhenius Plots of DC Conductivities of $\text{Al}_{0.1}\text{Ni}_x\text{Zn}_{0.9-x}$ at H_2 atm.
- Figure 4(b): Arrhenius Plots of AC conductivities of $\text{Al}_{0.1}\text{Ni}_x\text{Zn}_{0.9-x}$ at H_2 atm.
- Figure 5(a): Linear Fit Curve of Sample 44(b) for Activation Energy from DC Conductivity at Hydrogen Atmosphere
- Figure 5(b): Linear Fit Curve of Sample 44(b) for Activation Energy from AC Conductivity at Hydrogen Atmosphere
- Figure 6: AC Electrochemical Impedance Spectra at Different Temperatures.
- Figure 7: Performance Measurements of Samples 44(a), 44(b), 44(c), 44(d) and 44(e)

Table Captions

Table 1: Conductivity, Activation Energy and Particle Size Data of ANZ Anode

Table 2: Performance data of ANZ-GDC composite anodes

Table 1: Conductivity, Activation Energy and Particle Size Data of ANZ						
<i>Sample Category</i>	<i>Temp.(°C)</i>	<i>Conductivity at H₂ Atmosphere (S/cm)</i>		<i>Activation Energy (E_a) (eV)from Conductivity of</i>		<i>Particle Size (nm)</i>
		<i>DC</i>	<i>AC</i>	<i>DC</i>	<i>AC</i>	
44(a)	600	2.79	1.39	5.9x10 ⁻²	7.3x10 ⁻²	34.55
44(b)	600	10.84	4.88	2.7x10 ⁻²	9.6x10 ⁻²	50.21
44(c)	600	0.90	0.96	4.9x10 ⁻²	7.6x10 ⁻²	24.70
44(d)	600	1.31	1.54	4.4x10 ⁻²	8.3x10 ⁻²	24.57
44(e)	600	0.79	0.85	3.1x10 ⁻²	8.0x10 ⁻²	31.10

Table 2: Performance Data of ANZ-GDC Composite Anodes

Cell Category	Temp. (°C)	Maximum OCV (V)	Maximum Current Density (mA/cm ²)	Maximum Power Density (mW/cm ²)
44(a)	550	0.965	991.25	303.28
44(b)	550	1.030	1725	705.56
44(c)	550	0.908	503.12	139.5
44(d)	550	0.875	1003.12	296.48
44(e)	550	0.760	801.56	171.94

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Figure 6: AC Electrochemical Impedance Spectra at Different Temperatures.

Figure 7: Performance Measurements of Samples 44(a), 44(b), 44(c), 44(d) and 44(e)

Graphical Abstract

Ni contents have been reduced by addition of Zn contents and $\text{Al}_{0.10}\text{Ni}_x\text{Zn}_{0.90-x}$ nanostructured anodes were synthesized. The maximum power density was found to be 705mWcm^{-2} at reduced temperature of 550°C using GDC electrolyte. The new ANZ anodes have been found cost effective as compared to traditional solid oxide fuel cell without sacrificing the power density.