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Structural and Morphological Characterization of $Ni_{1-x}Co_xO$-SDC Nano-Powder Synthesized by Glycine - Nitrate Combustion Synthesis for Its Application in IT-SOFC

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ABSTRACT

$Ni_{1-x}Co_xO$-SDC nano-composite powder was synthesized by single step Glycine Nitrate Combustion Process (GNP). In case of GNP, glycine to nitrate ratio (g/n) plays an important role in deciding the powder characteristics. In the present study, selected (g/n) ratio is 0.97. The powder properties were studied by using X-ray Diffraction Technique (XRD), Scanning Electron Microscope (SEM) and Energy Dispersive X-ray Analysis Technique (EDAX). XRD study revealed, the formation of composite powder consisting of separate phases corresponding to cubic NiO, cubic SDC and cubic Co_2O_4. Crystallite size of the powder is found to increase with the increase in annealing temperature. Calculated crystallite size of the as synthesized powder by using XRD technique is found to be 4.7 nm and 7.5 nm for powder heated at 600 ºC for 2hr. XRD studies confirmed the formation of nano-crystalline powders. SEM studies revealed the highly porous nature of the powder. No other impurity peaks were detected from the EDAX study. Thus nano-crystalline, composite, $Ni_{1-x}Co_xO$-SDC powder was synthesized successfully by using environmental friendly, inexpensive, rapid, single step glycine nitrate combustion method for its possible application to fabricate an anode for Intermediate Temperature Solid Oxide Fuel Cells (IT-SOFCs).

Keywords : $Ni_{1-x}Co_xO$-SDC, Glycine Nitrate Combustion Process, intermediate temperature solid oxide fuel cells, Nickel based anodes

I. INTRODUCTION

Solid Oxide Fuel Cell is an electrochemical device which converts the chemical energy in fuels to electrical energy directly with high system efficiency and low emissions [1]. Ni-based cermet has been considered as the most promising anode material for SOFCs fed with hydrogen due to its high catalytic activity, sufficient electrical conductivity and low cost [2].

Ni - Samaria Doped Ceria (SDC) is reported as potential candidate as anode material [3] for Intermediate Temperature Solid Oxide Fuel Cells (IT-SOFCs). It is reported as most suitable anode for SDC electrolyte - based SOFCs [4]. But Ni-based anodes have the Carbon deposition problem when used with hydrocarbon as a fuel. Also a marked drop in electrical conductivity of the anode cermet has been observed, which is associated with the growth of the Ni particle size [5]. This drop in anode performance may be due to a reduction in both three phase boundary and electrical conductivity.

And hence there is necessity to modify the Ni –SDC anodes, to make them catalytic active, carbon resistant, and these modified anodes will help to improved cell stability, and decrease the polarization resistance [6-8]. Attempts have been made by the researchers to replace Ni by metals and alloys such as Zn [6], Co [7], Mo [8], Mo-Co [9], Fe-Co [10], to maintain the electrochemical catalytic activity and electronic conductivity of NiO-SDC anodes.

Earlier, $Ni_{1-x}Co_xO$-SDC powders have been synthesized successfully by using hydrothermal method [7], but almost negligible reports are present on single step synthesis of $Ni_{1-x}Co_xO$-SDC powder using glycine nitrate combustion process (GNP) method. In GNP glycine is used as fuel and metal nitrates are used as oxidants. These react to undergo redox reaction and forms desired product. In the present work attempts have been made to synthesize and to study the structural and morphological characteristics of $Ni_{0.25}Co_{0.75}O$-SDC Nano-Powder for its application in IT-SOFC.

II. EXPERIMENTAL

2.1 Powder Synthesis

In order to prepare nano-crystalline $Ni_{0.25}Co_{0.75}O$-SDC powder, precursor solution was prepared by dissolving stoichiometric amount of reagent grade nickel (II) nitrate [Ni $(NO_3)_3.6H_2O$], cerium (III) nitrate [$Ce(NO_3)_3.6H_2O$], samarium (III) nitrate [$Sm(NO_3)_3.6H_2O$] and cobalt nitrate $Co(NO_3)_2.6H_2O$ in deionized water with Ni: $Ce_{0.8}Sm_{0.2}O_{1.9}$ as 7:3. Glycine (NH_2-CH_2-COOH) was also dissolved in deionized water separately. Glycine and metal nitrate solutions were then mixed thoroughly to form a homogeneous solution. Alfa Aesar (India) company's chemicals were used. As powder characteristics dependent on nature of the fuel and fuel to oxidant (g/n) ratio [11]. Here selected (g/n) ration is 0.97. The solution was then heated on hot plate at 80 ºC till dark purple color gel forms. The temperature of the hot plate was then raised to 180-220 ºC. Because of this the resultant viscous gel ignited automatically and an intense and self-sustaining flame was formed, and resulted into the formation of foamy, highly porous coffee brown color powder. The conversion process lasts for about a minute. The powder was sintered at 600 ºC for 2 h to remove the residual carbon and promote the crystallization and further used for characterization.

2.2 Powder Characterization

The phase identification of synthesized $Ni_{0.25}Co_{0.75}O$-SDC powders was made with X-ray diffraction (XRD) technique using Phillips PW-1710 diffractometer The micro-structural and compositional analysis of the synthesized powders was conducted with scanning electron microscope (2ELSS EVO series, model EVO 50) equipped with an energy dispersive X-ray (EDS) analyzer (Bruker–AXS, model Quan Tax 200).

III. RESULTS AND DISCUSSION

3.1 XRD Analysis

Fig. 1 shows the comparison of the XRD plot obtained for the as synthesized $Ni_{0.25}Co_{0.75}O$-SDC powder with fuel lean 0.97 ratio and powder heat treated at 600 ºC for 2h. Powder is showing polycrystalline nature. All the peaks have been identified and indexed from the known patterns of the standard data files [12-14].

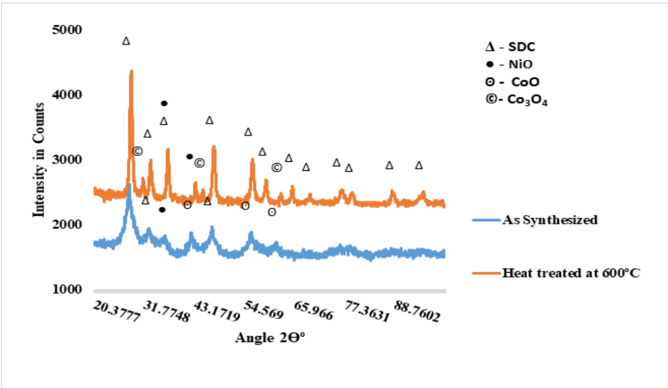

Fig. 1. XRD Patterns of as synthesized $Ni_{0.25}Co_{0.75}O$-SDC powder and powder heat treated at 600 ºC for 2h.

The XRD pattern corresponding to as synthesized powder shows separate peaks corresponding to cubic NiO phase - (111), cubic fluorite SDC phases - (111), (002), (022), (113) and cubic cobalt oxide (CoO) phases - (111), (200), (220). Calculated lattice parameters for NiO, SDC matches well with the reported values [15].
XRD plot obtained for powder heat treated at 600 ºC for 2 h, shows more crystallinity with peaks corresponding to cubic NiO as (111) and (311); cubic SDC as (111), (002), (022), (113), (222), (004), (133), (244), (115), and cubic Co_3O_4 as (440). It is seen that as synthesized powder consists of peaks corresponding to CoO but after heat treatment CoO gets converted to cubic Co_3O_4 [16]. For both the powders (111) peak corresponding to cubic (SDC) is most intense, hence it was further analyzed for getting the crystallite size by using Scherrer's formula. Calculated crystallite size of as synthesized powder and powder heat treated at 600 ºC for 2 h is mentioned in Table 1.

Table 1 Effect of calcinations temperature on the crystallite size of the $Ni_{0.25}Co_{0.75}O$-SDC powder

Calcination Temperature (ºC)	Powder Color	Crystallite size (from XRD) nm
0	Coffee brown	4.7
600	Coffee brown	7.5

From Table 1 it is concluded that, the crystallinity of the powder increases with the increase in the calcination temperature. The lattice constants (a) of Ceria and SDC are reported as 0.5411 nm and 0.5433 nm respectively [17]. Their comparison showed, increase in 'a' value. This increase in 'a' value is due to the substitution of Sm^{3+} at Ce^{3+} site. This gives rise to increase in lattice parameter.
Thus, powder synthesized by using glycine nitrate combustion synthesis is having nanometer size grains. This is due to atomic and molecular level mixing of reagents during combustion synthesis process. The large volume of the gases evolved during the combustion reaction limits the inter particle contact. Also the combustion process occurs at such a fast rate that, sufficient energy and time are not available for long path diffusion of the atoms or molecules as a result of which the initial nano-size of the powder is retained [18].

3.2 Powder Microstructure

Fig. 2 shows Scanning Electron Microscope (SEM) image of as synthesized $Ni_{0.25}Co_{0.75}O$-SDC powder. The powder is found to be highly porous, this is due to release of large amount of gases during combustion synthesis. This helps to restrict the further grain growth of the powder to maintain its nano-crystallinity [18].

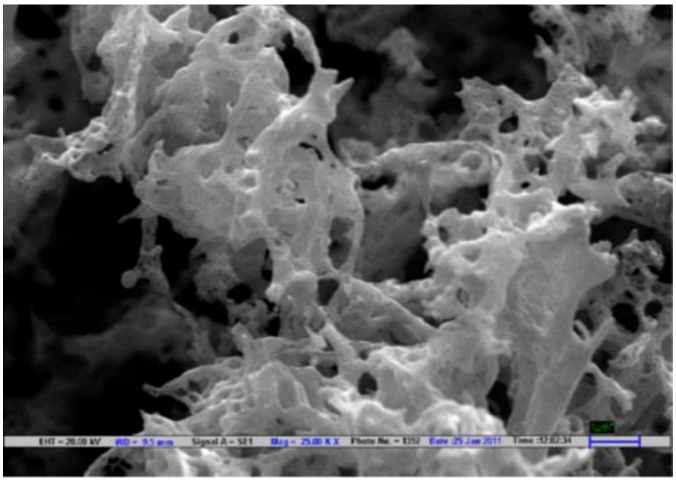

Fig. 2 SEM image of $Ni_{0.25}Co_{0.75}O$-SDC powders heat treated at 600°C for 2 h.

3.3 EDAX Analysis

Fig. 3. shows, the Energy Dispersive X-ray Analysis (EDS) plot of the $Ni_{0.25}Co_{0.75}O$-SDC powder, heat treated at 600 ºC for 2h. From Fig. 3 it is clear that, synthesized powder shows elemental peaks corresponding to Ni, Ce, Sm, Co, O and C only. No peaks other than these were observed, showing the formation of phase pure powder. Presence of C is due to the carbon tape used during sample mounting.

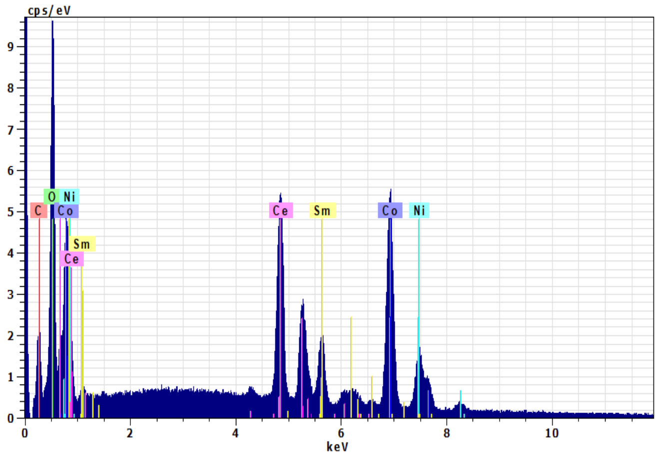

Fig.3 EDAX Plot of $Ni_{0.25}Co_{0.75}O$-SDC powders heat treated at 600ºC for 2h.

IV. CONCLUSION

Nano-crystalline, composite power of $Ni_{0.25}Co_{0.75}O$-SDC have been successfully synthesized by single step glycine nitrate combustion synthesis. We are among the few to report such type of synthesis of $Ni_{0.25}Co_{0.75}O$-SDC. Synthesized powder is highly porous, having high crystallinity, physical and chemical homogeneity and light weight. Nano-crystalline and highly porous nature of the powder will definitely help to increase the three phase boundary (TPB) area, and in turn the performance of anode. Hence $Ni_{0.25}Co_{0.75}O$-SDC nano-powder synthesized by environmental friendly, inexpensive, fast, energy efficient, single step glycine nitrate combustion method is going to be useful to fabricate efficient anodes for intermediate temperature solid oxide fuel cells (IT-SOFCs).

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VI. REFERENCES

[1]. B.C.H. Steele, A. Heinzel, Nature 414 (2001) 245-252.
[2]. S. Alayoglu, K. An, G. Maelaet, S. Chen, F .Bernardi, L.W. Wang, A.E. Lindeman, N.Musselwhite, J. Guo, Z. Lin, M.A. Marcus, G.A. Somorjai, J. Physics Chemistry-C, 117 (2013) 26608-26616.
[3]. B.B. Patil, V. Ganesan, S.H. Pawar, J. of Alloys and Compounds, 460 (2008) 680- 687.
[4]. J. Molenda, K. Swierczek, W. Zajac, Journal of Power Sources, 173 (2007), 657-670.
[5]. D. Simwonis, F. Tietz, D. Stover, Solid State Ionics, 132 (2000) 241.
[6]. X. Zhi, T. Gan, N. Hou, L.Fan, T. Yao, J. Wang, Y. Zhao, Y. Li, Journal of Power Sources, 423 (2019) 290-296.
[7]. G. Ding, T.Gan, J.Yu, P.Li, N.Hou, L.Fan, Y. Zhao, Y.Li, Catalysis Today, 298 (2017)250-257.
[8]. P. Li, B. Yu, J. Li, X. Yao, Y. Zhao, Y. Li, Journal of Power Sources, 320 (2016) 251-256.
[9]. J. Zhao, X. Xu, W. Zhou, Z. Zhu, J. of Materials Chemistry-A,14 (2017) 6494-6503.
[10]. Z. Xie, W. Zhu, B. Zhu, C. Xia, Electrochemica. Acta, 51 (2006) 3052-3057.
[11]. R.D. Purohit, B.P. Sharma, K.T. Pillai, A. K. Tyagi, Materials Research Bulletin, 36(2001) 2711.
[12]. ICSD database Code 28792.
[13]. Data file: PDF#780431
[14]. Data file: PDF#780643
[15]. B. B. Patil, S. Basu, Energy Procedia, 54 (2014) 669-679.
[16]. Data file: PDF#781970
[17]. H. Yoshida, H. Deguchi, M. Kawano, K. Hashino, T. Inagaki, H. Ijichi, M. Horiuchi,K. Kawahara, S. Suda, Solid State Ionics, 178 (2007) 399.
[18]. B. Liu, Y. Zhang, J. of Alloys and Compounds, 453 (2008) 418-422.