Synthesis of SDC Nano-Powder by Glycine - Nitrate Combustion Method

B. B. Patil[1] and S. Basu[2]

1-Department of Physics, Sanjay Ghodawat University, Kolhapur, MH-416118, bharatibpatil@gmail.com

2-Department of Chemical Engineering, Indian Institute of Technology Delhi, New Delhi 110016, sbasu@iitd.ac.in

ABSTRACT

Samaria doped ceria (SDC) nano-powder was synthesized by glycine nitrate combustion process (GNP) with glycine to fuel ratio as 0.85. Effect of annealing temperature on crystallite size of the powder was studied by using XRD technique. XRD study showed, the formation of single phase powder consisting of cubic fluorite SDC phases. The calculated crystallite size was found to be 11 nm at 600 ºC, 22nm at 600 ºC and 36 nm at 1000 ºC. SEM study reveals the formation of porous powder. Thus ultra fine, porous SDC powder was synthesized successfully by cost effective and rapid glycine nitrate combustion method.

Keywords: SDC, chemical synthesis, SOFC

1. INTRODUCTION

In case of Cerium oxide based materials especially, samarium doped ceria have the highest electrical conductivity because of the close ionic radius of Sm $^{3+}$ compared to the radius of Ce $^{4+}$ [1]. The samaria – doped ceria particles with nanometer size can be densified at much lower temperature because of high surface energy of the nano-particles, and expected to provide fast densification kinetics, better properties of the sintered materials contrast to the bulk ones [2].

Earlier SDC powders have been synthesized successfully by the combustion synthesis using different complexing agents/ fuels such as glycine [3], urea, and citric acid [4]. The synthesized powders are phase pure, and have higher surface area than powders prepared by conventional solid state methods. It is inexpensive method and takes short time as compared to sol gel method [5]. In glycine nitrate combustion synthesis (GNP), glycine is employed as fuel and complexing agent and the nitrates as oxidizers. Samaria doped ceria (SDC) nano-powder synthesized by single step glycine nitrate combustion process can be used as electrolytes for IT-SOFC [6]. In this study, work is reported on the synthesis of SDC nano powder by glycine nitrate combustion synthesis and effect of annealing temperature on the crystallite size of the powder.

2. EXPERIMENTAL

A glycine nitrate combustion synthesis method was used to synthesize the nano sized powder of $Ce_{0.8}Sm_{0.2}O_{1.9}$. High purity reagents $Ce(NO_3)_3.6H_2O$, $Sm(NO_3)_3.6H_2O$ and glycine (NH_2CH_2COOH) were used as starting materials. Glycine and metal nitrate solution were mixed with each other to form a clear and homogeneous solution. After homogenization of this solution, the temperature was raised to 80 °C, and the solution maintained under stirring to convert it to a transparent gel. The temperature of the hot plate was raised to 220°C. As soon as the gel was completely dried a combustion reaction took place and results into foamy yellow powder. The powder was heated at 600 °C, 800 °C and 1000°C for 2 h to promote crystallization of the fluorite phase.

The phase identification of synthesized SDC powders was made with the powder X- ray diffraction (XRD) technique using Phillips PW-1710 diffractometer. The micro-structural analysis of the synthesized powder was conducted with scanning electron microscope (2ELSS EVO series, model EVO 50).

3. RESULTS AND DISCUSSION

In this the selected glycine nitrate (g/n) combustion ratio was is 0.85.

3.1 XRD ANALYSIS

Fig. 1 shows the XRD plots obtained for the powder synthesized by using g/n ratio 0.85 and and heat treated at 600 ºC, 800 ºC and 1000 ºC for 2 h. Powder is found to have lime yellow colour. All peaks have been identified and indexed from the known patterns of the standard data files (JCPDS). XRD patterns of all powders shows the peaks (111), (002), (022), (113), (222), (044), (133), (024), (224) corresponding to only cubic phase of $Ce_{0.8}Sm_{0.2}O_{1.9}$, confirming their single phase and polycrystalline nature. The average values of the lattice parameters 'a' for fuel lean, is found to be 5.4446 Å, for cubic SDC. The calculated 'a' matches well with the reported values [7]. (111) peak was most intense hence further analyzed to yield the crystallite size of the material by using the Scherrer's formula.

 DOI : 10.18231/2454-9150.2018.0218

Corresponding crystallite size is found to be 8.46 nm for as synthesized powder. The calculated crystallite size of the powders heated at 600 ºC, 800 ºC and 1000 ºC for 2 h is given in Table 1.

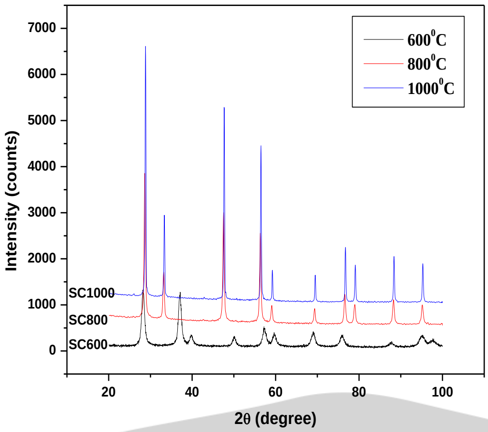

Fig.1. XRD patterns of SDC powder synthesized with g/n ratio 0.85 and heat treated at 600 ºC, 800 ºC, 1000 ºC

The lattice constant of SDC is reported as 0.5433 nm [8]. If lattice constant value corresponding to SDC cubic phase is compared to ceria phase (a = 5.4113 Å), an increase in 'a' value has been observed due to the substitution of Sm^{3+} at Ce^{3+} site since the effective ionic radius of Samarium (0.1219 nm) is higher than that of Cerium (0.1110 nm) [8], resulting in better solubility of Sm_2O_3 in CeO_2 lattice.

Table 1. Effect of annealing temperature on the crystallite size of SDC powder synthesized by using (g/n) ratio as 0.85.

Annealing Temperature (ºC)	Crystallite size (from XRD) in nm
600	11
800	22.1
1000	36.3

3.2 SEM ANALYSIS

POWDER MICROSTUCTURE:

Fig.2 shows SEM image of as synthesized SDC powder with g/n ratio 0.85 and heat treated at 600°C for 2 h. The powder is found to be highly porous.

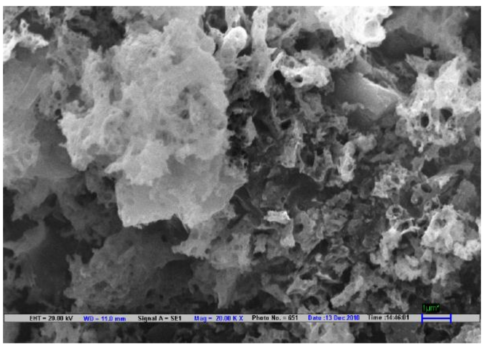

Fig.2. SEM images of as synthesized SDC powder with (g/n) ratio 0.85.

4. CONCLUSION

Single phase samaria doped ceria (SDC) nano-powder was synthesized successfully by glycine nitrate combustion process (GNP).The calculated crystallite size of the powder by using XRD technique is found to increase with the increase in post heating temperature. For 0.85 (g/n) composition, crystallite size was found to be 11 nm at 600 ºC , 22 nm at 800 ºC and 36 nm at 1000 ºC Thus ultra fine, porous SDC powder was synthesized successfully by low cost, rapid, energy efficient, single step glycine nitrate process.

ACKNOWLEDGEMENTS

Authors are thankful to the Ministry of New and Renewable Energy (MNRE), New Delhi and I.I.T. Delhi for providing financial assistance. Author B.B.P is also thankful to Ms.Siddhi Mulik for her kind co-operation and support.

REFERENCES

[1] Kuharuangrong, S. Ionic conductivity of Sm, Gd, Dy and Er-doped ceria. J. of Power Sources 2007; 171:506-510.
[2] Tuller, H. L.Ionic conduction in nanocrystalline materials.Solid State Ionics 2000; 131:143-157.
[3] Xia C. R., Rauch, W., Chen, F. L., Liu, M.L. $Sm_{0.5}Sr_{0.5}CoO_3$ cathodes for low-temperature SOFCs. Solid State Ionics 2002; 149:11-19.
[4] Zhang, Y., Huang, X., Lu, Z., Liu, Z., Ge, X., Xu, J., X., Xin, Sha, X., Su, W. A screen-printed $Ce_{0.8}Sm_{0.2}O_{1.9}$ film solid oxide $Ba_{0.5}Sr_{0.5}Co_{0.8}Fe_{0.2}O_{3-\delta}$ cathode. Journal of P. Sources2006; 160:1217-1220.
[5] Fu,Y. P. Microwave-induced combustion synthesis and ionic conductivity of $Ce_{0.8}(Gd_{0.2-x}Sm_x)O_{1.90}$ ceramics. Ceramics International. 2008 ; 34: 2051-2057.
[6] Zhao, L. Huang, X., Zhu, R. Lu, Z., Sun, W. Y.Zhang, X. Ge, Z. Liu, W. Su, Optimization on technical parameters for Fabrication of SDC film by screen – printing used as electrolyte in IT- SOFC. J. of Phy. and Chem. of Solids 2008; 69:2019-2024.
[7] Patil, B.B., Pawar, S.H. Applied Surface Science. Spray Pyrolytic Synthesis of Samarium DopedCeria ($Ce_{0.8}Sm_{0.2}O_{1.9}$) Films for Solid Oxide Fuel Cell Applications 2007; 253:4994-5002.
[8] Yoshida, H., Deguchi, H., Kawano, M., Hashino, K., Inagaki, T., Ijichi, H., Horiuchi, M. Kawahara, K., Suda, S. Study on pyrolysing behavior of NiO–SDC composite particles prepared by spray pyrolysis technique Solid State ionics. 2007; 178:399-405.

 DOI : 10.18231/2454-9150.2018.0218