

Synthesis of Ba_{0.5}Sr_{0.5}Co_{0.2}Fe_{0.8}O₃ (BSCF) Nanoceramic Cathode Powders by Sol-Gel Process for Solid Oxide Fuel Cell (SOFC) Application

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Abstract

The nano ceramic $Ba_{0.5}Sr_{0.5}Co_{0.2}Fe_{0.8}O_3$ (BSCF) powders have been synthesized by Sol-Gel process using nitrate based chemicals for SOFC applications since these powders are considered to be more promising cathode materials for SOFC. Glycine was used as a chelant agent and ethylene glycol as a dispersant. The powders were calcined at 850°C/3 hr in the air using Thermolyne 47,900 furnace. These powders were characterized by employing SEM/EDS, XRD and TGA/DTA techniques. The SEM images BSCF powder indicate the presence of highly porous spherical particles with nano sizes. The XRD results shows the formation of BSCF perovskite phase at the calcination temperature of 850°C. From XRD line broadening technique, the average crystllite size of the BSCF powders were found to be around 9.15 - 11.83 nm and 13.63 - 17.47 nm for as prepared and after calcination at 850°C respectively. The TGA plot shows that there is no weight loss after the temperature around 450°C indicating completion of combustion.

Keywords: SEM/EDS, XRD, TGA/DTA, Ba_{0.5}Sr_{0.5}Co_{0.2}Fe_{0.8}O₃ (BSCF)

1. Introduction

Solid oxide fuel cells (SOFCs) are considered as one of the most promising energy conversion devices that exhibit advantages such as high efficiency, system compactness and low environmental pollution [1-4]. The SOFCs are expected to be around 50% - 60% efficient at converting fuel to electricity. In applications designed to capture and utilize the system's waste heat (co-generation), overall fuel use efficiencies could top 80% - 85%. Since SOFC operate at very high temperatures ~1000°C, it removes the need for precious metal-catalyst, thereby reducing cost. It allows SOFCs to reform fuels internally, which enables the use of a variety of fuels and reduces the cost associated with adding a reformer to the system. SOFCs are also the most sulfur-resistant fuel cell type; they can tolerate several orders of magnitude more sulfur than other fuel cell types. In addition, they are not poisoned by carbon monoxide (CO), which can even be used as fuel. The traditional SOFC operate at around 1000°C, which causes several problems such as; degradation of performance due to the formation of second phase, sintering of the electrodes and interfacial diffusion

between electrode and electrolyte. The current status of the development of a cell unit is based on yttria-stabilized zirconia (YSZ) solid electrolyte and electrodes consisting of Sr-doped LaMnO3 (LSM-Cathode) and Ni-YSZ cermet (Anode) [5,6]. The state-of-the art cathode material for high temperature (1000°C) operation is LSM perovskite oxide. However, due to its poor ionic conductivity, search of alternative cathode materials for intermediate temperature SOFC (IT-SOFC) is necessary. Therefore, the present trend is to develop reduced temperature SOFC technology which could be operated at below 800°C. So much attention is being made for IT-SOFC in recent years [7-9]. Development of alternative cathode materials with adequate mixed ionic-electronic conductivity (MIEC) is needed to make the IT-SOFC technology successful. In spite of significant efforts have been put until now by various researchers, fundamental questions on the mechanism and kinetics of the O₂ reduction reaction and on the electrode behavior of LSM materials under fuel cell operation conditions still remain unsolved. The Table 1 shows the suitable materials for SOFC components [10].

Kim et al. [11] prepared nano BSCF, LSCF and LSM

Component	Requirements	Preferred Materials	Possible Alternatives
Electrolyte	$\sigma_{\rm i}$ > 0.05 S·cm ⁻¹	ZrO ₂ -Y ₂ O ₃ (3 - 10 mol%)	$ZrO_2-Sc_2O_{3,}$ CeO ₂ -Gd ₂ O ₃ , (Sm ₂ O ₃)
Cathode	>100 S·cm ⁻¹ (electronic/mixed)	$La_{1-x}Sr_{x}MnO_{3}$	$(La_{1-x}Sr_x)Co, FeO_3$
Anode	>100 S·cm ⁻¹ (electronic/mixed)	Ni/ZrO ₂ -Y ₂ O ₃	Ru/ZrO ₂ -Y ₂ O ₃ Ni/CeO ₂ -ZrO ₂ -M ₂ O ₃ cermets
Interconnect	Inert material, high temp. stability	High temp. alloys La _{1-x} (Sr,Ca,Mg) _x CrO ₃	-
Manifold	Non-volatile, inert	Ceramics, metals	
Seal	Non-volatile, inert	Glass, glass-ceramic, Metal/ceramic	-

Table 1. Suitable materials for SOFC components [10].

cathode powers by glycine-nitrate process (GNP) method for metal-supported SOFCs and studied their particle size distribution, SEM, XRD, EIS measurement and electrochemical performance at 1073 K. The powder density obtained were 0.91 W·cm⁻², 0.77 W·cm⁻², 0.69 W·cm⁻² for the above powders respectively.

Park *et al.* [12] studied zinc-doped barium strontium cobalt ferrite (Ba_{0.5}Sr_{0.5}Co_{0.2-x}Zn_xFe_{0.8}O_{3- δ}(BSCZF), x = 0, 0.05, 0.1, 0.15, 0.2) perovskite oxide powders for cathodes of SOFCs application using the eyhylene diamine tetraacetic acid (EDTA)-citrate method with repeated ball-milling and calcining. They were evaluated as cathode materials for SOFC at intermediate temperature (IT-SOFCs) using XRD, H₂-TPR, SEM and electrochemical tests. It is reported that the lowest doping of 0.05 (BSCZF05) resulted in the highest electrical conductivity of 30.7 S·cm⁻¹ at 500°C.

Chen and Shao [13] synthesized BSCF powders by combined EDTA-citrate complexing sol-gel process. The powder was calcined in air for 5 hr, ball milled and then pressed into bar-shaped pellets using a stainless steel die under a hydraulic pressure of 200 MPa and sintered at 1100°C and evaluated their properties using XRD, ESEM and electrical conductivity relaxation (ECR) using Ar and O₂ mixture. Based on chemical bulk diffusion coefficient (D_{chem}), the calculated ionic conductivity of BSCF reached 0.07, 0.25 and 0.96 S·cm⁻¹ at 600°C, 700°C, 800°C respectively.

Sun *et al.* [14] prepared BSCF cathode materials for ceria-composite electrolyte low temperature SOFC application using sol-gel process with polyacrylicaced (PAA) with adjusted pH with ammonia and they were evaluated with XRD, and electrochemical properties. It is reported that the maximum power density achieved was about 800 mW·cm⁻² at 500°C with Ni as anode and Sm_{0.2}Ce_{0.8}O₂ (SDC)-carbonate composite electrolyte and cathode as BSCF.

Recently a new cathode material Ba_{0.5}Sr_{0.5}Co_{0.8}Fe_{0.8}O₃

(BSCF) reported [15], which showed excellent performance as cathode material in conjunction with ceria-based electrolyte at reduced temperature compared with conventional cathode materials. Generally synthesis of BSCF is carried out by the traditional solid-state reaction methods [16] and other wet chemical methods [17-21]. These methods are time consuming and require high temperature calcination (>950°C) and long time to prepare the precursor, which faces the difficulties in preparing homogenous material with good reproducibility. So to overcome such difficulties a sol-gel process is used generally for preparing the pure BSCF nano-powder for SOFC cathode application.

The main design requirements for SOFC Cathode Materials [22] include: 1) High electronic conductivity, 2) Chemically compatible with neighboring cell components (electrolyte), 3) Stable in oxidizing environment, 4) Large triple phase boundary, 5) High ionic conductivity, 6) Thermal expansion coefficient similar to other SOFC materials, 7) Relative simple fabrication, 8) Relatively inexpensive materials.

In the present investigation, nano-crystalline cathode material of BSCF (5528) powders were prepared by the Sol-Gel process since it is a simple and more economical way of making nano powders for SOFC application. While the physical characterization of the powders were carried out using SEM/EDS, XRD techniques, the thermal properties were carried out using TGA/DTA techniques.

2. Experimental Procedure

2.1. Preparation of BSCF Powders

The Ba_{0.5}Sr_{0.5}Co_{0.2}Fe_{0.8}O₃ (BSCF-5528) nano ceramic powders were prepared by modified Sol-Gel Process [23-27] using Ba(NO₃)₂ (BDH), Sr(NO₃)₂ (BDH), Co(NO)₃·9H₂O (Fluka), Fe(NO₃)₃·9H₂O, Citric Acid (BDH), Ethylene

glycol (BDH), Ammonia Solution and distilled water. The precursor solution was prepared by mixing individual aqueous solution of the above chemicals in a molar ratio of 0.5:0.5 and 0.2:0.8 respectively (Table 2). To the mixed all nitrate solutions, required citric acid, ammonia solution and ethylene glycol were added. The citrate/ nitrate ratio used in the present experiments was 0.5. The solution was heated in a pyrex glass beaker on a hotplate using magnetic stirrer until a chocolate colored gel was formed. When heated further, the gel burns to a light and fragile ash. The ash was calcined at 850°C/3 hr in air in a Barnstead Thermolyne 47,900 Furnace (USA). Figure 1 shows the flow sheet for the preparation of $Ba_{0.5}Sr_{0.5}Co_{0.2}$ $Fe_{0.8}O_3$ powder using the Sol-Gel process. Table 2 shows the batch preparation of nano cathode materials (BSCF-5528) by the Sol-Gel process.



Figure 1. Flow Sheet for the preparation of $Ba_{0.5}Sr_{0.5}Co_{0.2}$ Fe_{0.8}O₃ Nanoceramic cathode powders by Sol-Gel process [23-27].

 Table 2. Cathode (BSCF-5528) powders prepared by solgel process.

Sample no	Cathode Powder	With c/n Ratio
#77a,b	$Ba_{0.5}Sr_{0.5}Co_{0.2}Fe_{0.8}O_{3}$	0.50
#78a,b	$Ba_{0.5}Sr_{0.5}Co_{0.2}Fe_{0.8}O_{3}$	0.50

2.2. SEM/EDS Characterization

Small amounts of the samples were spread on adhesive conductive aluminum tapes attached to sample holders, coated with thin films of gold and examined with a FEI Quanta 200 Scanning Electron Microscope. An attached OXFORD INCA250 Energy Dispersive Spectroscopy (EDS) unit was used to determine area and spot elemental compositions. Images at higher magnification were collected with a FEI Quanta 3DF SEM. Imaging was performed in secondary electron (SEI) mode using an accelerating voltage of 20 keV.

2.3. XRD Characterization

A part of the samples were analyzed with a PANnytical X'Pert PRO XRD for phase characterization. The X-ray diffractometry with CuK radiation at 35 kV and 20 mA was used for phase analysis with a diffraction angle 2 theta range 10° - 80° and particle size determination from X-ray line broadening technique using the following Debye Scherrer Equation [28]:

$$t = 0.9\lambda/B \cos \emptyset$$

where t = average particle size in nm, λ = the wave length (0.15418 nm) of Cu K α radiation, B the width (in radian) of the XRD diffraction peak at half of its maximum intensity (FWHM), \emptyset the Bragg diffraction angle of the line, and B is the line width at half peak intensity.

2.4. TGA/DTA Characterization

In order to determine the decomposition behavior of the BSCF Gel samples, around 7 mg - 8mg of the Gel samples were loaded in an alumina crucible and put inside the thermo balance of TG machine (Perkin-Elmer Thermal Analysis Controller TAC7/DX, USA). The thermal decomposition behavior was studied up to 700°C that was raised at a rate of ~10°C per minute. TGA and DTA plots were presented.

3. Results and Discussion

3.1. SEM/EDS Characterization

Figures 2-5 show the SEM images and EDS spectra of





(b)

Figure 2. (a) SEM image of $Ba_{0.5}Sr_{0.5}Co_{0.2}Fe_{0.8}O_3$ cathode powder as prepared (#77a); (b) SEM image of $Ba_{0.5}Sr_{0.5}Co_{0.2}$ $Fe_{0.8}O_3$ cathode powder calcined at 850°C (#77b).



Figure 3. (a) EDS of $Ba_{0.5}Sr_{0.5}Co_{0.2}Fe_{0.8}O_3$ #77a (as prepared); (b) EDS of $Ba_{0.5}Sr_{0.5}Co_{0.2}Fe_{0.8}O_3$ #77b (calcined at 850°C).

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Figure 4. (a) SEM image of $Ba_{0.5}Sr_{0.5}Co_{0.2}Fe_{0.8}O_3$ cathode powder (#78a); (b) SEM image of $Ba_{0.5}Sr_{0.5}Co_{0.2}Fe_{0.8}O_3$ cathode powder (#78b).

nano-sized particles of the Ba_{0.5}Sr_{0.5}Co_{0.2}Fe_{0.8}O₃ (BSCF-5528) powder as prepared samples and calcined at 850°C/ 3 hrs which were prepared with the Sol-Gel process using metallic nitrates. It is seen in the SEM images that the particles are homogeneous with the presence of highly porous spherical particles of nano size with agglomeration. It is noted from the figures that the particle size of the calcined powders at 850°C are larger than the as prepared powders since growth kinetics are favored and the spherical particles became larger with porous structure which resembled the typical cathode structure for SOFC application. The SEM images of BSCF obtained here are similar to the other authors reported [30]. **Figures 3** and **5** show the EDS patterns of $Ba_{0.5}Sr_{0.5}$ $Co_{0.2}Fe_{0.8}O_3$ powders (as prepared and calcined at 850°C). The figures show the presence of Ba, Co, Fe, C, O peaks. The residual C element from the citric acid probably that

had not been combusted fully yet is shown in EDS in the as prepared BSCF powder. However, C content has been reduced in the calcined powders at 850°C as seen in the EDS. Also this would further be reduced by increasing the calcination temperature.

3.2. XRD Characterization

Figures 6 and **7** show the XRD patterns of the as prepared and calcined powders of $Ba_{0.5}Sr_{0.5}Co_{0.2}Fe_{0.8}O_3$ at 850°C respectively. It is seen that as prepared powder is partly amorphous in nature. It is seen that the calcined powder has well crystalline perovskite phase of $Ba_{0.5}Sr_{0.5}$ $Co_{0.2}Fe_{0.8}O_3$. These results are in agreement with the other authors reported elsewhere [14,15,19]. However, a minor amount of $BaCoO_3$ phase is also present in the powders. This impurity will be disappeared and a single cubic perovskite structures may form when the calcination temperature is raised to ~1000°C [18,19]. Also, this could be achieved by using pure metal nitrate chemicals (99.99%). Table 3 shows the average crystallite sizes of BSCF powders calcined at 850°C. It is seen that the average crystllite size of the BSCF powders were found to be around 9.15 nm - 11.83 nm and 13.63 nm - 17.47 nm for as prepared and calcined powders respectively. The results obtained here for BSCF (5528) are in the agreement with the other authors reported elsewhere [29] for BSCF using glycine/nitrate (g/n) ratio of 0.56. A number of factors are responsible for the nano size of the resulting powders. Before the reaction, all the reactants are uniformly mixed in solution at atomic or molecular level. So during combustion, the nucleation process can occur through the rearrangement and short-distance diffusion of nearly atoms and molecules. When the combustion process takes place such a fast rate that sufficient energy and time are not available for long distance diffusion or



Figure 5. (a) EDS of $Ba_{0.5}Sr_{0.5}Co_{0.2}Fe_{0.8}O_3$ cathode #78a (as prepared); (b) EDS of $Ba_{0.5}Sr_{0.5}Co_{0.2}Fe_{0.8}O_3$ cathode #78b (calcined at 850°C).



Figure 6. XRD Patterns of Ba_{0.5}Sr_{0.5}Co_{0.2}Fe_{0.8}O₃ cathode powder (#77) (bottom: as prepared; top: calcined at 850°C).

Sample no.	2Ø	В	Average Particle size,
		FWHM	t (nm)
#77a	24.432	0.680	11.83
#77b	32.08	0.600	13.63
#78a	24.84	0.880	9.15
#78b	31.92	0.468	17.47

Table 3. XRD Data to determine the average particle size of the BSCF (5528) powders prepared by Sol-Gel.



Figure 7. XRD pattern of Ba_{0.5}Sr_{0.5}Co_{0.2}Fe_{0.8}O₃ cathode powder (#78) (bottom: as prepared; top: calcined at 850°C).

migration of the atoms or molecules which may result in crystalline growth. So the initial nano size of the powders is retained after the combustion reaction in sol-gel process. The average particle size of BSCF powder obtained here with c/n of 0.50 are much better than particle sizes obtained for BSCF using combination of PVA and urea as fuel [30].

3.3. TGA/DTA Characterization

Figures 8(a) and **(b)** depict the TGA and DTA plots of the BSCF gels derived from precursor solution in the temperature range of 25° C - 650° C respectively. The endothermic peak observed for BSCF gel in the DTA curves at ~130°C with small weight loss is due to evaporation of residual and hydrated water in the high temperature combustion process. It is seen from 180°C -300°C that two strong exothermic peaks in DTA curves are observed which seemed to be associated with the decomposition/oxidation of the metal-chelates producing oxide phases. No further weight loss is observed after temperatures reaching around 450°C in the TGA plots which indicates the completion of combustion and formation of expected perovskite phase of BSCF. These results are similar to the other authors reported elsewhere [30] for BSCF gels.

4. Conclusions

The following conclusions are drawn from the present investigation:

- The Ba_{0.5}Sr_{0.5}Co_{.2}Fe_{0.8}O₃ (BSCF-5528) nano ceramic powders for cathodes were successfully prepared by Sol-Gel process with c/n ratio of 0.50.
- SEM images of BSCF powders indicate the presence of highly porous spherical particles of nano size in the powders calcined at 850°C.
- XRD patterns show the presence of the perovskite Ba_{0.5}Sr_{0.5}Co_{0.2}Fe_{0.8}O₃ phases.
- TGA plot depicts that there is no weight loss after the temperature of about 450°C indicating completion of the combustion and forming oxide phases.



Figure 8. (a) TGA plot of $Ba_{0.5}Sr_{0.5}Co_{0.2}Fe_{0.8}O_3$ gel; (b) DTA plot of $Ba_{0.5}Sr_{0.5}Co_{0.2}Fe_{0.8}O_3$ gel.

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