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Microwave assisted sintering of gadolinium doped barium cerate electrolyte for intermediate temperature solid oxide fuel cells

Arumugam Senthil Kumar [a, *], Ramamoorthy Balaji [a], Srinivasalu Jayakumar [b], Chandran Pradeep [c]

[a] Department of Physics, PSG College of Technology, Coimbatore, 641 004, Tamilnadu, India
[b] Department of Physics, PSG Institute of Technology and Applied Research, Coimbatore, 641 062, Tamilnadu, India
[c] Department of Physics, Indian Institute of Technology, Madras, 600 036, Tamilnadu, India

HIGHLIGHTS

- To synthesis the composite electrolyte by chemical method and sinter using microwave.
- To reduce the operating temperature of electrolyte for high ionic conductivity in SOFC's.
- To study the phase purity and to develop nanocomposite at reduced temperature.

GRAPHICAL ABSTRACT

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ABSTRACT

In Solid Oxide Fuel Cell (SOFC), electrolyte plays a vital role to increase the energy conversion efficiency. The main hurdle of such electrolyte in fuel cell is its higher operating temperature (1000 °C) which results in design limitation and higher fabrication cost. In order to reduce the operating temperature of SOFC, a suitable electrolyte has been prepared through co-precipitation method followed by microwave sintering of solid ceramic. The calcination temperature for the as-prepared powder was identified using Differential Scanning Calorimetry. The crystal structure of the sample was found to exhibit its orthorhombic perovskite structure. The particle size was determined using High-Resolution Transmission Electron Microscope with uniform in shape and size, match with XRD results and confirmed from structural analysis. Thus, the sample prepared via co-precipitation method and the solid ceramic sintered through microwave can be a promising electrolyte for fuel cells operated at intermediate temperature.
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1. Introduction

Solid oxide fuel cells (SOFCs) have attracted a great deal of consideration among the promising systems for electrical power generation [1], high efficiency energy conversion [2] and friendly environment nature [3]. The most commonly studied electrolyte material with high ionic conductivity was yttrium-stabilized zirconia (8%YSZ), a polycrystalline ceramic added with zirconia as dopant exhibited good stability in both oxidizing and reducing atmospheres. However, YSZ electrolyte supported solid oxide fuel cells have shown higher ionic conductivity at higher operating temperature and faced a lot of problems during elevated temperature operation such as thermal degradation of materials, thermal expansion mismatch between the interconnectors and the interfacial reaction between the electrodes-electrolyte were became a great deal of challenge for such electrolyte material used in the fuel cell for energy conversion [4]. In order to overcome those problems, the operating temperature of fuel cell must be reduced below

* Corresponding author.
E-mail address: senthu.ramp@gmail.com (A.S. Kumar).

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800 °C to achieve the ideal conductivity of 0.1 Scm^{-1}. By reducing the operating temperature will ultimately improve the stability performance of the electrolyte materials and the interconnecting ceramic materials can be replaced by stainless steel, a cost effective one to avoid corrosion for fuel cell environment. Hence, it became an essential need to show the sturdy impetus to develop the suitable oxide ion conducting electrolyte for the solid oxide fuel cells operated at intermediate temperature (400°C- 700 °C) [5]. Cerium oxide (CeO_2) doped with aliovalent cation was considered as one of the most studied material with the cubic fluorite structure of Fm3m space group and shown high ionic conductivity when compared with the state of art YSZ at 1000 °C [6]. Undoped ceria systems are not considered as the best electrolyte for lower temperature operation because they have no defects formation in their crystal structure and the creation of oxygen vacancies are very negligible. But, doped ceria with aliovalent cations like Gd^{3+} and Sm^{3+} ions, creates the oxygen vacancies which aggressively increases the ionic conductivity of doped ceria systems for intermediate temperature operation [7–10]. However, at lower oxygen partial pressure this doped ceria will undergo the reduction process of cerium from Ce^{4+} ions to Ce^{3+} ions, leads to undesired electronic conduction [11]. In order to achieve higher ionic conductivity and to lower electronic conductivity, it is necessary to go for composite materials. In such composites, one phase will increase the ionic conduction and another phase suppresses the electronic conductivity. Hence a strong motivation is required to develop the composite electrolyte for reducing the reduction mechanism of cerium by suppressing the electronic conduction through one phase of a solid electrolyte and by improving the migration of oxide ions through the other phase in the composite matrix, favors mixed ionic conduction (H^+, O_2^-) may result in higher open cell voltage for fuel cells operated at intermediate temperature [12,27].

Ceria as well as barium cerate based electrolytes were usually prepared by using its water soluble salts, such as oxalates, nitrates, and acetates. These doped ceria based electrolytes samples was processed and synthesized by different techniques such as spin coating method [2], conventional solid-state method [9], pechini method [10], carbonate co-precipitation [14], citrate complexation method [15],precipitation [16], oxalic co-precipitation [17], freeze drying [18], combustion [19] gel-casting process [20], hydrothermal method [21] co-precipitation [22] and glycine-nitrate process [23] were reported in the literature, particularly on Gd^{3+} ion doped ceria system. Among the various methods of research groups, a very little work have been carried out to prepare barium cerate electrolyte through wet chemical route and the microwave assistance for reducing the sintering temperature of solid electrolyte was not studied in detail so far. In order to synthesis composite electrolyte, a chemical route was adopted to achieve nanoparticles of CG-BCG matrix phases with less agglomeration. The nanosized particle helps to distribute the individual phase uniformly. Otherwise, a channel of single phase may form and affects the ionic conductivity by increasing the electronic conduction in such composite electrolyte [24]. The composite electrolyte prepared through solid state method leads to segregation of phases which subsequently results in increasing the electronic conductivity and decreases the OCV of the fuel cells. So the chemical route was followed to synthesis composite electrolyte [1,24]. In addition, the wet chemical method has been considered as one of the well established and widely adopted synthesis techniques of low cost and has more promising advantages in preparing the well-defined nanoparticles for good densification of solid ceramics [15].

Microwave sintering method was adopted to reduce the sintering temperature of electrolyte and to prevent the phase segregation in composites matrix effectively by heating the solid electrolyte very rapidly. The development of matrix phase in composite will acts as a productive (or) blocking layer to suppress the electronic conductivity in CG phase and increase ionic conduction in BCG phase as shown in Fig. 1. The necessity for fast sintering of the pellets is to reduce the evaporation of barium content from the composition of the electrolyte at elevated temperature and the sintering process was carried out using microwave furnace with a programmable heating rate of 45 °C/min and minimum soaking time of 20 min at 1400 °C to avoid the barium loss. In microwave sintering (MS), the material gets heated directly due to energy conversion process taking place at the molecular level [25,26]. The transformation the heat energy to the materials is mainly due to the interaction of electromagnetic wave taking place very rapidly, results in the reduction of sintering temperature of electrolyte and decreases the energy consumption significantly. Whereas, in conventional sintering the energy transferred from heating element of the furnace to the sample surface and the longer soaking time of the electrolyte leads to the formation of cracks on the surface of the solid electrolyte [26]. Chemical stability of electrolyte in Co_2 atmosphere and mechanical strength of solid ceramic can be improved by increasing the development of matrix phase in composite electrolyte through microwave sintering for fuel cell operated at lower temperature [27]. Moreover, barium cerate composites have attracted its choices as a best electrolyte with increased ionic conductivity at a higher operating temperature and therefore it becomes a very crucial need to reduce the processing and operating temperature of such barium cerate based electrolyte to be operated at the intermediate temperature for SOFC application [13,14].

Hence, the objective of the present work is to prepare gadolinium doped barium cerate powder by means of co-precipitation technique and to develop the composite CG-BCG matrix by sintering the solid electrolyte very rapidly through microwave sintering technique. The as-prepared powder was calcined and pellets were sintered using microwave furnace followed by its characterization using DSC, TG-DTA, XRD and HRTEM techniques and results were presented.

2. Experimental work

2.1. Powder synthesis

The Gadolinium doped barium cerate (BCG) powder has been synthesized via chemical route (Co-precipitation technique). Barium nitrate (99.5% pure purchased from Sigma–Aldrich, Italy) and Cerium nitrate hexahydrate (99.5%, Sigma–Aldrich, Italy) were

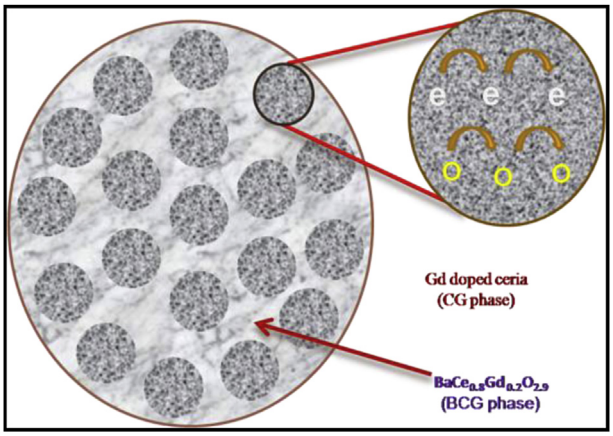

Fig. 1. CG-BCG composite matrix phase prediction.

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