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Preparation and characterization of 10 mol% Gd doped CeO_2 (GDC) electrolyte for SOFC applications

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Abstract

Ceria-based materials are prospective electrolytes for low and intermediate temperature solid oxide fuel cells. In the present work, fully dense CeO_2 ceramics doped with 10 mol% gadolinium ($Gd_{0.1}Ce_{0.9}O_{1.95}$, GDC) have been prepared with a Pechini method. Characterization studies were realized with thermo-gravimetric analysis (TGA), differential thermal analysis (DTA), mass spectroscopy (MS), high temperature FT-IR (HT-FTIR) and X-ray diffraction analysis (XRD). A single-phase with a fluorite type structure was found to form at a relatively low calcination temperature of 500 °C. Dense GDC pellets having 98% of the relative density were obtained at sintering temperature of 1400 °C/6 h, which gave significantly higher total ionic conductivity of 3.4×10^{-2} S cm^{-1} at 500 °C in air. The present work showed that the Pechini method is a relatively low-temperature preparation technique to synthesize $Gd_{0.1}Ce_{0.9}O_{1.95}$ powders that provided high sinterability and good ionic conductivity.

Keywords: CeO_2; $Gd_{0.1}Ce_{0.9}O_{1.95}$; Ionic conductivity; Pechini method

1. Introduction

Solid oxide fuel cells, SOFCs, have a great potential for being the cleanest, most efficient and versatile technology for the conversion of chemical to electrical energy. The cost of the current SOFC systems has a restrictive effect on a wide range of commercial applications. In order to be economically competitive, the cost of the materials and the fabrication should be considerably reduced. One way for cost reduction is to lower operating temperature so that interconnection, i.e. heat exchanges between the structural components will decrease. Hence, working with lower temperatures provides utilization from comparatively cheap metal components in low and intermediate temperature SOFCs. For this purpose, by using high ionic conductivity electrolyte at low temperatures, operating temperatures can be reduced [1,2].

Most commonly, solid electrolyte materials which are used in SOFC are yttria stabilized zirconia, doped ceria (e.g. gadolinia doped and samaria doped ceria), stabilized Bi_2O_3 and strontium/magnesium doped lanthanum gallate. A typical SOFC has an electrolyte with yttria-stabilized zirconia (YSZ). YSZ-based SOFC is required to operate at high temperature of 800–1000 °C. However, at high operating temperatures, some destructive factors such as thermal mismatch between materials, interfacial reactions at electrode/electrolyte interfaces and electrode/interconnector interfaces, may occur. For this reason the yttria-stabilized zirconia electrolytes should be replaced with the alternative solid electrolytes having a higher conductivity at lower temperatures. Ceria based electrolytes having higher electrical conductivity than that of yttria-stabilized zirconia at lower operating temperatures (500–800 °C) have received much attention in recent years [3].

Ceria (CeO_2) compounds doped with rare-earths which are fluorite type oxides with oxygen vacancies show a higher oxide ionic conductivity than yttria stabilized zirconia in oxidizing atmospheres. Moreover, doped ceria have lower cost in comparison with lanthanum gallate based electrolytes. Doping with R^{3+} ions (such as R=Gd, Sm, Y etc.) in the crystal structure of ceria was found to

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increase the concentration of oxygen vacancy due to the reduction of Ce^{4+} to Ce^{3+}. Subsequently, as reported in the previous work, doping with Gd enhanced the ionic conductivity of ceria [4,5]. Gd^{3+} ion doped ceria has attracted much attention as a potential electrolyte for fuel cells at low operating temperatures [4,6,8–11]. Among doped ceria compounds, especially Sm_2O_3 and Gd_2O_3 doped ceria compounds were remarked to possess the highest conductivity [7,8,12].

Therefore, researchers have recently focused on the ceria based electrolytes. Since ceria based electrolytes have higher densities, they provide SOFCs to work under lower operation temperatures. There are several techniques for preparing Gd_2O_3 and Sm_2O_3-doped ceria nanopowders such as citrate complexation [11], combustion [10,13], hydrothermal synthesis [14], microwave-induced combustion [15], co-precipitation [16,17] and sol–gel [18]. The main advantage of Pechini method which is a modified sol–gel technique has the capability of producing ultrafine powders with high purity and homogeneous phase composition at lower temperatures.

This study aims to prepare Gd doped ceria ultra-fine crystallites using Pechini method as an electrolyte for low temperature SOFC applications. Thermal decomposition of these products was investigated in dry air atmosphere by simultaneous thermo-gravimetric analysis (TGA), differential thermal analysis (DTA) and mass spectroscopy (MS). Crystallite structures and morphologies of these products were characterized by XRD and SEM techniques. Moreover, bond characterization of the samples was performed by using high temperature Fourier Transform-Infrared Spectroscopy (HT-FTIR) technique. The electrochemical properties of dense GDC samples sintered at 1300 and 1400 °C were also studied with impedance spectroscopy at 250–550 °C in air.

2. Experimental

Cerium (III) acetate hydrate ($Ce(CH_3CO_2)_3 \cdot 1.5H_2O$, 99.9%, Aldrich) and gadolinium (III) acetate hydrate ($Gd(CH_3CO_2)_3 \cdot H_2O$, 99.9%, Aldrich) were used as metal precursors and ethylene glycol (R.P. Normopur), citric acid (Boehringer Ingelheim) were selected for the polymerization treatment. Acetates were dissolved in de-ionized water individually and then the solutions were mixed in a beaker. Anhydrous citric acid was dissolved in de-ionized water and then was added to the cation solution. The molar ratio of total oxide (TO): citric acid (CA) and ethylene glycol: citric acid was selected as 2:1, 4:1, respectively. After homogenization of this solution, temperature was raised to 80 °C and the solution was kept for 5 h at this temperature with magnetic stirring to remove excess water. During this time, the color of the liquid turned from white to light brown. Then the obtained light brown gel was cooled down to room temperature. The initial thermal decomposition of the precursor was dried 110 °C for 24 h (see Fig. 1).

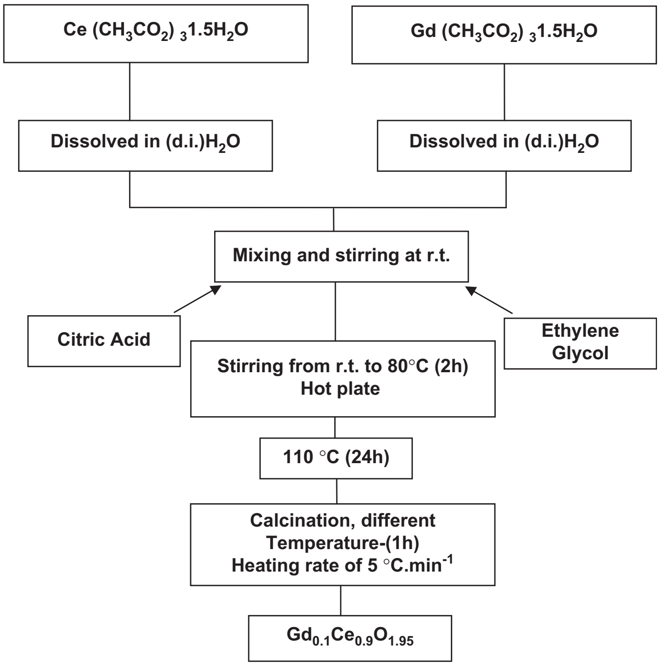

Fig. 1. Flow chart of the Pechini method.

The thermal behavior of $Gd_{0.1}Ce_{0.9}O_{1.95}$ (GDC) powders was carried out with SII Exstar 6000 TG/DTA 6300 from room temperature to 850 °C at a heating rate of 5 °C min^{-1} in air. DTA, TGA and MS analysis were carried out for the obtained powders. Afterwards, the conversion of the so-prepared amorphous precursors into crystalline gadolinium-doped ceria was achieved by heating the dried solids at a heating rate of 5 °C min^{-1} to 200, 400, 500 and 700 °C, and keeping them at these temperatures for 1 h. XRD technique was used to determine the crystal structure of the crystalline phases. X-ray spectra of gadolinium-doped ceria powders were obtained over the 2θ range of 10–90° by using Rigaku D/Max-2200 PC X-ray diffractometer with CuKα radiation. The average crystallite size, D, of the calcined powders was estimated using the Debye–Scherrer equation

$$D = 0.9\lambda/\beta \cos\theta, \quad (1)$$

where λ is the wavelength of the X-rays (1.5418 Å), θ is the scattering angle of the main reflection (111) and β is the corrected peak at full width at half-maximum (FWHM) intensity.

To measure the density of the calcined powders, the powders were calcined at 1000 °C and pressed to disk at 200 Mpa with cold isostatic press (CIP). The compact disk of $Gd_{0.1}Ce_{0.9}O_{1.95}$ powders were then sintered at 1200, 1300, 1400 °C for 6 h with a heating rate of 5 °C min^{-1}. The densities (D_{rs}) of the resultant sintered disks were determined by the Archimedes's method;

$$D_{rs} = W_{obj}g/W_1 - W_2, \quad (2)$$

where W_{obj} is the dry weight, W_1 is the wet weight (water in the body), W_2 is the body's submerged weight without fine wire and g is the density of the solvent (water at 25 °C, 0.997 g cm^{-3}). The value of the theoretical density was calculated as 7.21 g cm^{-3}.

The morphology of the sintered samples according to sintering temperature at 1200, 1300, and 1400 °C was determined by FEI Quanta FEG 450 scanning electron microscopy. The oxygen conductivities of the sintered GDC powders were measured using AC Impedance analyzer (Solartron 1260FRA and 1296 interface) at disk-shaped GDC sample prepared by cold isostatic pressing (CIP) at 95 MPa.

3. Results and discussion

3.1. TG-DTA and MS analysis

Thermal decomposition of $Gd_{0.1}Ce_{0.9}O_{1.95}$ powders was investigated in dry air atmosphere by simultaneous TGA, DTA and MS techniques and the results are shown in Figs. 2 and 3. As can be seen from the TG curve in Fig. 2, the thermal decomposition of $Gd_{0.1}Ce_{0.9}O_{1.95}$ takes place in three steps. In the first step, a minor weight loss of 5.5% between 40 and 138 °C which is related to the loss of moisture and the dehydration of acetate. Due to the combustion of organic materials, in the second step, the slight weight loss is observed between 138 and 223 °C and then a drastic weight loss of 31% at 250 °C is observed due to the combustion of the residual organic materials in the sample.

The MS data (see Fig. 3) show that the gases evolved during the combustion reaction were identified as a mixture of acetone (main ions of m/z 15, 43, 58 assigned to CH^{3+}, CH_3CO^+ and $(CH_3)_2CO^+$, respectively) and carbon dioxide (m/z 44 ions assigned to CO_2^+, respectively). However, the MS data confirmed that CO, CO_2 gases were predominant gases during the combustion reaction. Similar results were found by Arii et al. [19].

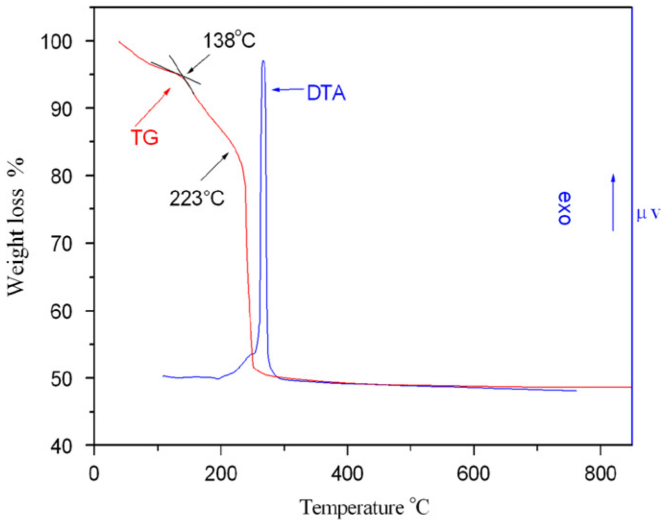

Fig. 2. TGA–DTA curves of thermal decomposition of crystalline GDC powder precursors at a heating rate of 5 °C min^{-1} in dry air.

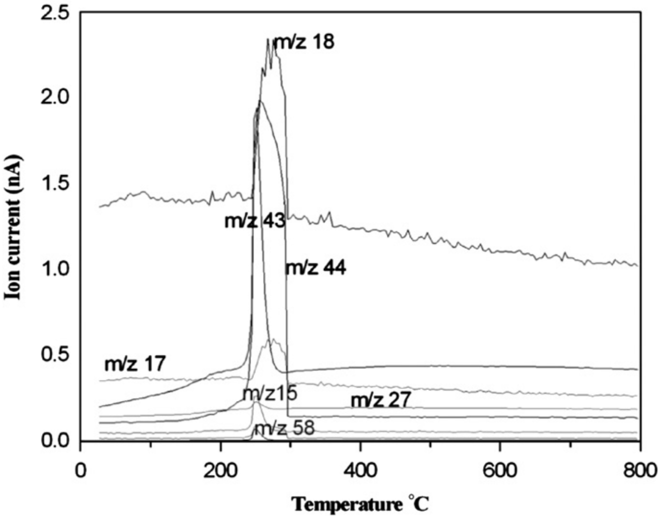

Fig. 3. Mass spectra of GDC powder in dry air.

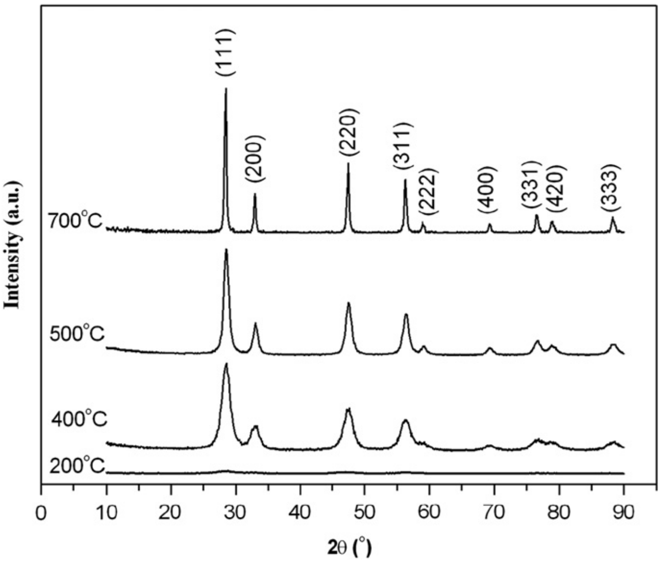

Fig. 4. XRD patterns of GDC powders calcined at 200, 400, 500, and 700 °C 1 h in air.

The total weight loss was found to be 51% from TGA curves in Fig. 2 (up to 320 °C). An absence of any weight loss in TGA and endothermic or exothermic peak in DTA above 500 °C confirms formation of $Gd_{0.1}Ce_{0.9}O_{1.95}$ as a decomposed product. This finding shows that the appropriate calcination temperature for the preparation of the GDC powders is likely to be 500 °C. The XRD results shown in Fig. 4 also prove this finding.

3.2. X-ray analysis and crystallite size results

X-ray diffraction patterns obtained for the GDC powders calcined at 200, 400, 500, 700 °C are shown in Fig. 4. Amorphous phase was observed after calcination of GDC powder at 200 °C. The main diffraction peaks of GDC start to be resolved after calcination at 400 °C. However, as can be clearly seen from the XRD patterns in Fig. 4, these peaks are relatively broad, indicating that the crystallite size was very small. But, these peaks became sharper and narrower with increase in heating temperature, showing that the crystal sizes increased. Therefore, the crystallinity of the powder became better during the calcination process.

After calcination at 500 °C, almost all the characteristic diffraction peaks corresponding to the fluorite structure of CeO_2 (JCPDS Card No. 34-394) was observed. Crystalline phases corresponding to Gd_2O_3 could not be found at any calcination temperatures for $Gd_{0.1}Ce_{0.9}O_{1.95}$ powders, indicating the direct formation of solid solution. These XRD results were also supported with the MS results (see Fig. 3) which show that there is no ion current above 500 °C. The crystallite sizes as calculated by the Debye–Scherrer equation were found to be 11, 13, 55 nm for calcined powder at 400, 500, 700 °C, respectively.

3.3. HT-FTIR analysis results

HT-FTIR spectroscopy analysis was performed to characterize GDC gel precursor (dried at 110 °C) under nitrogen gas atmosphere (gas flow rate: 100 ml min^{-1}) from 25 °C up to 700 °C. Fig. 5 shows in-situ high temperature FTIR results. A characteristic broad band in the range of 3000–3700 cm^{-1} was related to the presence of hydroxyl groups (O–H) at 25 °C. The characteristic absorption lines of the infrared rays are useful for identifying the functional groups of the organic compounds. The band at about 1610 cm^{-1}, which decreased with increase in heating temperature, is attributed to COO^- vibration (the carboxylate salt group). The vibrations of the carboxylic groups ($-COO^-$) were located at around 1400 cm^{-1} [20]. These two groups of bands disappeared during heating process. After decomposition at 150 °C, intensity of the band related to hydroxyl groups (O–H) decreased significantly with increasing temperature. Most of the infrared transmission bands related to the carbon–oxygen bonds disappeared at 500 °C. However, only the bands related to characteristic metal–oxygen bond were detected at 500 °C and 700 °C [21]. The high temperature FTIR results were in good agreement with the TG-DTA, X-ray diffraction data.

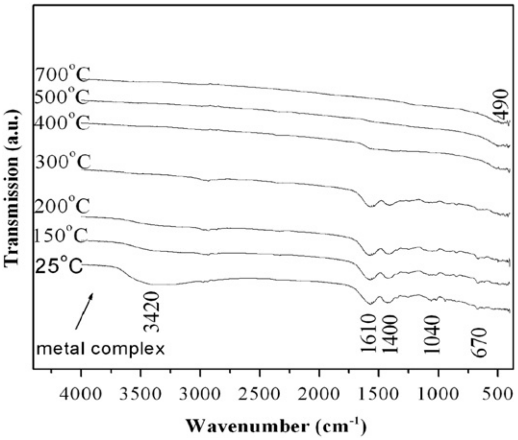

Fig. 5. The high temperature FTIR spectra of GDC powders dried at 110 °C in air.

3.4. Density results

Fig. 6 shows the relative densities of the compact disks of GDC powders. As the sintering temperature increased, GDC samples became denser. The densities of $Ce_{0.9}Gd_{0.1}O_{1.95}$ samples sintered at 1400 °C for 6 h was 98% of theoretical density. This value is comparatively higher than those of the reported results [6,22–24]. This indicates that the $Ce_{0.9}Gd_{0.1}O_{1.95}$ powders prepared with the Pechini method enhances the sinterability.

3.5. SEM analysis

The SEM micrograph of the calcined at 500 °C $Ce_{0.9}Gd_{0.1}O_{1.95}$ powders is given in Fig. 7 and the SEM micrographs of the fractured surface of sintered $Ce_{0.9}Gd_{0.1}O_{1.95}$ pellets are shown in Fig. 8(a–d). The micrographs of the samples calcined at 500 °C indicated that the structure is composed of loosely agglomerated spheroid crystals. These images correspond to the sintering temperatures of 1200, 1300 and 1400 °C with a dwelling time of 6 h. It can be seen from the SEM micrographs given in Fig. 8(a) that the sintered $Ce_{0.9}Gd_{0.1}O_{1.95}$ pellets have the grain size of approximately 0.5 µm at 1200 °C for 6 h. The micrographs shown in Fig. 8(c,d) clearly show that the grains are denser and the microstructure has no porosity except the presence of few residual pores.

The samples sintered at 1400 °C have fully dense structure (98% of the theoretical density) with relatively small volume of grain boundary. Therefore the dense $Gd_{0.1}Ce_{0.9}O_{1.95}$ pellet sintered at 1400 °C had fairly high total conductivity value.

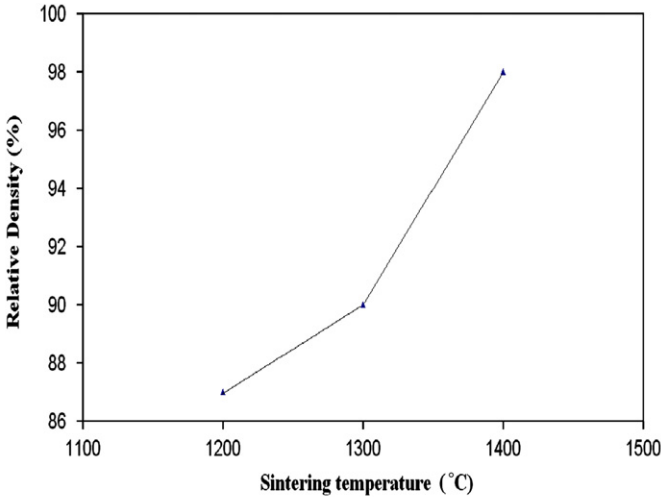

Fig. 6. Relative density as a function of sintering temperature.

3.6. Conductivity results

The complex impedance spectra measured at 350 °C in air for GDC sintered at 1300 °C and 1400 °C are shown Fig. 9(a) and (b). In Fig. 9(a) two well-defined semi-circular arcs which could be confidently assigned as the crystal lattice (bulk) and grain boundary for the GDC electrolyte were observed at 350 °C in air. As can be seen in Fig. 9(a) and (b), grain boundary resistance decreased as sintering temperature increased from 1300 to 1400 °C. As seen in Fig. 8(c) in the SEM micrograph, a highly dense structure with barely noticeable grain boundary was obtained. As the operating temperature was increased above 350 °C, the arc corresponding to the bulk contribution was seen to disappear. Two semi-circles merge together and the bulk and grain boundary processes cannot be completely separated. At higher temperatures, above 550 °C, the grain boundary arc also disappeared. It was observed that the $Ce_{0.9}Gd_{0.1}O_{1.95}$ samples sintered at 1400 °C showed a higher conductivity, due to high solubility limit of Gd-ions in ceria and the increased sintered density.

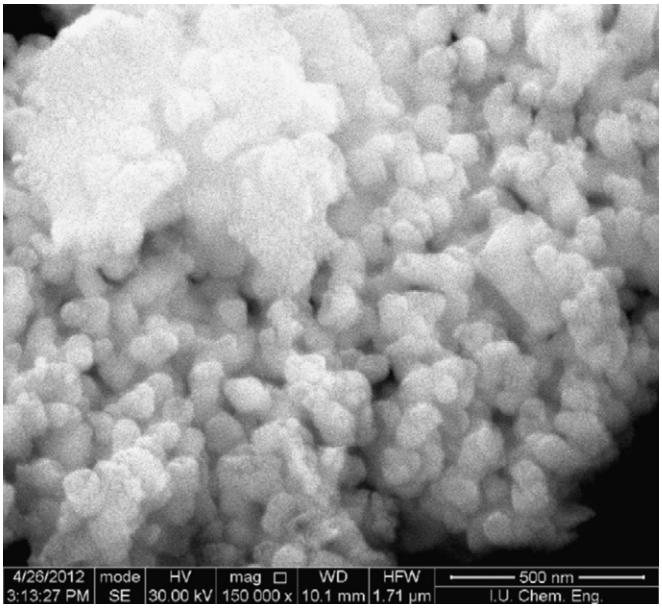

Fig. 7. SEM micrograph of surface of GDC samples calcined at 500 °C for 1 h (× 150.000).

The total conductivity value of $Gd_{0.1}Ce_{0.9}O_{1.95}$ ceramics sintered at 1400 °C, owing to the relatively high density, is 3.4×10^{-2} S cm^{-1} at 500 °C, which is usually considered to be the promising operational temperature for low-temperature SOFCs with ceria-based electrolytes. The increased electrical conductivity may be due to the low grain size, the homogeneous distribution of Gd^{3+} in ceria lattice and its high ionic-mobility. The ionic conductivity values obtained at 500 °C in our work was found to be better than the ones reported earlier by other researches at

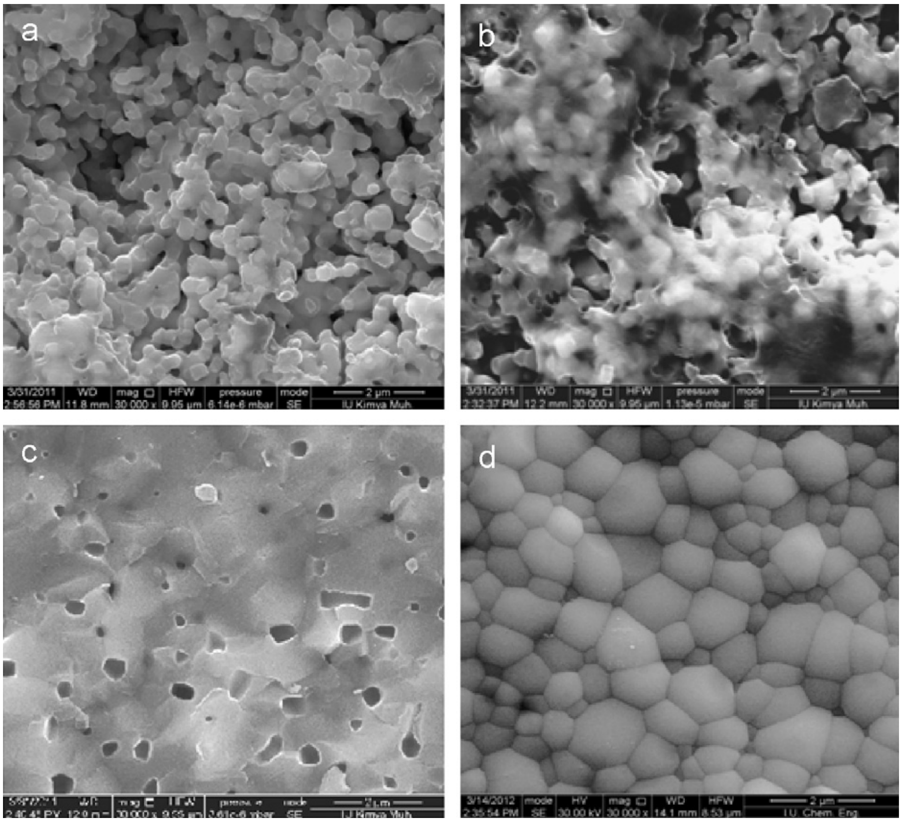

Fig. 8. SEM micrographs of cross-sectional fracture surfaces of GDC samples sintered at (a) 1200 °C, (b) 1300 °C, (c) 1400 °C for 6 h and (d) the surface of GDC pellet sintered at 1400 °C for 6 h (× 30.000 for all samples).

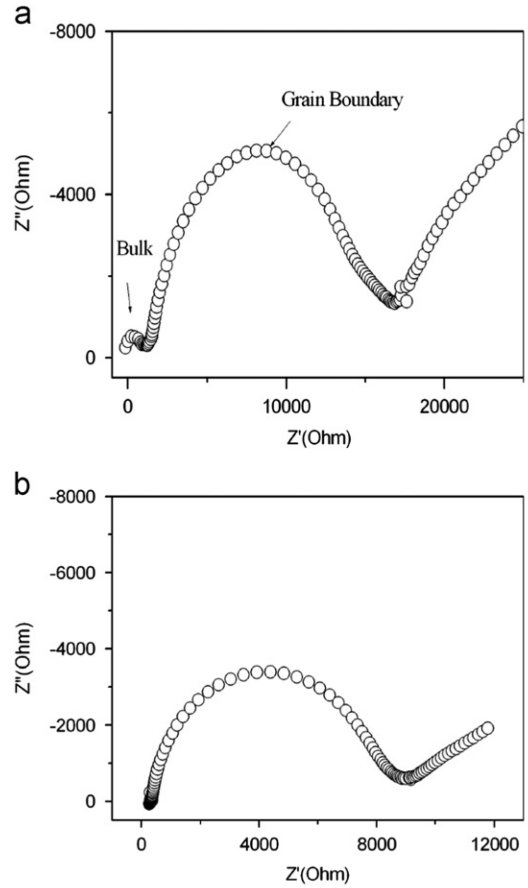

Fig. 9. Impedance spectra measured at 350 °C in air for gadolinia-doped ceria sintered at (a) 1300 °C and (b) 1400 ºC.

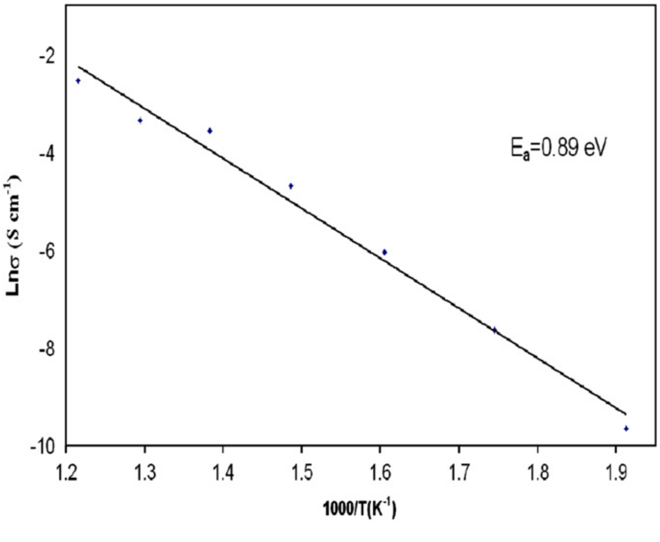

Fig. 10. Arrhenius plot of the total conductivity (σ) of $Gd_{0.1}Ce_{0.9}O_{1.95}$ ceramics in air.

600 °C and 700 °C for similar compositions of gadolinium doped ceria [6,23,25–28].

Conductivity values for bulk and grain boundary contributions were calculated from the corresponding resistances obtained by using the impedance spectra. Arrhenius plot of the total conductivity were calculated using these data and presented in Fig. 10. As seen in Fig. 10, the activation energy was calculated as 0.89 eV. The activation energy value obtained in this work is in good agreement with the values reported earlier. [24,29].

4. Conclusions

In order to obtain a fluorite-like type single-phase structure, 10 mol% gadolinia doped ceria powders have been synthesized via Pechini process in which a relatively low calcination temperature (500 °C) was required compared to the conventional solid state reaction techniques. This cheap method provides an efficient way to produce ceramics with high densities as a result of sintering at 1400 °C (98% of theoretical value). Also GDC powders sintered at 1400 °C gave a significantly higher total ionic conductivity of 3.4×10^{-2} S cm^{-1} at 500 °C in air atmosphere. High density, small grain size and the dopant ion mobility were assumed to be the probable factors for the increased conductivity. The ionic conductivity value obtained at such a lower operating temperature, at 500 °C in our work was found to be higher than those of the values reported earlier in the literature [23,28,29]. In our research, for the electrolyte of $Gd_{0.1}Ce_{0.9}O_{1.95}$ produced with a Pechini method, the optimum total conductivity value was also obtained at such a lower operating temperature, 500 °C. This result showed that $Gd_{0.1}Ce_{0.9}O_{1.95}$ can be used as an electrolyte material in low (500 °C) operating temperature SOFC electrolytes. Ultra-fine $Gd_{0.1}Ce_{0.9}O_{1.95}$ powder with fluorite-like type single-phase structure was synthesized successfully with the Pechini method at low calcinations temperatures and characterized with TGA/DTA-MS, XRD and HT-FTIR techniques.

Acknowledgments

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