

Synthesis of nanocomposite ceramic oxide materials

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Abstract

Nanocrystalline materials play an important role in the field of material chemistry. They are having various applications. Now a day nanocrystalline ceramic based materials is very popular in the fuel cells application, due to low temperature. We have synthesised nanocomposite ceramic oxide materials and characterise through TGA analysis.

Keywords: nanocrystalline, TGA, ceramic, analysis

1. Introduction

Nanocrystalline materials are interesting from the point of view of fundamental research for studying interface characteristics. Nanocrystalline materials are expected to play an important role in developing new materials exhibiting unusual properties [1].

Ceria doped materials as electrolytes

The ceria-based electrolytes for SOFCs have exhibited good performance improvements that have demonstrated a breakthrough in SOFC research. New research activities on the ceria-based materials have shown super ionic and dual or hybrid H^+/O^{2-} conduction with conductivity of 0.1 Scm^{-1} below 600°C [2]. The ionic conductivity of doped ceria is nearly an order of magnitude greater than that of stabilized zirconia for similar doping conditions.

2. Experimental Method

2.1 Sample preparation

Initially, the precipitating solution (sodium hydroxide) was mixed with 2 ml of 10% CTAB (as surfactant material). In order to avoid agglomeration this surfactant added to solution. To this mixture, $\text{Ce}(\text{NO}_3)_3$, $\text{Gd}(\text{NO}_3)_3$ and $\text{Y}(\text{NO}_3)_3/\text{Sm}(\text{NO}_3)_3$ solutions were subsequently added one by one drop wise. They were mixed thoroughly by a magnetic stirring apparatus (1,000 rpm) at room temperature for 2-3 hours. The pH was maintained at greater than 9 during the experiment by the addition of alkali. The resultant yellow-colored precipitate ($\text{Ce}(\text{OH})_4 + \text{Gd}(\text{OH})_3 + \text{Y}(\text{OH})_3$) or ($\text{Ce}(\text{OH})_4 + \text{Gd}(\text{OH})_3 + \text{Sm}(\text{OH})_3$) with CTAB was filtered and then washed with deionized water and ethanol in the ratio of 9:1 (v/v) five to ten times. The product was dried at 50°C to 100°C for 24 h. The resultant material was calcined at 300°C , 450°C , 600°C and 750°C for 2 h each. During calcination, the surfactant was removed, and phase-pure, yellow-colored nanocrystalline material was formed

3. Results and Discussion

3.1 Thermo Gravimetric Analysis (TGA)

The precursor samples were subjected to thermo gravimetric analysis (TGA) to study the thermal decomposition process. The dried precursor precipitate materials [$\text{Ce}(\text{OH})_4 +$

$\text{Gd}(\text{OH})_3 + \text{Y}(\text{OH})_3$, $\text{Ce}(\text{OH})_4 + \text{Gd}(\text{OH})_3 + \text{Sm}(\text{OH})_3$ and $\text{Ce}(\text{OH})_4 + \text{Gd}(\text{OH})_3$] with C-TAB] with an initial mass 8-13 mg was placed in an open platinum crucible. The mass scale of the instrument was calibrated with standard reference materials based on the measurement of Curie Points (T_c) of alumel alloy ($T_c = 427.35 \text{ K}$) and nickel ($T_c = 628.45 \text{ K}$). The TGA patterns obtained with the precursor precipitate materials [$\text{Ce}(\text{OH})_4 + \text{Gd}(\text{OH})_3 + \text{Y}(\text{OH})_3$, $\text{Ce}(\text{OH})_4 + \text{Gd}(\text{OH})_3 + \text{Sm}(\text{OH})_3$ and $\text{Ce}(\text{OH})_4 + \text{Gd}(\text{OH})_3$] with C-TAB] are indicated in Figure 1.

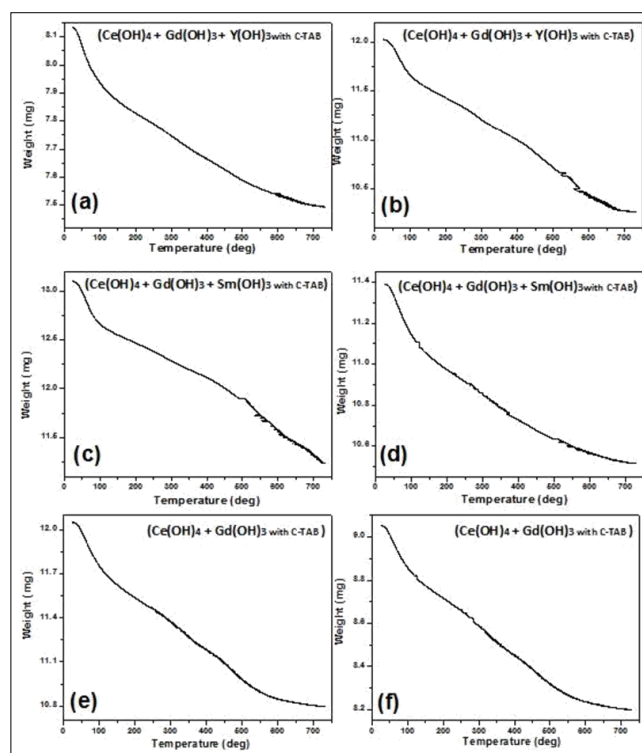
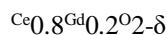


Fig 1: TGA spectra obtained with the doped ceria precursor materials (a)

$\text{Ce}_{0.9}\text{Gd}_{0.1}\text{O}_{2-\delta}$ - $\text{Ce}_{0.9}\text{Y}_{0.1}\text{O}_{2-\delta}$, (b) $\text{Ce}_{0.8}\text{Gd}_{0.2}\text{O}_{2-\delta}$ - $\text{Ce}_{0.8}\text{Y}_{0.2}\text{O}_{2-\delta}$,
(c) $\text{Ce}_{0.9}\text{Gd}_{0.1}\text{O}_{2-\delta}$ -
 $\text{Ce}_{0.9}\text{Sm}_{0.1}\text{O}_{2-\delta}$, (d) $\text{Ce}_{0.8}\text{Gd}_{0.2}\text{O}_{2-\delta}$ - $\text{Ce}_{0.8}\text{Sm}_{0.2}\text{O}_{2-\delta}$, (e)
 $\text{Ce}_{0.9}\text{Gd}_{0.1}\text{O}_{2-\delta}$ and (f)



The TGA of the doped ceria precursor materials represented the curve of weight loss. From the figures, the total weight loss at the temperatures between of 25 °C to 700 °C was found to be in different percentages for different compositions. From the curves of all the samples, it was understood that the weight loss begins to appear from the initial stage i.e., from 25 °C. From curve the thermal decomposition of the molecule can be split into four separate sections [5].

The first one is related to the evaporation of the absorbed water between 50 and 100 °C, at which most of the water will be evaporated. In the second section, the residue water will be evaporated at temperature rate. In third section the structural water, which has band structure with metal and oxygen atoms inside the molecule, will be eliminated before 650 °C. During heat treatments, the compound may lead to form phase pure doped ceria nanocomposite materials. In the last section, the molecule will be stable and the weight of molecule is nearly constant without any change in weight loss. Thermo gravimetric analysis data obtained from the precursor materials is given in Table: 1

Table 1: Thermo gravimetric analysis data obtained from the electrolyte precursor materials

Sample	Initial weight	Final weight	Total	Total
	(mg) at 25 °C	(mg) at 700 °C	weight loss	weight loss
			(mg)	(%)
$\text{Ce}_{0.9}\text{Gd}_{0.1}\text{O}_{2-\delta}$	8.13	7.49	0.64	7%
$\text{Ce}_{0.9}\text{Y}_{0.1}\text{O}_{2-\delta}$				
$\text{Ce}_{0.8}\text{Gd}_{0.2}\text{O}_{2-\delta}$	12.02	10.26	1.76	18%
$\text{Ce}_{0.8}\text{Y}_{0.2}\text{O}_{2-\delta}$				
$\text{Ce}_{0.9}\text{Gd}_{0.1}\text{O}_{2-\delta}$	11.38	10.51	0.87	9%
$\text{Ce}_{0.9}\text{Sm}_{0.1}\text{O}_{2-\delta}$				
$\text{Ce}_{0.8}\text{Gd}_{0.2}\text{O}_{2-\delta}$	13.09	11.23	1.86	19%
$\text{Ce}_{0.8}\text{Sm}_{0.2}\text{O}_{2-\delta}$				
$\text{Ce}_{0.9}\text{Gd}_{0.1}\text{O}_{2-\delta}$	12.05	10.79	1.26	13%
$\text{Ce}_{0.8}\text{Gd}_{0.2}\text{O}_{2-\delta}$	9.05	8.19	0.86	9%

From the Table: 1, it was understood that the weight loss of about 2-3% is found at around 100 °C, which may be due to the removal of water molecule from the samples. Then, the total weight loss is found to be different (1-2 %) for all samples at around 250 °C and which may be attributed to the removal of organics present in the samples. The further weight loss observed in the sample until 700 °C may be due to the decomposition of remaining carbon/nitrogen-based compounds from the sample. At around 700 °C, the weight loss is stable, which indicates the formation of phase-pure doped nanocomposite ceramic materials.

Conclusion

In this work, a set of nanocrystalline electrolyte materials ($\text{Ce}_{0.9}\text{Gd}_{0.1}\text{O}_{2-\delta}$, $\text{Ce}_{0.9}\text{Y}_{0.1}\text{O}_{2-\delta}$, $\text{Ce}_{0.8}\text{Gd}_{0.2}\text{O}_{2-\delta}$, $\text{Ce}_{0.8}\text{Y}_{0.2}\text{O}_{2-\delta}$, $\text{Ce}_{0.9}\text{Gd}_{0.1}\text{O}_{2-\delta}$, $\text{Ce}_{0.9}\text{Sm}_{0.1}\text{O}_{2-\delta}$, $\text{Ce}_{0.8}\text{Gd}_{0.2}\text{O}_{2-\delta}$, $\text{Ce}_{0.8}\text{Sm}_{0.2}\text{O}_{2-\delta}$, $\text{Ce}_{0.9}\text{Gd}_{0.1}\text{O}_{2-\delta}$ and $\text{Ce}_{0.8}\text{Gd}_{0.2}\text{O}_{2-\delta}$) for SOFC

was developed. The following conclusions are drawn from the results achieved from this research activity. Doped ceria nanocomposite ceramic oxide materials have been successfully developed using economically cheap method, i.e., chemical precipitation method. TGA patterns obtained on the precursor samples revealed the formation of product and the removal of molecules.

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Conflict of Interest: Author shows no conflict of interest

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