Synthesis and characterization of NiO-Al_2O_3 nano-composite as anode for IT-SOFCs

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Abstract The solution combustion synthesis technique was used to prepare nano-particles, nano composites and nano ceramic oxides. In the present paper, we proved the usefulness of the technique in producing NiO-Al_2O_3 nano-composite materials. In solution combustion, the stoichiometric ratio, according to propellant chemistry, needs oxidizer to fuel ratio unity. The combustion of nickel nitrate, aluminium nitrate and glycine at stoichiometric ratio results in NiO-Al_2O_3 nano-composites. The NiO-Al_2O_3 nano-composite powder is further applicable in anode material for Intermediate temperature solid oxide fuel cell (IT-SOFCs). This powder were characterized by different techniques such as XRD, TG/DTA.

Keywords: Combusion method, Fuel Cell, XRD, composite material, nanoparticals.

INTRODUCTION

In the recent time, the world's total energy consumption is increasing as the life style of human being is changing with new modern technology, which has generated the need of more electricity. Solid oxide fuel cells give many advantages such as high efficiency, modularity and low emissions of NO_x and SO_x. Particularly, at intermediate temperatures SOFC (500 ~ 700 °C), function is more attractive inexpensive metals can be used for external materials of the cells. So *solid oxide fuel cell is an electrochemical device that continuously converts chemical energy into electric energy*.

Ni-Al_2O_3 as an active and highly coking resistant catalyst layer for solid-oxide fuel cells operation [1], NiO/Al_2O_3 is suitable for preparation of Oxygen Carrier [2], Ni-Al_2O_3 is also used as functional material [3], Nickel aluminate spinal is also used as anode for aluminium electrolysis and for internal reforming solid oxide fuel cell anodes [4].

The NiO-Al_2O_3 can be prepared by many methods such as ceramic method [4], sol gel method [3], chemical looping combustion method [5]. The solution combustion method has emerged as a potential technique of preparing nano materials. It is low cost, quite simple and fast, fast heating rates and short reaction times compairing to other techniques. It is used in verity of applications such as catalyst, fuel cells and biotechnology. In this paper, we report synthesis of NiO-Al_2O_3 nano-composites by solution combustion technique. The phase purity of nano-composite was ascertained with XRD and the nano-composite were thoroughly studied for TG/DTA analysis. subsequently, the as prepared powder were heat treated at 600 ^{o}C and were characterized by XRD, TG/DTA.

EXPERIMENTAL

The pure chemicals, nickel nitrate, aluminium nitrate and glycine, obtained from Alfa Aesar with analytical grade were used for the synthesis. Ni nitrate and Al nitrate was used as an oxidizer and glycine as a fuel and their required amounts were dissolved in double distilled water. The mixture was then kept on hot plate to evaporate excess water till gel is formed. It was then allowed to auto-ignite with the rapid evolution of large volume of gases to produce fine powder. As prepared powder was heat treated at 600 ^{o}C for 2hrs to remove any carboneous impurity and hence to obtain pure and well crystalline powder.

Stoichiometric composition of the redox mixture was calculated based on the principles of propellant chemistry, keeping the oxidizer to fuel ratio unity [6]. The oxidizing valencies of Ni nitrate is -10 and Al nitrate is -15 and total reducing valencies of fuel is +9, so the oxidant to fuel ratio becomes 1.1 and 1.6. Structural studies of these samples were carried out by XRD (Phillips-3710 powder X-ray diffractometer) in the 2θ range 10 - 100° using $CuK_{\alpha 1}$ radiation (λ= 1.54056Å). The XRD patterns were compared with standard JCPDS files of NiO (JCPDS No. 73-1519) and Al_2O_3 (JCPDS No. 75-1865). The thermogravimetric and differential thermal analysis (TG-DTA) study has been carried out using a Perkin-Elmer TGA-DTA-DSC instrument with a heating rate of 10 °C/min in air to find the post heat treatment of material phase formation.

Results and Discussion

The nature of combustion depends upon the precursor material and fuel. Many times, with glycine, volume combustion is explosive type with no flame but results into high surface area dry powder with soft agglomerates but in our case it is not explosive. We have used glycine in all our work. Parameter that affects the properties of powder and phase purity is the oxidant to fuel ratio. The combustion at stoichiometric ratio gives NiO-Al_2O_3 nano-compposites.

X-ray diffraction (XRD)

The X-ray diffraction pattern of the as prepared and calcined powder are shown in Fig. 1a and 1b. There are different phases like NiO, Al_2O_3 and $Ni_2Al_{18}O_{29}$ are shown in Fig. 1a. The diffraction peaks are low and broad due to the small size effect and incomplete inner structure of the particle. The X-ray reflection peaks at 2θ =37.40 º and 65.15º can be readily indexed as (012) and (006) crystal planes of the Al_2O_3, respectively. This shows the rhombohedral crystallinity of Al_2O_3. The average crystallite size was calculated by X-ray diffraction line broadening using the Scherrer formula,

$$d = K\lambda / B \cos\theta \qquad (1)$$

where λ is the wavelength of X-ray in nanometer, B is full width half maxima (line broadening), K = 0.9 is Scherrer constant and θ is the Bragg's angle.

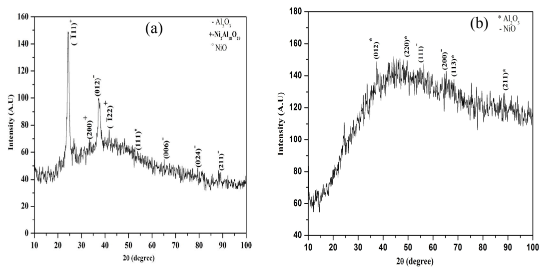

FIGURE 1. XRD patterns of (a) as prepared and (b) heat treated at 600 °C NiO-Al_2O_3 nano-composite.

Thermal Analysis of NiO-Al_2O_3 Nano-composite

TG/DTA of the as synthesized NiO-Al_2O_3 nano composite is shown in Fig. 2a.

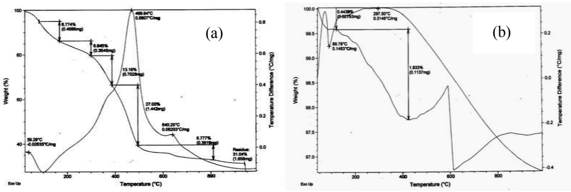

FIGURE 2. TG/DTA of (a) as prepared and (b) heat treated at 600 °C NiO-Al_2O_3 nano-composite.

A weight loss observed in the range between 50 °C to 100 °C with endothermic peak at 100 °C is attributed to dehydration of surface moisture. In range between 200 °C to 600 °C, oxidation of Ni to NiO begins slowly at 400 °C. This change is accompanied with a broad exothermic peak at 469.94 °C.

In Fig. 2b the peak attribute at 89.79 °C to the decomposition of water. The peak attributed at 297.50 °C which shows the formation of NiO.And further small increase atribute because of Al_2O_3 at 800 °C.

Conclusions

The NiO-Al2O3 nano-composite was successfully synthesized by solution combuston method and it gives the pure cubic and rhombohydral phases.

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