International Journal of Engineering Science Invention
ISSN (Online): 2319 – 6734, ISSN (Print): 2319 – 6726
www.ijesi.org || PP. 59-63

Synthesis and Characterization of $Nd_{1-x}Ba_xAlO_3$ Cathode Material for Intermediate Temperature Solid Oxide Fuel Cell

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ABSTRACT: *$Nd_{1-x}Ba_XAlO_3$ ($0 \leq x \leq 0.5$) nano crystalline powders were synthesized by assisted combustion method using Neodymium (III)nitrate, Barium (II)nitrate, Aluminum nitrate and Glycine (fuel). This process remarkably reduced the synthesis time to obtain$Nd_{1-x}Ba_XAlO_3$ and energy save. The crystalline structure of $Nd_{1-x}Ba_XAlO_3$ confirmed by X-ray diffraction. The average crystallite size is conformed from X-ray line broadening analysis by using the Scherer equation. The particlesize is determined by TEM analysis. The surface morphology of the synthesized product was observed by SEM studies. The electrical conductivity studies revealed that $Nd_{1-x}Ba_XAlO_3$ possessed the maximum electrical conductivity which is determined from dc four probe method.*

Keywords: *Electrical conductivity, SEM, Scherer equation, TGA & DTA, XRD and FTIR.*

I. INTRODUCTION

Recent studies in Solid Oxide Fuel Cell Technology as new power generation devices due to their high conversion efficiency, low emissions and excellent fuel flexibility. The intermediate operating temperature range from 500^0 -700^0 C it leads to a significant decrease in electrode activity. The considerable reduction in reaction temperature and time involved with this recent synthesis. It has the interesting effect that the particle growth during reaction and the resulting particle size of the products are usually reduced efficientl

High performance of Cathode require in addition to a large amount of reaction sites, the efficient transport of gases, electrons and ions. A potential cathodic material must exhibit electronic as well asoxygen-ion conductivity to increase the triple phase boundary to enhance the efficiency of SOFCs. In such materials, the gas-electrode-electrolyte triple-phase boundaries converted to a simple gas lead to a significant reduction in cathodic polarization losses.A Combustion process of oxide material since the homogeneous aqueous solution is the combustion product. A Potential Cathode material must exhibit electronic as well as oxygen ion Conductivity to increase the triple phase boundary to enhance the efficiency of SOFCs. In addition, the material should exhibit catalytic activity and compatible mechanical properties. In this method, reduction-diffusion was used to synthesize exchange coupled Nd_{1-x} Ba_xAlO_3 nano composites in <100 nm size range.

$Nd_{1-x}Ba_xAlO_3$ shows better properties as cathode material. The change in the oxidation state of Ba^{2+} due to the partial substitution of Ba for Al in $Nd_{1-x}Ba_xAlO_3$ has induced significant magnetic properties. It has partial replacement of Al by Ba has led to an improvement in electrical and electrochemical properties. That is improved by the sub electrochemical performance due to demonstrate in partial substitution of Ba in $Nd_{1-x}Ba_x$ AlO_3.Aluminium based perovskite compounds have attracted a lot of attention as IT-SOFCs electrode due to their mixed conducting characteristics and ion conductivity in the intermediate temperature range the ability to present in the mixed oxidation states and the important in covalent character of Al-O bonds are that these essential factors.

In Scope of present work Ba-doped $NdAlO_3$ was aimed to improve the electrochemical properties for ITSOFCs cathode applications-combustion technique, due to its high-energy efficiency, short reaction time, and intermediate temperature. The prepared samples were characterized by X-Ray Diffraction, Scanning electron microscope, Electrical Conductivity, Thermogravimetry analysis, and Differential thermal analysis, Infrared Spectroscopy. We report on the results of a complete characterization study including the electrical performance of the single cells, conductivity and the structural characterization from data, which is powerful to that the electrode materials under the usual conditions of a SOFC. In complement, thermogravimetry analysis (TGA) was used to demonstrate the reversibility of the $Nd_{1-x}Ba_xAlO_3$ oxidized and reduced phases.

II. EXPERIMENTAL STUDIES

Assisted combustion synthesis of Nanocrystalline $Nd_{1-x}Ba_xAlO_3$ (x= 0.1, 0.3, 0.5) powder

2.1 Materials Used

We used Neodymium nitrate $Nd(NO_3)_2$ (99.9%), Barium nitrate $Ba(NO_3)_2$ (99.99%) and Aluminium nitrate $Al(NO_3)_2$ (99.9%) were purchased from Sigma-Aldrich. All of the Chemicals were used without further purification.

2.2 Preparation

The $Nd_{1-x}Ba_xAlO_3$ was synthesized by assisted combustion method. Which using $Nd(No_3)_2$, $Ba(No_3)_2$, $Al(NO_3)_2$ and aspartic acid were (purity>99.9%, Aldrich) used. All the substances, required stoichiometric ratio, were dissolved in double distilled de ionised water. The aspartic acid was used as fuel. All of the solutions were mixed together to form homogeneous solution. This solution becomes pink colour and it is kept at Constant heating at 80^{o} C. To obtain foamy –like powder it was continuously heated and then crushed. The crushed powder was taken in a crucible, and heated on muffle furnace at 550˚C for 6 hrs. The same procedure was followed by other ratio such as 0.3, 0.5.

III. CHARACTERIZATION

3.1.X-ray diffraction analysis is made to determine the crystallite size and structure of the synthesized samples. A curved graphite crystal was used as a monochromator. The X-ray diffraction measurements were carried out in a 2Θ range from 10-80^{o} respectively.

Thermal analysis was carried out in a matter TA3000 system equipped with a TC10 processor unit. Thermo gravimetric curves were obtained in a TG50 unit, which working at a heating rate of 10^{o} C min^{-1}. The experimental measurements were heating from 25 to 500^{o} C with a rate of 10^{o} C min^{-1} for powder samples.

The morphology and microstructure of the powders were observed with a scanning electron microscope. FTIR Spectra were recorded on FTIR Spectrometer (Agilent Cary 630 FTIR Spectrometer) using. The electrical Conductivity of the sample was measured by a standard four-probe technique in the temperature range of 300^{o} C-850^{o} C in air.

IV. RESULTS AND DISCUSSION

4.1. Analysis of Crystalline Structure

The oxidized $Nd_{1-x}Ba_x AlO_3$Perovskite is obtained as well- Crystallized powder. Orthorhombic Structures were entified from laboratory X-ray diffraction for the oxidized materials. No impurity phases were detected. The peak broadening of an individual reflection decreases and becomes sharper with an increase in the reaction temperature. The average crystallite sizes of the particle calculated from X-ray broadening of the diffractions using Scherer's equation.

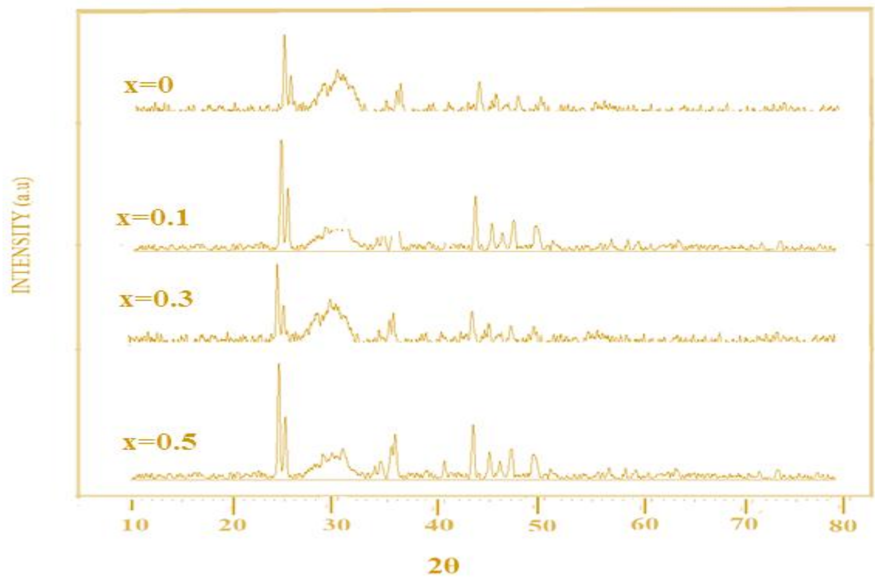

Fig.4.1. XRD Analysis of $Nd_{1-x}Ba_x AlO_3$

4.2. SEM analysis:

The electrode microstructure is related to the characteristics of the surface area, electrochemically active area, volume fraction of chemical phases present and electron transport. This sample shows porous morphology properties in the cathode as well as connectivity between the cathode. The microstructure of $Nd_{1-x}Ba_x AlO_3$ sample is similar to that the microstructure of the sample is insensitive to this substitution.

The electrical conductivity of the $Nd_{1-x}Ba_xAlO3$ Cathodes is presented in the temperature range is 100-750^{0} C dc four probe method. All the samples shown a decrease in electrical conductivity with increasing temperature for metallic behavior over the entire temperature range. Their conductivities start to decrease significantly at about 300^{0} C due to such lattice detects breaking Al-O bond, resulting in loss of oxygen atoms from the lattice and reduction of Al^{3+} to Al^{2+}.

Thethermal evolution of the samples was studied under both reducing and oxidizing atmosphere. The TGA and DTA Spectrum Shows, Obtained on $Nd_{1-x}Ba_x AlO_3$ Cathode Powder. The major weight loss occur in between 200-300^{0} C. In the TGA pattern the $Nd_{1-x}Ba_x AlO_3$ Sample showed a weight loss of about 0.037mg/min

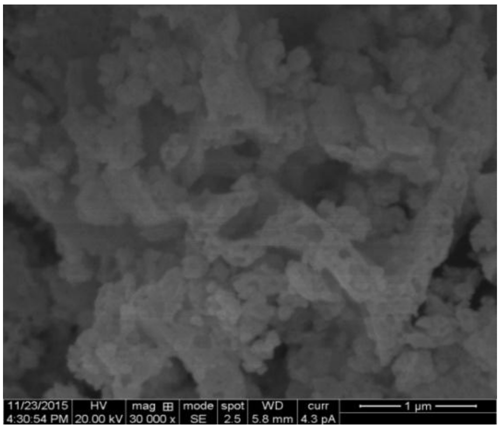

Fig: 4.2 SEM image of $Nd_{0.7}Ba_{0.3}AlO_3$

4.3. FTIR analysis:

FTIR Spectra showed strong bond at 682.3cm^{-1}which due to stretching vibration mode of the Nd-O bond in the structure. The peak appeared at 1481.6cm^{-1}corresponds to the H-OH bond mode confirming the presence of moisture in the sample. But in the region $Nd_{1-x}Ba_x AlO_3$exhibited in only two peaks. The bond appeared at 1406.0cm^{-1} is due to the presence of AlO_2 in the sample. The Sample $Nd_{1-x}Ba_x AlO_3$exhibited peak at 682.3cm^{-1}

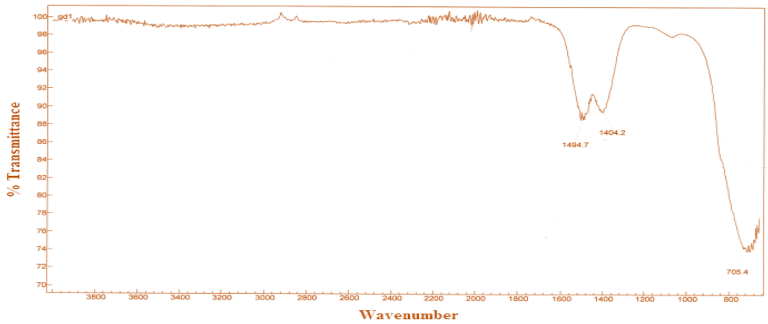

Fig 4.3 FTIR Spectrum obtained on $GdAlO_3$

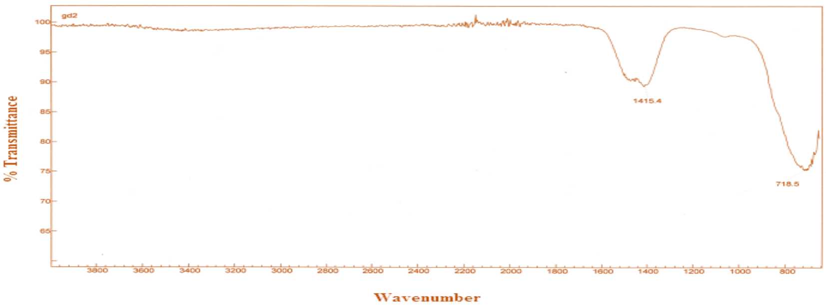

Fig 4.4 FTIR Spectrum obtained on $Gd_{0.9}Sr_{0.1}AlO_3$

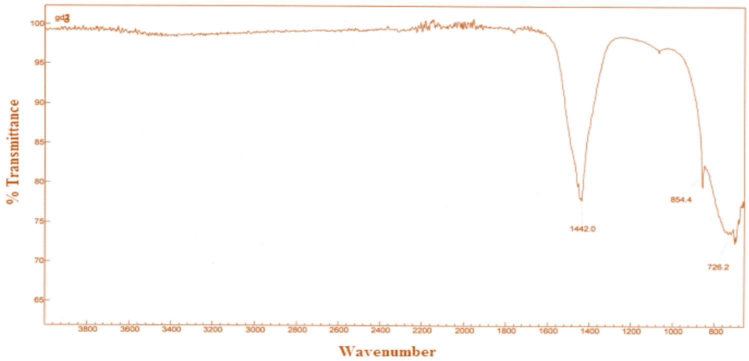

Fig 4.5 FTIR Spectrum obtained on $Gd_{0.7}Sr_{0.3}AlO_3$

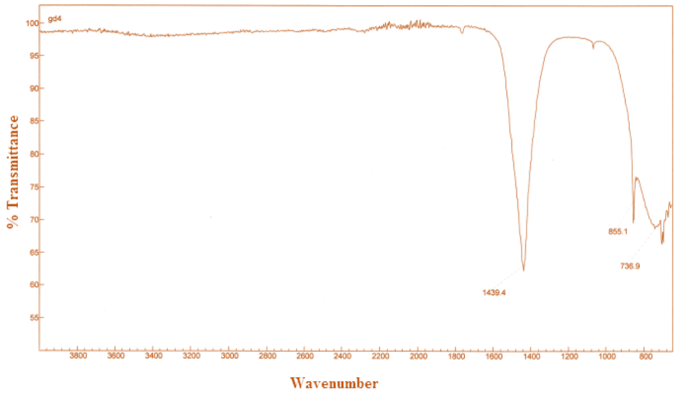

Fig 4.6 FTIR Spectrum obtained on $Gd_{0.5}Sr_{0.5}AlO_3$

4.4. Thermal Analysis TGA/DTA:

Thethermal evolution of the samples was studied under both reducing and oxidizing atmosphere. The TGA and DTA Spectrum Shows, Obtained on $Nd_{1-x}Ba_x\,AlO_3$ Cathode Powder. The major weight loss occur in between 200-300^0 C. In the TGA pattern the $Nd_{1-x}Ba_x\,AlO_3$ Sample showed a weight loss of about 0.037mg/min

In the DTA curve, broad exothermic peak 400^0 C occurred due to the weight losses between 40^0 C.As the TGA curve shows, the sample showed a weight loss of about 0.026mg/min. The sample continuously loss in weight at 4.0. The sample on further heating from 100-800^0 showed slight weight gain and weight loss of about 0.026mg/min in 347.33.

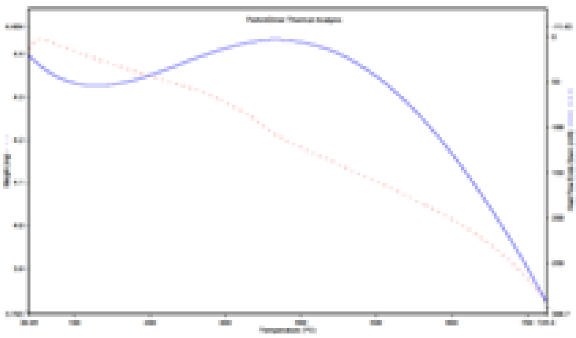

Fig: 4.7 TGA&DTA analysis of $Nd_{1-x}Ba_x\,AlO_3$

4.5. Conductivity Studies

The temperature dependence of electrical conductivity of Nd1-xBaxCoO3-□ □is shown in Fig.4.8. It shows the variation of electrical conductivity with Ba content (x) at various temperatures. At a given temperature, the conductivity value increased with x due to increasing Al+4 content and the charge carrier concentration. The conductivity increased with temperature for x□ 0.3, implying small polaron semiconductor behaviour, but decreased with increasing temperature for x □ □ 0.3, implying the metallic behaviour. Thus Nd1-xBaxCoO3-δexhibits, a semiconductor- to- metal transition around x =0.3. The faster decrease in conductivity at higher temperatures for samples with x □ □ 0.3 is due to the loss of oxygen from the lattice is shown in Fig4.8, and the cell volume increased with Ba content (x),Nd0.7Ba0.3CoO3 possessed the maximum electrical conductivity of around 445 Scm-1 at 500ºC.

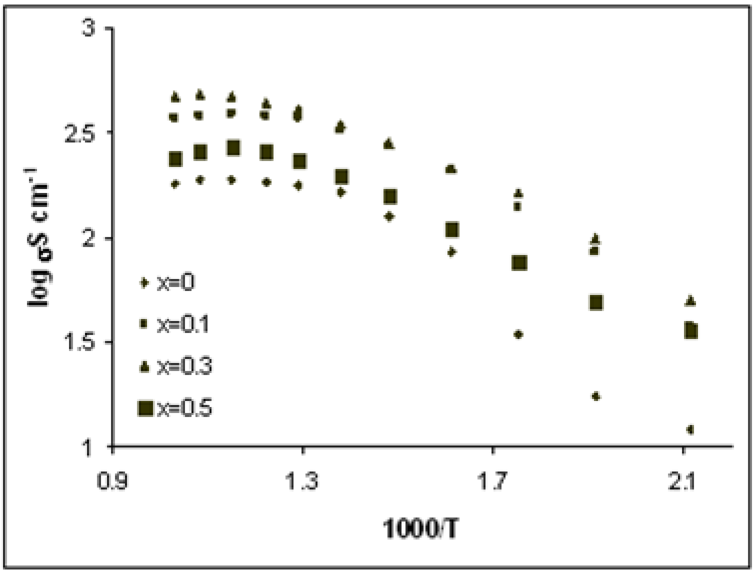

Fig.4.8 Arrhenius plots for the $NdGa_{1-x}Sr_xO_3$ samples sintered at different ratios

V. CONCLUSION

The nano-sized $Nd_{1-x}Ba_xAlO_3$ powder, synthesized by the combustion method, has been investigated. The Rietveld refinement of the X-ray diffraction profile of the sample at room temperature shows the orthorhombic phase symmetry. The increased concentration of $Nd_{1-x}Ba_xAlO_3$in composite reduces the crystallite size of host $Nd_{1-x}Ba_x$ AlO_3. This crystalline powder maybe potential cathode material intermediate temperature solid Oxide fuel cell.

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