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Title of the Project

Dense Membranes for Anode Supported all Perovskite IT-SOFCs

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ABSTRACT

During this first year of the project, a post doctoral fellow (Dr. Hrudananda Jena), and two graduate students (Mr. Vinay B. V. Sivareddy, Aswin Somuru), were supported through this project funds. Also, partial support was provided to three undergraduate students (Jonathan Dooley, India Snowden, Jeremy Gilmore) majoring in Chemistry, Physics, and Engineering disciplines.

Various wet chemical methods of synthesis have been attempted to prepare perovskite oxide powders with a hope to improve and engineer its properties to meet the requirements of Intermediate Temperature Solid Oxide Fuel Cell (IT-SOFCs) components. Various compounds were synthesized, characterized by XRD, TEM, SEM, XPS, electron microprobe and their electrical transport properties were measured by EIS at elevated temperatures and compared.

Sonochemical technique (power of ultra sonic probe 750 watt) combined with hydrothermal treatment of precursors for the preparation of calcium hydroxy apatites (Ca-HAp) was used for the first time. Ca-HAp was substituted with Sr and Mg (50 % replacement of Ca in Ca-HAp) to study the effect of substitution on Ca-HAp. Calcium hydroxy apatite is a bioceramic and has potential applications as artificial bone, enamel materials. In this study we tried to investigate its use as proton conductors in PC-SOFC. The properties like electrical conductivity, crystal structure, compositions of CaHAp were studied and compared with the natural bone material. The comparison found to be excellent indicating the efficiency of the preparation techniques. The typical value of conductivity measured is $0.091 \times 10^{-6} \text{ Scm}^{-1}$ at 25°C and $19.26 \times 10^{-6} \text{ Scm}^{-1}$ at 850°C with an applied frequency of 100 kHz. The conductivity increases on increasing frequency and temperature and reaches 0.05mS/cm at 500°C . The crystal structure and phase stability of perovskites as well as apatites were investigated with respect to substitution of various iso-valent and alivalent ions to determine the % of solubility in the crystal lattice of perovskite, apatites

Various electrode and electrolyte material compositions were prepared and characterized by XRD, SEM, XPS and electron microprobe. The material compositions were selected based on their thermo-physical properties to achieve compatibility with each other in ideal fuel cell operating conditions. The series of electrode materials investigated are $\text{LaGa}_{1-x}\text{M}_x\text{O}_3$ (M = Mn, Mg, x = 0.1), $\text{LaCr}_{1-x}\text{M}_x\text{O}_3$ (M = Mn, Mg, Co, x=0.1), $\text{LaNi}_{1-x}\text{Fe}_x\text{O}_3$ (0<x<0.6) and $\text{Gd}_{1-x}\text{M}_x\text{CoO}_3$ (M=Ca, x=0.1).

Attempts were made to prepare proton-conducting perovskites of $\text{SrCe}_{1-x}\text{M}_x\text{O}_3$ (M= Dy, Eu, Er, Tb, x=0.1) by using sonochemical and hydrothermal technique followed by microwave sintering processes. These compositions were prepared characterized by XRD, TEM, SEM and electrical conductivity of the pellets was measured. The interest of low temperature proton conducting electrolyte is to replace the well known oxide ion conducting solid electrolyte in SOFCs, thereby reducing the operating temperature of SOFC to lower temperature (i.e $400\text{-}600^{\circ}\text{C}$) and named it as PC-SOFC (proton conducting-solid oxide fuel cell).

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4. Introduction

The influence of preparation techniques on the microstructure, grain-size and consequently on the electrical transport properties of the ABO_3 structured materials used as electrode and electrolytes in all perovskite IT-SOFC were investigated. Nano-crystalline dense electrolyte, porous LSCF and $LaNi_{1-x}Fe_xO_{3\pm\delta}$ ($x=0-0.5$) as cathode and anode, and $LaCrO_3$ as interconnect were synthesized by various wet chemical methods. The wet chemical methods like metal-carboxylate gel decomposition, hydroxide co-precipitation, sonochemical and regenerative sol-gel process followed by microwave sintering of the powders. Microwave sintering parameters were optimized by varying sintering time, and temperature. The phase pure systems were obtained at sintering duration as low as 30 min and 1200 °C. The average grain size of these perovskites was ~ 22 nm range. The electrical conductivities of the compositions were measured by ac (5Hz-13MHz) and dc techniques. The conductivity of the sintered pellets was found to be ~0.01- 0.21 S/cm at 550-1000 °C range respectively. The effect of wet chemical methods especially sonochemical, and regenerative sol-gel in processing large quantities of nano-crystalline perovskites with multi-element substitutions at A- and B-sites to achieve physico-chemical compatibility for fabricating zero emission all perovskite IT-SOFCs are reported in this report.

Proton conducting perovskite solids have been investigated by many research groups across the globe to arrive at optimized compositions to achieve enhanced proton conductivity at relatively lower temperatures. The property of selective hydrogen permeation through the solid in a wide a range of temperatures makes it attractive to be considered as hydrogen separation membrane. Proton conducting solids can be used as electrolyte in solid oxide fuel cells in place of traditionally known oxide ion conducting yttria stabilized zirconia (YSZ) electrolyte (Fig.1). YSZ or Sr- and Mg-doped $LaGaO_3$ (LSGM) are good oxide ion conducting solid electrolytes, however, these materials need to be operated at 700-1000°C temperatures to work as an effective electrolyte. The higher operating temperature of the electrolyte affects the material life, problems in operating the cell at higher temperature, selecting the compatible electrode and interconnects components. The higher operating temperature of solid oxide fuel cell can be avoided by replacing intermediate temperature proton conducting perovskite solid electrolytes in place of oxide ion conducting electrolytes. Perovskites can be suitably tailored to exhibit proton

conduction at relatively lower temperatures which will enable fabrication of low or intermediate temperature fuel cells and hydrogen separation membranes. This could be done by doping a single phase oxide or by forming a two-phase composite material. We are investigating the structure and transport properties of a number of doped and undoped perovskites.

High proton conductivity has been reported for various perovskite-type oxides such as cerates and zirconates in hydrogen/humid atmosphere. However, only a few, if any, satisfy both high protonic conductivity and thermodynamic stability that are prerequisites for the application of such membranes as successful separators. BaCeO_3 , $\text{SrCe}_{1-x}\text{M}_x\text{O}_3$ compositions were synthesized by sonochemical treatment followed hydrothermal method and sintering is done by microwave heating. CeO_2 hydrated gel was obtained from Ce(IV) ammonium nitrate and mixed with $\text{Sr}(\text{OH})_2$ and sonicated for 30 min., then the reactants were subjected to hydrothermal treatment at 150°C for 4 h. SrCeO_3 was found to form orthorhombic perovskite above 1200°C of heat-treatment. The 10% substitution of dysprosium at Ce site does not form single phase. Instead Sr_2CeO_4 is formed along with SrCeO_3 . The electrochemical and surface chemistry, cell testing measurements were performed using advanced spectro-electrochemical characterization techniques such as XRD, TEM, and EIS techniques. The effect of ball milling was also studied on sintering of the pellets.

Hydroxy apatite, $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$ (Ca-HAp) is a bio-ceramic and is an alternative candidate for hard tissue replacements such as stainless steel and titanium based solids [1]. The existing materials such as stainless steel etc. are prone to as corrosion, wear, fibrous tissue encapsulation, inflammation, implant loosening due to poor adhesion, and stress shielding which leads to bone resorption. Ceramic based hydroxyapatite (HAp) are better suited as these are bioactive providing an interfacial bond between the implant and the surrounding tissue forms, leading to good fixation, and generally no fibrous tissue encapsulation. The major drawbacks however are their low strength, low toughness and brittleness, which limit their use for non-load applications, such as coatings and implants in non-stressed areas of the body. A possible solution is to obtain nanocomposites based on hydroxyapatites that show superior mechanical properties. A prerequisite for the formation of HAp based nanocomposites is to obtain uniform nano size particles of HAp through innovative chemical methods. A number of chemical routes have been reported in literature for the synthesis of submicron particles of HAp with narrow size distribution. In

our investigation we will emphasize on achieving nano-hydroxyapatite by hydrothermal or sonochemical reaction methods. These methods will be extended to synthesize other alkaline earth substitutions in the hydroxyapatite. The alkaline earth substitutions are strontium (Sr), barium (Ba) and magnesium (Mg). The alkaline earth analogues of Ca-HAp find use as bone replacement, bone fillings, bone adhesives and for the treatment of osteoporosis. Attempts will also be spared to substitute HAp with other elements (compatible to body tissues) to enhance mechanical strength so that these HAp can be used in load bearing areas of the body. Since these materials are going to be used in human body these needs to be thoroughly characterized by various sophisticated techniques and various tests should be conducted in vivo. In this study an attempt has been made to synthesize nano-composites of Ca-HAp with varying Ca^{2+} to $(\text{PO}_4)^{3-}$ ratio and alkaline earth (Ba, Sr and Mg) doping at Ca-site of the HAp by using sonochemical, hydrothermal followed by microwave sintering. The structural and transport properties will be correlated with the natural human bone powder measured under identical experimental conditions. The ordering mechanism of hydroxy groups in hydroxy-apatite $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$, which is the major constituent of mammalian bones and tooth enamel has been discussed. Apart from its use as biomaterials, Ca-HAp may be a candidate for future proton conductors for proton conducting ceramic fuel cell application as well as the composite electrolyte in the direct methanol fuel cells.

Experimental, Results, and Discussion

Novel wet chemical methods and advanced synthesis routes were used to prepare and optimize the particle size of the mixed conducting and proton conducting perovskites. Detailed procedures were reported earlier in our reports. On adding citric acid ($\text{COOH}-\text{CH}_2-\text{COH}(\text{COOH})-\text{CH}_2-\text{COOH}$) or tartaric ($(\text{CHOHCOOH})_2$) or glycine ($\text{H}_2\text{NCH}_2\text{CO}_2\text{H}$) to the metal nitrate solutions, metal nitrate tartarate or citrate or glycine complex is formed. These complexes on heat-treatments at 873–1073 K range of decomposed to yield nano-materials of LSGM and other perovskite compositions. In this method, the loss of constituents from the precursor was completely ruled out because of low temperature heat-treatment and losses due to co-precipitation, filtration etc. was avoided. This method has been used for preparation of rare-earth manganites, gallates Sr and Mg doped gallates. The formation of complex precursor is evident from the broad hump observed in XRD as shown in Fig. 1 and Fig.2. The complexation of the metal cations with tartaric

acid/citric acid helps in bringing all the reactants to a single or nearly unique boiling and melting point. Therefore they boil or melt at a particular temperature. On calcining the powders in furnaces at higher temperatures facilitates the growth of crystallites to bigger size. In contrast to solid state reactions where several grinding, mixing and palletizing is required to attend homogeneous mixing of the ions. In wet chemical methods, especially carboxylate gel decomposition methods, the insitu exothermic decomposition process provide enough heat for the crystallites to grow.

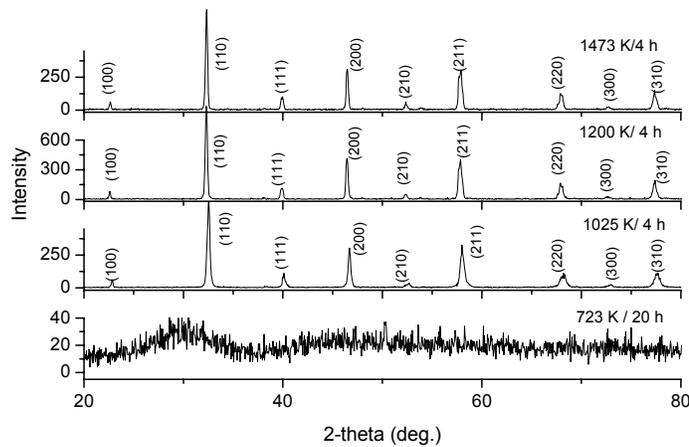


Figure 1. Room temperature XRD patterns of $\text{La}_{0.9}\text{Ga}_{0.9}\text{Mn}_{0.1}\text{O}_{3-\delta}$ prepared by tartarate route, heated at various temperatures in air and cooled to ambient temperature.

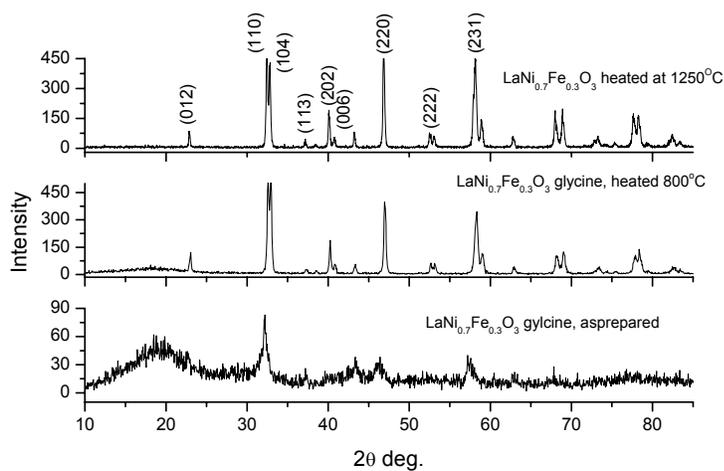
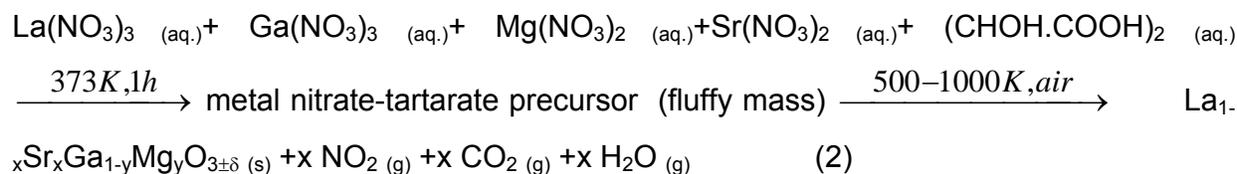


Fig.2 XRD pattern of $\text{LaNi}_{0.7}\text{Fe}_{0.3}\text{O}_3$ prepared by glycine nitrate decomposition process.

The nitrate groups provide oxygen for the effective burning of the organic matter. The chemical reaction taking place during the process is given below.



The gases evolved on calcinations of the precursor were identified by evolved gas analysis –mass spectrometry (EGA-MS) studies [7]. Based on the EGA-MS and TGA-DTA study of the typical rare-earth gallates the equation 2 has been formulated. The formation of citrate complexes can also be written like equation 2. In case of sol-gel route, the complexation of the metal nitrate with the carboxylate ions forms sols and adding glycerol (trihydric alcohol, HOCH₂-CHOH-CH₂OH form gel). The glycerol helps in esterifying the carboxylic acid and subsequently ends up in forming a resin like product. The resin like product does not stick to the wall of the glass (preparation vessel) beaker; hence it facilitates handling the precursors for further processing.

The XRD pattern of the product obtained on heat-treatment of the resins are given in Fig. 1. The formation of phase pure La_{0.9}Ga_{0.9}Mn_{0.1}O₃ and LaGaO₃ at relatively low temperatures (1273 K) are shown in Fig.1. The formation of phase pure LaNi_{0.7}Fe_{0.3}O₃ is shown in Fig.2. The formation of phase pure powders at low temperatures as compared to conventional methods of preparation is attributed to the provision of heat insitu by exothermic decomposition of the metal carboxylate complex. The formation of La-deficient compositions indicates accommodation of cation vacancy (10 % vacancy) in the lattice. This was confirmed from XRD analysis of the powders by showing absence of Ga as Ga₂O₃ phases in the XRD pattern. The TEM examination showed the phase pure powders of (La_{0.9}Ga_{0.9}Mn_{0.1}O₃) are nano-crystalline in nature and the particle size measured is found to be ~22 nm

Fig.4 shows the log (σT) vs. 1/T plots of several compositions prepared by wet chemical methods. The conductivities fall in the 0.01S/cm to 0.21 S/cm at 600 to 1000°C. The activation energy of electrical conduction estimated from the Arrhenius plot of these compositions were ~1.00 eV. The activation energy of conduction shows that these materials are oxide ion conductors. The electrical conductivity of the films prepared by microwave sintering and bulk pellets will be communicated in a separate manuscript.

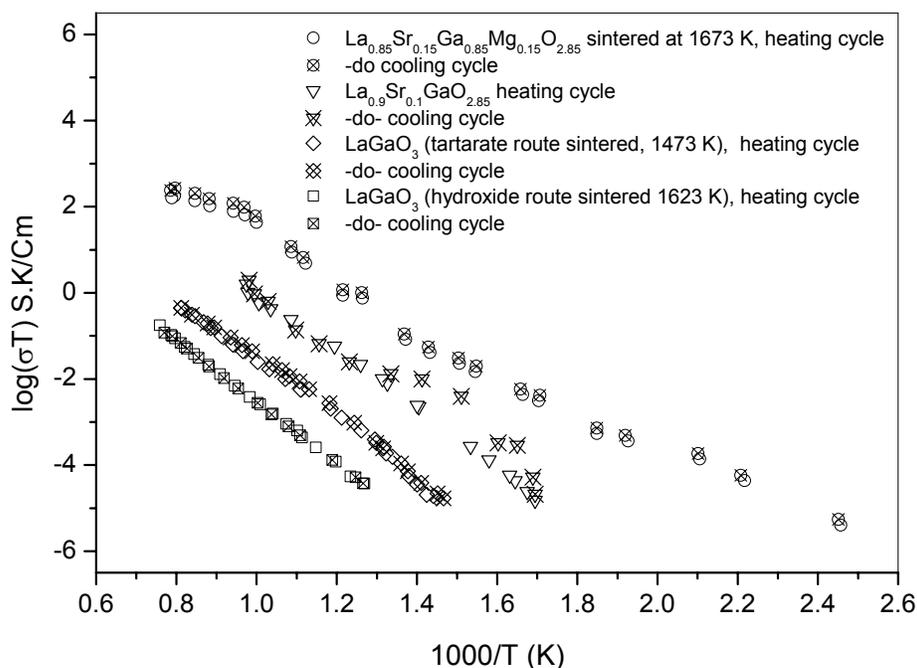
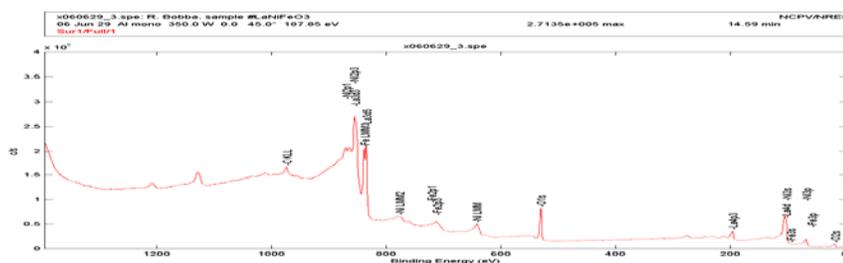


Figure 3. Log σT vs. $1/T$ of LaGaO_3 and $\text{La}_{0.85}\text{Sr}_{0.15}\text{Ga}_{0.85}\text{Mg}_{0.15}\text{O}_{3-\delta}$ in air

XPS studies of the samples show the oxidation state of the constituent ions in the perovskite lattice. The Fig. 5 shows XPS spectra of $\text{LaNi}_{0.7}\text{Fe}_{0.3}\text{O}_3$. The binding energy (B.E) of Ni and Fe are shown in the Fig. 5 and Fig.6. The peak at B.E 705 eV to 720 eV is a broad one indicating the presence of Fe in multiple oxidation states (majority Fe^{3+} along with small concentrations of Fe^{2+}). The broad peak is not well resolved; the B.E matches with B.E of Fe_3O_4 and Fe_2O_3 ($\text{Fe}2p_{3/2}$) given in the XPS data base. The B.E of Ni matches with $\text{Ni}2p_{3/2}$, $\text{Ni}2p_{1/2}$. B.E of $\text{Ni}2p_{3/2}$ ranges from 850 eV -857.5 eV with well resolved 3 peaks and a broad peak at 869.9 which corresponds to $\text{Ni}2p_{1/2}$. Ni is found to exist in Ni^{2+} and Ni^{3+} states.



XPS spectrum of $\text{LaNi}_{0.7}\text{Fe}_{0.3}\text{O}_3$

The interconnect materials like $\text{LaCr}_{0.9}\text{M}_{0.1}\text{O}_3$ ($\text{M}=\text{Co},\text{Mg},\text{Mn}$) series of compositions were synthesized and were characterized to find its suitability for all perovskite SOFC applications. The XRD pattern of $\text{LaCr}_{0.9}\text{Co}_{0.1}\text{O}_3$ is given in Fig.8 (a-b).

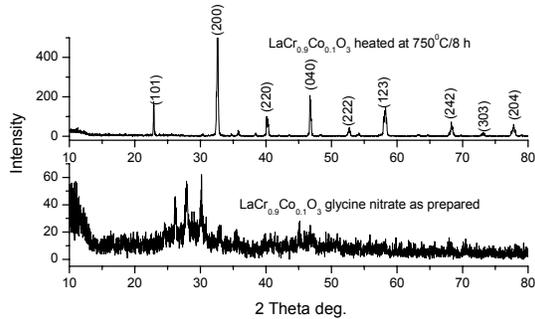


Figure 9 XRD of $\text{LaCr}_{0.9}\text{Co}_{0.1}\text{O}_3$ Fig.

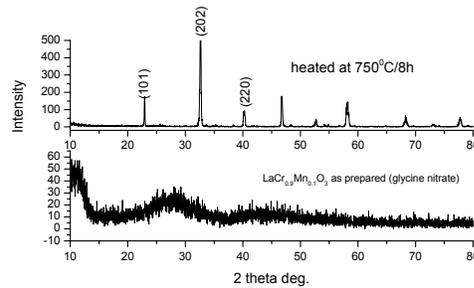
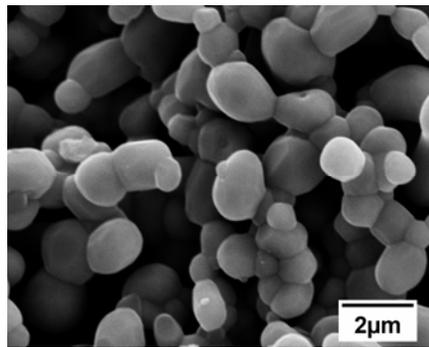
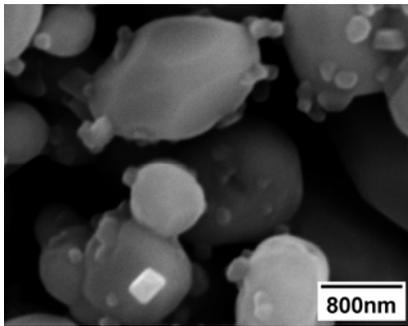


Figure 10 XRD of $\text{LaCr}_{0.9}\text{Mn}_{0.1}\text{O}_3$



$\text{LaCr}_{0.9}\text{Mn}_{0.1}\text{O}_3$ heated at $1050^\circ\text{C}/6\text{h}$ $\text{LaCr}_{0.9}\text{Co}_{0.1}\text{O}_3$ heated at $1200^\circ\text{C}/6\text{h}$

The XPS spectrum of $\text{LaCr}_{0.9}\text{Co}_{0.1}\text{O}_3$ show the prevalence of Cr in Cr^{3+} state ($\text{Cr}2\text{p}3/2$ at 576.5 eV. Fig. 14 shows the spectrum of $\text{Mn}2\text{p}3/2$ in $\text{LaCr}_{0.9}\text{Mn}_{0.1}\text{O}_3$. The broad spectrum (not resolved) is attributed to the prevalence of Mn in Mn^{3+} and Mn^{4+} oxidation state. The mixed conducting perovskite $\text{Gd}_{0.9}\text{Ca}_{0.1}\text{CoO}_3$ was synthesized by glycine nitrate method and characterized by various techniques. The electrical conductivity and phase stability studies were carried out.

The chemical composition analysis of the compounds was investigated by electron microprobe. The electron microprobe results indicate the compositions of the compounds are in well agreement as per the stoichiometry of constituent ions taken while preparing the material. This confirms the correctness of the preparation process and there is no loss of constituent elements during processing of the material.

PROTON CONDUCTING PEROVSKITES

The hydrothermal technique is a very suitable method of preparation for perovskites with higher concentration of hydroxyls. The hydrothermal method used here did not yield single phase perovskites of SrCeO_3 or its doped compositions after hydrothermal treatment at $150^\circ\text{C}/4\text{h}$. The precursor formed contains nano-crystalline CeO_2 hydrated gel along with other amorphous constituents. This was observed from the XRD peaks which correspond to CeO_2 . The nanocrystalline nature of CeO_2 was also confirmed from the selected area electron diffraction (SAED) pattern as shown in the TEM.

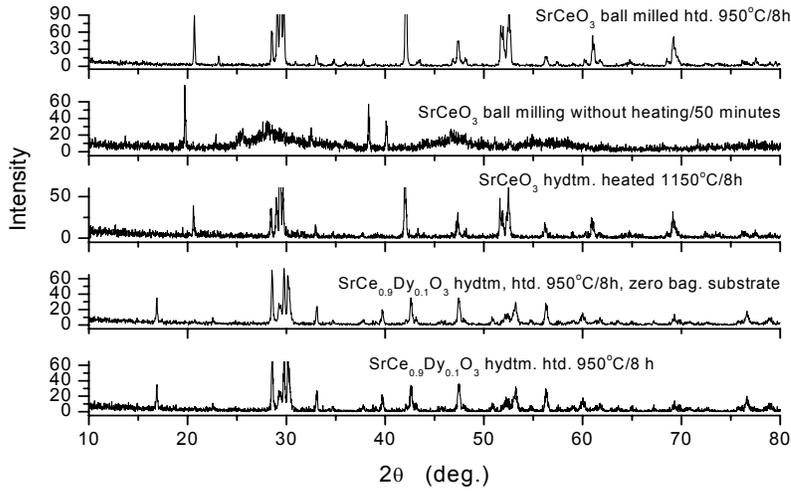
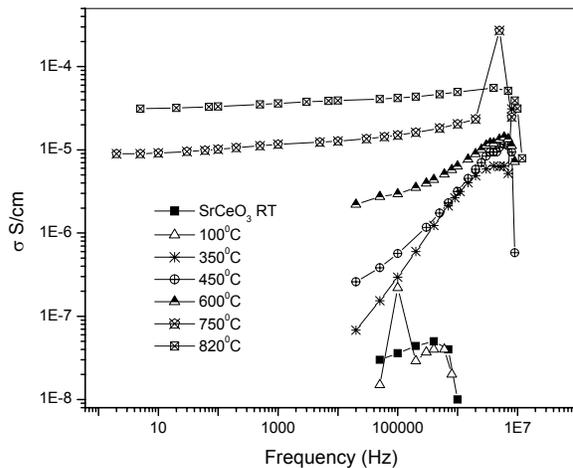


Figure 3 XRD patterns of $\text{SrCe}_{1-x}\text{M}_x\text{O}_3$ ($\text{M}=\text{Dy}$,) shows the evolution of phase on calcining at various temperatures



(a) SAED of SrCeO_3 hydrothermal precursor heated at 400°C , showing nano-crystalline nature of CeO_2 gel (b) TEM micrograph of the hydrothermal precursor gel.

The electrical conductivity of the compositions was measured by ac impedance method. The electrical conductivity plot on SrCeO₃ is shown in Fig. 5. The electrical conductivity of the sample increases with increasing applied frequency and temperature. The electrical conductivity observed at 25⁰C or room temperature is attributed to adsorbed water and the hydroxyls present in the lattice. On increasing the temperature above 100⁰C the electrical conduction seen is attributed to the prevalence of hydroxyl protons in the SrCeO₃. Similar experiments on alkaline zirconates and titanates [8] showed the presence of hydroxyl protons in the perovskite lattice. The electrical conduction in these perovskites is enhanced by oxygen vacancies at and above 500⁰C. The electrical conductivity observed to increase above 350⁰C as shown in Fig.5. The electrical conduction observed below 500⁰C is attributed to the migration of hydroxyl protons (Grotthuss mechanism) along the oxygen octahedra in the perovskite lattice. Above 500⁰C to 820⁰C it is observed to be higher and the charge carriers are observed to be mobile at low applied frequency of 20-50 Hz. The details on the mechanism of electrical conductivity, phase and structural stability of these compositions are beyond the scope of this paper.



Electrical conductivity measurements on SrCeO₃ prepared by ball-milling and sintered at 1250⁰C/6 h

The CeO₂ lattice constants calculated by indexing the corresponding XRD patterns are a= 5.411 Å, space group=Fm3m. The Sr₂CeO₄ lattice constants are a=6.119 Å, b= 10.349 Å, c=3.597 Å. On calcinations to higher temperature (1250⁰C/6h) single phase SrCeO₃ was formed. The crystal structure of SrCeO₃ on indexing the XRD pattern was found to be orthorhombic with a lattice constant of a=6.153Å, b=6.012 Å and c=8.589 Å, Space group=Pbnm.. I

8. Accomplishments/Presentations:

During this reporting period, the PI and his team have published the following papers in the refereed journals and in international conference proceeding volumes.

- 1) Innovative processing of dense LSGM electrolytes for IT-SOFC's, B. Rambabu, Samrat Ghosh, Weichang Zhao and Hrudananda Jena, Journal of Power Sources, 159 (2006) 21-28
- 2) Novel wet-chemical synthesis and characterization of nanocrystalline CeO_2 and $\text{Ce}_{0.8}\text{Gd}_{0.2}\text{O}_{1.9}$ as solid electrolyte for intermediate temperature solid oxide fuel cell (IT-SOFC) applications, B.Rambabu, Samrat Ghosh and Hrudananda Jena, J. Mater. Sci. (2006) 41:7530-7536
- 3) An exploratory study on solution assisted synthetic routes to prepare nano-crystalline $\text{La}_{1-x}\text{M}_x\text{Ga}_{1-y}\text{N}_y\text{O}_3$ (M=Sr, N=Mn,Mg) for IT-SOFC applications. Hrudananda Jena and B. Rambabu, Mater. Chem. Phys (2006), vol 101, 21-22.
- 4) Effect of sonochemical, regenerative sol gel, and microwave assisted synthesis techniques on the formation of dense electrolytes and porous electrodes for all perovskite IT-SOFCs. Hrudananda Jena and B.Rambabu, Accepted in Fourth International ASME (American Society for Mechanical Engineers) Conference on Fuel Cell Science, Engineering and Technology, Irving, California, USA, June19-21, 2006. (Accepted for publication ASME, Journal of Fuel Cell Science and Technology, paper # FUELCELL2006-97262).
- 5) Nanocrystalline TiO_2 (anatase) for Li-ion batteries,. V. Subramanain, A. Karki, K.I.Gnanasekar, Fanney E. Posey, and B. Rambabu, Journal of Power Sources, 159 (2006) 186-192
- 6) Proton conducting (pc) perovskite membranes for hydrogen separation and pc-sofc electrodes and electrolytes, Hrudananda Jena, B. Rambabu, F. P. Eddy, Proceedings of the 10th Asian Conference on Solid State Ionics, Advanced Materials for Emerging Technologies, edited by B.V.R. Chowdari, M.A. Careem, M.A.K.L. Dissanayake, R.M.G. Rajapakse,

V.A.Seneviratne, Kandy, Srilanka, World Scientific Publishing Co.Pte.Ltd., Singapore, 12-16 June 2006, Pp 789-796.

- 7) Proton transport in nano-crystalline bioceramic materials: an investigative study of synthetic bone with that of natural bone, Hrudananda Jena, B. Rambabu and Ramsey Saunders, Proceedings of the 10th Asian Conference on Solid State Ionics, Advanced Materials for Emerging Technologies, edited by B.V.R. Chowdari, M.A. Careem, M.A.K.L. Dissanayake, R.M.G. Rajapakse, V.A.Seneviratne, Kandy, Srilanka, World Scientific Publishing Co.Pte.Ltd., Singapore, 12-16 June 2006, Pp 69-76.
- 8) Electroceramic materials for the development of natural gas fuelled SOFC/GT plant in developing country (Trinidad & Tobago (T&T)), Rramsey Saunders, Hrudananda Jena and B. Rambabu, Proceedings of the 10th Asian Conference on Solid State Ionics, Advanced Materials for Emerging Technologies, edited by B.V.R. Chowdari, M.A. Careem, M.A.K.L. Dissanayake, R.M.G. Rajapakse, V.A.Seneviratne, Kandy, Srilanka, World Scientific Publishing Co.Pte.Ltd., Singapore, 12-16 June 2006, Pp 799-810.

In addition to the above, all undergraduate students presented their work (poster presentations) in the regional and national conferences in the U.S-DOE-REAP conference in Denver, CO.

9. Patents: None