6.1. Introduction

The generation of power in 19th and 20th centuries was mainly with the help of conventional sources of energy consisting of basically coal, oil and gas. Due to exhaustive use of these sources, they are depleting very fast. In addition, their use is causing severe damage to the environment. A change in environmental politics and the depletion of natural resources renewed the interest in fuel cell technologies. Because of the potential to reduce the environmental impact and geopolitical consequence of the use of fossil fuels, fuel cells have emerged as a great alternative to combustion engines. Due to their high efficiency and low emissions, fuel cells are attractive for their modular and distributed nature and zero-noise pollution. Fuel cells can offer clean and pollution-free technology to generate electricity at high efficiency by an electrochemical combination of fuel with an oxidant [1] they will also play an important role in any further hydrogen fuel economy. Among all the types of fuel cells, Solid Oxide Fuel Cell (SOFC) provides high energy conversion efficiency due to direct conversion of fuels into electric power at high temperature in a cogeneration system [2].

Solid oxide Fuel Cell (SOFC) has received great consideration as one of the promising systems of electric power generation, because of its high power conversion efficiency and pollution free operation [3]. The existing SOFC technology has demonstrated much higher chemical to electrical energy conversion efficiency (50%) with minimal pollutant emission over conventional energy conversion technologies (16-34%) [4]. Conventional SOFCs use YSZ as electrolyte which have to operate at high temperatures (~1000 °C) in order to get sufficient ionic conductivity. However, long-term degradation problems associated with the high –temperature operation, and relatively expensive manufacturing costs pose main challenges for the commercialization of this technology. Lowering the operating temperature to an intermediate temperature (IT) range (400–700 °C), not only improves
reliability and operational life, but also lowers the cost and time and energy it takes to heat up to the operating temperature. This will extend its application domain to residential power and portable devices [5].

Now days, an IT-SOFC which is operated at 600-800 °C and compatible with other forms of hydrocarbon fuels, has attracted the interest of many researchers [6-8]. However low operating temperatures leads to polarization loss of electrodes [9]. The electrolytes based on ceria are good candidates for IT-SOFCs, due to their inherent property of higher ionic conductivity at these temperatures compared to YSZ [10]. Ceria based electrolytes have relatively large unit cells compared to systems based on zirconia and, as a consequence, ceria-based systems have larger channels through which oxygen ions can pass during the conduction [11]. Hence Gadolinia- doped Ceria (GDC) is considered to be one of the most promising electrolytes used for SOFCs to be operated at 700 °C [12]. Partial internal shorting arising from the electronic conduction of GDC becomes insignificant at low temperatures [13]. In order to increase the ionic conductivity at intermediate temperature, reduction in the thickness of the GDC electrolyte (10-20 µm) will be another option [14-15]. To form the thin film electrolyte for SOFCs, it has to be supported by some kind of substrate and if the substrate itself acts as an electrode of SOFC, it would be an added advantage.

In this particular study, attempt has been made to synthesize an anode material having appropriate percentage of electronic as well as ionic phases. The series of such kind of anode materials have been synthesized as discussed in previous Chapter V. From these findings, NiO-GDC nano-composites with 30 and 40 mol % of NiO have came up as suitable anode. Secondly GDC has been coated on these anodes by using Spray Pyrolysis Technique (SPT). The SPT is a well known method to prepare tailored metal oxide thin films on either conducting or non conducting substrates. The versatility of SPT and requirement of fabrication of dense, thin solid electrolyte film on functional ceramic substrate makes SPT a suitable technique for development of electrode
Chapter VI: Formation and characterizations of NiO-GDC/GDC structure for IT-SOFC

electrolyte structure (half cell) [16]. This chapter reports reduction of NiO-GDC to Ni-GDC in Ar plus H₂ atmosphere. The Ni-GDC cermets were characterized by different method such as XRD, microstructural and electrical characterizations. Thin GDC electrolytes were deposited on NiO-GDC substrates containing 30 and 40 mol % of NiO. The microstructural and electrical characterizations of half cell were carried out.

6.2. Experimental

6.2.1. Synthesis of Ni-GDC

In the present study, NiO-GDC nano-composites with 30 and 40 mol % of NiO were prepared by physical mixing of combustion synthesized GDC and NiO nanopowders. The details are given in Chapter V. Since the optimized % of NiO was 30 and 40 mol %, NiO-GDC composites with these mol% were used for studying heir characteristics as anode for IT-SOFCs.

The prepared powders of NiO-GDC nano-composites containing 30 and 40 mol % of NiO were firstly reduced in argon plus hydrogen atmosphere at 500 °C with heating rate of 5 °C/min. In our experiment it was noticed that pellets did not get reduced completely. Only the surface but not the bulk reduction occurred. So it was planned to reduce powders first. After complete reduction of powders, these were characterized by XRD to confirm reduction of NiO-GDC into Ni-GDC. After studying XRD, it was confirmed that NiO-GDC has been converted into Ni-GDC. Further these Ni-GDC nano-composites powders were strengthened in the form of cylindrical pellets having a diameter of 13 mm and thickness of about 1 mm by applying a uniaxial force of 7 tons for three minutes using a hydraulic press. Extreme care was taken to see that pelletisation of all the samples were done under identical conditions and these pellets were sintered in argon plus hydrogen atmosphere at 1000 °C for 5hrs. Also some rectangular pellets were prepared for four probe resistivity
measurement. The dimensions of these rectangular pellets were 2.5 cm in length and 0.5 cm in breadth. After formation of the pellets, these were sintered at 1000 °C in Ar + 4% H2 with the flow rate 1 L/min for 4 hr. Care was taken such that Ni-GDC should not get oxidized during the sintering process. The reduction treatment leads to formation of porous Ni-GDC nano-composite anode. These samples were named as N3G1-r (30% NiO and 70% GDC10) and N4G1-r (N4: 40% NiO and 60% GDC10).

After sintering, the pellets were characterized by FE-SEM for studying microstructure. Also four probe measurements were carried out to study the d.c. conductivity of Ni-GDC anode of IT-SOFC.

6.2.2. Synthesis of NiO-GDC/GDC/ LSCF-GDC Single Cell

For the synthesis of GDC thin films, cerium nitrate (Ce (NO\textsubscript{3})\textsubscript{3}.6H\textsubscript{2}O, 99.9% pure; ALFA AESAR) and gadolinium nitrate (Gd (NO\textsubscript{3})\textsubscript{3}.6H\textsubscript{2}O, 99.9% pure; ALFA AESAR) were dissolved separately in double distilled water to form solution with same concentration. The equi-molar aqueous solution of cerium nitrate and gadolinium nitrate were mixed in desired proportion i.e. (9:1) to form the spray solution. This solution was then sprayed by air blast type of nozzle on glass substrate to optimize the process, which has been studied in detail by Chourashiya [17].The optimized preparative parameters for the deposition of GDC10 thin films on glass substrate are tabulated in Table 6.1.

The optimized parameters were applied to deposit GDC films on NiO-GDC nanocomposite rather than Ni-GDC. If Ni-GDC cermet was used as substrate for deposition of GDC, during deposition Ni-GDC would get oxidized due to air presence in the spray set up. On the other hand NiO in the composite could be reduced in situ during the cell operation.

The solution was sprayed on the NiO-GDC ceramic substrate, kept on hot plate with controlled preset temperature of 250 °C. The GDC thin films
deposited on NiO-GDC nanocomposite substrates were adherent and thick. The films were annealed at 450 °C for 2 hr. Further crystallization of GDC films and improvement in film/substrate interface were achieved by subsequent heat treatment the structure at 1000 °C for 8 hr. in air.

**Table 6.1:** Optimized and fixed preparative parameters of SPT for deposition of GDC 10 thin films on glass substrate

<table>
<thead>
<tr>
<th>Fixed Parameters</th>
</tr>
</thead>
<tbody>
<tr>
<td>Type of salt</td>
</tr>
<tr>
<td>Atomization type</td>
</tr>
<tr>
<td>Nozzle to substrate distance</td>
</tr>
<tr>
<td>Nozzle motion</td>
</tr>
<tr>
<td>Linear distance traversed by nozzle</td>
</tr>
<tr>
<td>Temperature controller</td>
</tr>
</tbody>
</table>

**Optimized parameters for GDC 10 on glass substrate**

<p>| | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Solution quantity</td>
<td>100ml</td>
</tr>
<tr>
<td>Substrate temperature</td>
<td>300 °C</td>
</tr>
<tr>
<td>Solution concentration</td>
<td>0.04 M</td>
</tr>
<tr>
<td>Annealing (post heat treatment)</td>
<td>450 °C for 2 hr.</td>
</tr>
</tbody>
</table>

**Table 6.2:** The optimized preparative parameters for the deposition of GDC thin films for both the ceramic as well as glass substrate

<table>
<thead>
<tr>
<th>Preparative Parameters</th>
<th>Substrate</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Glass</strong></td>
<td><strong>NiO-GDC</strong></td>
</tr>
<tr>
<td>Solution concentration (M)</td>
<td>0.04</td>
</tr>
<tr>
<td>Solution Flow Rate (ml/min.)</td>
<td>3±0.2</td>
</tr>
<tr>
<td>Deposition Time (min.)</td>
<td>30 min. 3 sec.</td>
</tr>
<tr>
<td>Substrate Temperature (°C)</td>
<td>300</td>
</tr>
<tr>
<td>Post annealing (°C)</td>
<td>450/2 hrs. + 1000 /8hrs.</td>
</tr>
</tbody>
</table>
After co-sintering of NiO-GDC/GDC half cell at 1000 °C, LSCF-GDC cathode was applied on GDC thin film by screen printing to form NiO-GDC / GDC / LSCF-GDC single cell.

For the single cell fabrication, LSCF-GDC powder was prepared by using separately synthesized LSCF and GDC powders by solution combustion synthesis. LSCF and GDC powders were taken in weight ratio of 70:30 and mechanically ground for 3 hr. to form LSCF - GDC nanocomposite. The preparation of slurry for screen printing was done by using ethyl cellulose as organic binder and terpenol oil. Whole assembly was sintered at 1000 °C for 4 hrs. The detailed of slurry preparation for screen printing has been describe in the flow chart.
6.3. Results and Discussion

6.3.1. Ni-GDC nanocomposite

Structural studies of Ni-GDC nanocomposite samples were carried out by XRD (Rigaku D/Max 2200 X-ray diffractometer) in the 2θ range 20–80° using CuKα1 radiation (λ = 1.54056 Å). The XRD patterns were compared with standard JCPDS files of NiO (04-0850; cubic Ni) and GDC10 (75-0161). Morphology of the composites was studied using HITACHI S-4700 cold field emission scanning electron microscope (FE-SEM). The dc electrical conductivity measurements were carried out by four-probe method within temperature range of 450 °C to 900 °C.

6.3.1.1. X-ray Diffraction (XRD)

The XRD patterns of Ni-GDC nano-composites reduced in Ar + 4% H2 atmosphere at 1000 °C are shown in Fig. 6.1a and Fig.6.1b. These XRD patterns were compared with JCPDS files of GDC and Ni. These were also compared with JCPDS file (78-0643; cubic NiO) of NiO to confirm the absence of peak of cubic NiO due to incomplete reduction of NiO. From Fig. 6.1 (a and b) it is observed that there is no any peak of NiO and hence NiO-GDC nanocomposite has been completely reduced to Ni-GDC. All the peaks were indexed and are shown in Fig. 6.1a and Fig.6.1b. The average lattice parameters calculated for GDC10 and Ni are 5.42 Å and 3.52 Å respectively. These values of lattice parameters well match with standard values (5.418 Å GDC and 3.523 Å Ni) showing cubic and fluorite type structure for Ni and GDC respectively. The absence of peak due to Gd2O3 and comparatively larger lattice parameter than host lattice of Ceria (5.410 Å) confirms the complete dissolution of Gd into ceria lattice. The average crystallite size calculated by using Debye Scherrer’s formula for GDC and Ni lies between 48-49 nm and 46-49 nm respectively. The peak (331) corresponding to GDC10 is absent for
N4G1-r whilst it contains one additional peak (220) of Ni. It might be due to higher concentration of Ni in Ni-GDC cermet indicating that with increase in Ni content, the intensity of peak corresponding to Ni goes on increasing [18].

6.3.1.2. Microstructural Studies (FE-SEM)

Fig. 6.2 (a-d) shows cross sectional FE-SEM micrographs of Ni-GDC nano-composites before and after reduction. From Fig. 6.2 it was observed that Ni-GDC anode has a homogeneous porous structure with hexagonal grains. Ni phase is dispersed uniformly in the anode. The porosity seems to be an open porosity which is required for the gas phase transport. As NiO content in the nanocomposite anode is increased, porosity of the anode increased.
This is consistent with result obtained from density measurement of the series of NiO-GDC nano-composites as discussed in Chapter V and [19]. The homogeneous microstructure leads to good connection between Ni-Ni, GDC-GDC and Ni-GDC grains, which are important in fabricating a high performance Ni-GDC anode because of improving triple phase boundaries (TPB) [20].

A high TPB length and good Ni interconnections both contributes to improve electrochemical performance of Ni-GDC cermet anode [21].
6.3.1.3. Electrical Conductivity

Electrical conductivity data was collected under hydrogen atmosphere condition with rectangular pellets having dimensions 20mm × 6mm × 2mm. The pellets heated in hydrogen atmosphere from room temperature to 900 °C for 4 hrs. The conductivity data was then collected during cooling from 900 to 200 °C, by using standard four probe techniques. The electrical conductivity of data was collected as a function of temperature. A temperature controller, Oxford ITC 503 (Oxford Instrument, Oxon, UK) was used for heating the pellets in the temperature range 200 – 900 °C.

For oxide materials, there is conduction in the temperature range 200 – 900 °C is due to a small polaron hopping mechanism. In case of polar materials when a sufficiently slow moving free charge carrier (a slow conduction electron or a valence band hole) remains in the neighborhood of a particular atomic site over a time interval longer than a typical vibrational period, the surrounding lattice atoms become polarized by the electric field of the charge of the carrier by displacement of positive and negative ions in opposite directions and relax into a new equilibrium configuration. These ionic displacements will create a potential well for the charge carrier. Thus, the carrier gets trapped inside the well created by the carrier-induced displacement of ions. A polaron consists of the carrier and this associated polarization of the lattice atoms. If the interaction of the carrier with the lattice is sufficiently strong and the extent of lattice distortion surrounding the carrier is confined to the small region of the crystal and is of the order of one typical unit cell distance, polaron so formed is called a small polaron [22]. The small polaron moves by thermally activated hoping mechanism. The conductivity by small polaron hopping fits linearly into the Arrhenius relation:

$$\sigma = \frac{A}{T} \exp \left( \frac{-E_a}{kT} \right) \quad (6.1)$$
Arrhenius plots of electrical conductivity for Ni-GDC samples (N3G1-r and N4G1-r) in hydrogen atmosphere in temperature range 200 – 900 °C by four probe technique are shown in Fig. 6.3.

![Arrhenius plot of Ni-GDC anode with different Nickel content](image)

**Figure 6.3:** Arrhenius plot of Ni-GDC anode with different Nickel content N4G1-r (40mol % NiO and 60mol% GDC10) and N3G1-r (30mol % NiO and 70mol % GDC10)

Conductivity of both N4G1-r and N3G1-r increases with the temperature which indicates that conduction mechanism in these cermets is thermally activated process. The linearity of the plot of \( \ln (\sigma T) \) vs \( 1000/T \) shows that the charge carrier transport process is caused by the small polaron hopping mechanism. It was observed that electrical conductivity values of N3G1-r and N4G1-r at 900 °C in hydrogen atmosphere are found to be 2.11 S/cm and 1.944 S/cm respectively. At higher temperature (900 °C) the cermet containing higher Ni content gives lower conductivity; it might be due to metal like behavior of N4G1-r. Hence the conductivity of anode N3G1-r is slightly higher than that of the anode N4G1-r. The existence of pores would results in either diminished
electronic or ionic conductivity since they have occupied the place where there otherwise would be nickel or GDC particles to provide electrical connection [3, 23]. The conductivity results are consistent with microstructure observed in Fig. 6.2 i.e. porosity of the cermet goes on increasing with Ni content and hence conductivity at higher temperature decreases.

The activation energy ($E_a$) in equation (6.1) is the minimum energy required by small polaron to hop from one site to another neighborhood site at given temperature. The activation energies for these two cerments were calculated from the slopes of the Arrhenius plots shown in Fig. 6.3. The values of $E_a$ for N3G1-r and N4G1-r anodes are 1.28 eV and 1.29 eV respectively.

6.3.2. GDC/NiO -GDC Structure (Half Cell)

Structural studies of half cell was carried out by XRD (Phillips-3710 powder X-ray diffractometer) in the 2θ range 10–90° using CuK$_{\alpha1}$ radiation ($\lambda= 1.54056$ Å). Cross-sectional study of half cell was done by using scanning electron microscope (JEOL JSM 6360 model). While the ac electrical conductivity measurements were carried out with the help of electrochemical impedance spectroscopy in temperature range of 500 °C to 700 °C using platinum electrodes in air. Pt paste obtained from Ma TecK Germany was applied on pellets and heated at 600 °C in air for 4 hr. to remove the organic binders. The complex impedance was measured at different signal frequencies ranging from 5Hz to 10 MHz using a Solartron SI 1260 impedance analyzer. These measurements were performed at an interval of 25 °C, each temperature being kept constant with an accuracy of ± 5 °C. Sufficient stabilization time was ensured at each particular temperature. The data analysis was done using Z-view frequency response analyzer.
6.3.2.1. X-ray Diffraction (XRD)

Fig. 6.4 shows XRD pattern of (a) NiO$_{40}$GDC$_{60}$ substrate and (b) spray deposited GDC thin film on NiO$_{40}$GDC$_{60}$ substrate (annealed at 1000 °C). The peaks originating from the substrate are almost negligible in case of XRD of GDC thin film (i.e. Fig. 6.4 (b)) as film thickness was ~ 8µm. The lattice parameter of GDC thin film was 5.435Å, which is nearly same as that of the standard lattice parameter.

The reflection peaks due to GDC thin film, deposited by SPT are seen broad enough forming nano crystalline grains [16]. The crystallite size values calculated from Debye Scherrer’s formula lies between 14-16 nm.

![XRD patterns of (a) NiO$_{40}$GDC$_{60}$ substrate and (b) GDC thin film on NiO$_{40}$GDC$_{60}$ substrate (N for NiO and G for GDC)](image)

**Figure 6.4** XRD patterns of (a) NiO$_{40}$GDC$_{60}$ substrate and (b) GDC thin film on NiO$_{40}$GDC$_{60}$ substrate (N for NiO and G for GDC)
6.3.2.2. Scanning Electron Microscopy (SEM)

Fig. 6.5 (a and b) shows an SEM micrographs of the surface and cross-section respectively of NiO-GDC/GDC half cell.

Figure 6.5 SEM images of (a) Surface of GDC thin film  
(b) cross section of GDC thin film deposited on NiO-GDC
It is important to note that the resulting GDC thin film is as thin as ~ 6 - 8 µm and it appears to be dense enough. Although there is appearance of crack like morphology, no open porosity was observed. GDC film is well adhered to the NiO-GDC substrate forming dense interface between substrate and the film.

6.3.2.3. Electrochemical Impedance Spectroscopy (EIS)

The impedance measurement is a well-developed tool to separate out the bulk and grain boundary contribution to the total conductivity. The impedance spectrum is usually represented as negative of imaginary component of impedance ($-Z''$) versus real component of impedance ($-Z'$), which is referred as Nyquist plot.

The electrical characterization of GDC / NiO-GDC structure was carried out using impedance spectroscopy in the temperature range 500 – 700 °C. The typical electrochemical impedance spectra at different temperatures are shown in Fig. 6.5 (a-d). The impedance spectra show trend of semicircles. The impedance data were analyzed by using Zview version 2.4, software.

The intercepts on real axis at high frequency correspond to the half cell ohmic resistance while the semicircle is assigned to the charge transfer across the electrolyte-electrode interface. This impedance is very high since the half cell is not being operated in reducing atmosphere and because of the presence of NiO rather than Ni. Impedance data analysis was done by using Z-view program.

The single semi-circle is fitted with two RC networks. Fig. 6.6 shows fitted result for half cell at 665 °C. The corresponding conductivity values at 700 °C are $6.83 \times 10^{-3}$ S.cm$^{-1}$ and $1.11 \times 10^{-3}$ S.cm$^{-1}$ respectively for grain (g) and grain boundary (gb).
Figure 6.5 Impedance spectra of GDC / NiO-GDC structure (a) at 635 °C (b) at 645 °C
Figure 6.5 Impedance spectra of GDC / NiO-GDC structure
(c) at 655 °C (d) at 665 °C
Figure 6.6 Typical fit result from impedance analysis of NiO GDC/GDC half cell at 665 °C
6.3.3. NiO-GDC/GDC/LSCF-GDC Structure

To study the performance of SOFC, I-V curves have been given much more importance. I-V curves show only overall loss of a cell but the electrochemical impedance spectroscopy is one of the most promising methods for describing complex electrochemical systems with different relaxation time constants. So in the present study, importance is given to study impedance spectroscopy of the NiO-GDC/GDC/LSCF-GDC single cell.

However, in this study, impedance spectra are not recorded under cell conditions but they are recorded in air. The impedance spectra of NiO-GDC/GDC/LSCF-GDC structure (single cell) measured in the temperature range 500-600 and 600 – 700 °C is respectively shown in Fig. 6.7 (a and b). It shows presence of two semi-circles. This indicates at least two different electrode processes, corresponding to the high and low frequency arcs.

Kao et al. [26] has also reported that the high-frequency arc can be attributed to the charge-transfer of oxygen ions from electrode to the electrolyte. The low-frequency arc can be attributed to the oxygen adsorption and desorption and the diffusion of the oxygen ions on the electrode. Increasing the temperature, the shape of the impedance curve became smaller reducing the impedances.

The different impedance arcs were allocated to the corresponding processes using the capacitance (CPE) related to each arc. As a result, R$_1$ corresponds to the electrolyte ($C_1 \sim 1.05 \times 10^{-11}$ F cm$^{-2}$). The resistances R$_2$ and R$_3$ correspond to the electrodes (anode and cathode) ($C_2 \sim 2.37 \times 10^{-6}$ F cm$^{-2}$, $C_3 \sim 2.11 \times 10^{-8}$ F cm$^{-2}$). The semi-circles with pseudo capacitance in the range $4 \times 10^{-3}$–$10^{-5}$ F cm$^{-2}$ are assigned to the charge transfer in [25].
Figure 6.7 Impedance spectra for NiO-GDC/GDC/LSCF-GDC structures in the temperature range (a) 500-600 °C and (b) 600-700 °C
The fitting of the impedance data was carried out with the commercially available program Zview. The equivalent circuit used for fitting is shown in Fig. 6.8 (a). The applied equivalent circuit consists of an ohmic resistor $R$ and three RC-elements connected in series.

![Equivalent circuit](image)

**Figure 6.8 (a)** Equivalent circuit (consisting of a series connection of three RC-elements ($R_1C_1$, $R_2C_2$, and $R_3C_3$) and an ohmic resistance ($R$)) employed for evaluating the electrode and electrolyte resistance

Typical fitting result has been shown in Fig. 6.8 (b) and the fitting results using the Zview program are listed in Table 6.3

![Impedance analysis result](image)

**Figure 6.8 (b)** Typical fit result from impedance analysis of NiO-GDC/GDC/LSCF-GDC cell at 700 °C
Table 6.3 Fitting parameters from AC-impedance results obtained at different temperatures

<table>
<thead>
<tr>
<th>Fitting resistances</th>
<th>Temperature °C</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>600</td>
</tr>
<tr>
<td>R</td>
<td>45.68</td>
</tr>
<tr>
<td>R1</td>
<td>3515</td>
</tr>
<tr>
<td>R2</td>
<td>170380</td>
</tr>
<tr>
<td>R3</td>
<td>5028</td>
</tr>
</tbody>
</table>

In the equivalent circuit, R is resistance due to connecting wires. The resistances R1, R2 and R3 are the charge-transfer resistances. From Table 6.3, it is observed that the charge transfer resistance decreases with increase in temperature. R increases with temperature because connecting wires are of Pt. However, impedance under cell conditions will have to be done for further insights.

6.4. Conclusions

In the present research Ni-GDC cermet has been studied as an anode for intermediate temperature Solid Oxide Fuel Cell (IT-SOFC). Two compositions, Ni30GDC70 and Ni40GDC60 were optimized as anode for IT-SOFC. The XRD studies of these anodes show that there is no NiO in the anode. Hence NiO-GDC nanocomposite was successfully reduced to form Ni-GDC anode. The microstructural characterization of these anodes shows porous structures. From SEM micrographs it was confirmed that with increase in NiO content in anode, porosity goes on increasing which is consistent with density measurement studies from chapter V. An anode, Ni30GDC70 showed higher conductivity ~ 2.11 S/cm while Ni40GDC60 showed a conductivity of ~ 1.944 S/cm.
Hence after confirmation of anode composition, the study was directed towards the synthesis of NiO-GDC/GDC/LSCF-GDC single cell. For the synthesis of single cell firstly GDC thin films were deposited on NiO-GDC ceramic substrate. The whole assembly of NiO-GDC/GDC forms half cell. The XRD study of half cell confirms the phase pure synthesis of GDC thin films by Spray Pyrolysis Technique. Thickness of GDC thin film was ~ 6-8 µm and it is sufficient to form electrolyte layer on cermet substrate. Cross sectional SEM studies confirm that SPT synthesized GDC thin films were thin enough and dense satisfying requirements of electrolyte for IT-SOFC. SPT gives reliability in sintering temperature as low as 1000 °C. It is the main advantage as GDC needs higher temperature for its densification. The impedance of half cell decreases with increase in temperature. The conductivity of half cell at 700 °C was found to be $6.83 \times 10^{-3}$ S.cm$^{-1}$ and $1.11 \times 10^{-3}$ S.cm$^{-1}$ respectively for grain (g) and grain boundary (gb). The ability of co-sintering at lower temperature avoids the possibility of electrode/electrolyte interfacial problems during fabrication of complete cell and allows better electric performance.

After the characterization of half cell, NiO-GDC/GDC/LSCF-GDC structure (single cell) was fabricated and characterized by impedance spectroscopy in air. It confirms that the charge transfer resistance and electrolyte resistance decreases with increase in temperature.
Chapter VI: Formation and characterizations of NiO-GDC/GDC structure for IT-SOFC

References
