

Synthesis and characterization of $Ba_{0.5}Sr_{0.5}(CO_{0.8}Fe_{0.2})_{1-x}Ti_xO_{3-\delta}$ (BSCF) Cathode for Solid Oxide Fuel Cell

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Abstract- The BSCF, Barium Strontium Cobalt Iron Titanate $\{Ba_{0.5}Sr_{0.5}(CO_{0.8}Fe_{0.2})_{1-x}Ti_xO_{3-\delta}\}$ [Where δ is the deficiency of oxygen & x is various compositions], powders have been synthesized by sol-Gel process using nitrate based powdered chemicals for SOFC applications as these powders are more useful for Cathodes and anodes for SOFCs since these powders are considered to be more promising cathode materials for SOFC. Ionic conduction, Adsorption and desorption rates are the major processes that control the electrode reactions. They contribute to the over potential. To obtain the low potential cathode materials suitable for the electrolytes, SOL-GEL method is used and nano powders are prepared. The Chelating agent used is Acetic acid, Ethylene glycol & Ammonia as dispersant. These powders were kept for calcinations at $900^{\circ}C$ for 16 hr and at $1100^{\circ}C$ for about 6 hr in crucibles of high alumina in furnace. These were characterized by XRD, SEM with EDAX, Densities, TGA and conductivities. XRD results proved the formation of Perovskite phase at all calcinations temperatures and the crystallite sizes were found to be 94.49nm, 103.9nm, 295.29 nm. From SEM, it is found that for some samples, there is presence of extreme porous particles present in nano sizes and conductivities, densities are studied

Index Terms- SEM/EDAX, TGA/DSC; XRD; Density; $Ba_{0.5}Sr_{0.5}(CO_{0.8}Fe_{0.2})_{1-x}Ti_xO_{3-\delta}$

I. INTRODUCTION

SOFCS (Solid oxide fuel cells) are one of the vital energy conversion devices with high oxygen vacancies. BSCF proved as good materials for solid cathode with appealing properties like ionic conductivity, super conductivity, Ferro electricity, magnetic resistance etc., Many cathodes were used in LSM, SSC, LSCF, BSCF, etc., but, the thermo-mechanical suitability between electrolyte and cathode can be improved by mixing cathode material with new element or a small impurity like Titanium. Such composite cathode will have a better ionic conductivity. As the cathode over potential is significant in SOFC's, it can be reduced by adding Titanium to BSCF. By increasing the percentage of Ti in site 'B' of ABO_3 (Perovskite structure), one can increase the efficiency and conductivity. Conventional SOFCs with an Ytria - Stabilized Zirconia (YSZ) cathode are not being operated at $900^{\circ}C$ or higher temperature operation causes serious problems such as internal mechanical stress due to the difference in the thermal expansion coefficients of the materials and chemical reactions. Therefore, operating temperature lower than $700^{\circ}C$ is desirable

[1]. because electrochemical and conduction processes in SOFC are strongly thermally activated, a reduced operating temperature leads to another difficulty in large voltage losses due to the ohmic resistance of the materials and polarization at the electrodes. Most of the voltage losses are generated from the ohmic resistance of the electrolyte, a number of approaches have been reported preparing a thinner electrolyte which reduces the corresponding resistance [2, 3], and high power density was obtained using YSZ thin film electrolytes [4, 5]. There are generally two categories making a robust anode-electrolyte-cathode cell while maintaining thin electrolyte layer: Cathode supported cell [6, 7] and

Anode supported cell [8-10]. Despite the fact that Cathode - supported cell can be produced by ceramic process (Toh) as well as by atmospheric plasma spraying (Siemens-Westing house) with scalable processes. Reducing the operating point temperature below $800^{\circ}C$ causes an increase of over potential at low temperature. Currently, La Sr Mn O (LSM) is commonly used as cathode material because of its high catalytic activity for oxygen reduction, thermal and chemical compatibility with Ytria stabilized new power generation system for their high - Zirconia (YSZ) [11, 12]. Recently strontium doped cobaltite has attracted much attention because of its mixed conduction characteristics and its relatively high ionic conductivity. Strontium doped samarium cobaltite (SmSrCoO: SSC) shows even higher conductivity, up to $10 S cm$ [13]. It shows good compatibility with ceria cathode. The cathodic reaction mechanism of SSC is not clearly understood. Ionic conduction, Adsorption and desorption rates are known as the major processes that control the electrode reactions. They contribute to the over potential. In practical the adsorption and desorption rates of SOFC are not equal. Therefore separate adsorption and desorption rates must be determined.

In the present paper, the objective is to prepare a cathode suitable for SOFC in single chamber fuel cell conditions. Hence, materials with a small percentage of Ti (with increasing values) are prepared and their characterization/values are compared.

Out of various fuel cells, solid-oxide fuel cells (SOFCs) have the benefit of eco friendly power generation with fuel [15] flexibility. High operating temperatures resulted in highly expensive, less compactness and low compatibility of cells. Hence, SOFCs [16] have been designed and developed and cathodes are prepared as they exhibit poor response for traditional and conventional cells, by using BSCF ($Ba Sr Co Fe$) newly, which exhibit high power densities i.e., $1,010mW/cm^2$ and $402mW/cm^2$ at $600^{\circ}C$ and $500^{\circ}C$. In this Hydrogen is used as fuel and air as cathode gas. BSCFs are suitable for single

chamber fuel cell operation. Hence, the alternative Perovskite Cathodes are chosen for high activity oxygen reduction.

These SOFCs will have high oxygen diffusivity. The important requirements for SOFC cathode materials are **i)**High electronic conductivity **ii)**Chemically suitable with electrolyte, **iii)**Stable in oxidation, **iv)** Large triple phase boundary, **v)**High ionic conductivity, **vi)**Suitable thermal expansion coefficient, **vii)**Simple fabrication and **viii)** Low cost. There are many materials which explain the reaction mechanisms of SOFC cathodes, like transport of oxygen, electrode kinetics, and involvement of chargeable particles in electrochemical processes. Reduction of oxygen at cathode is one of the important factors which contribute to the total resistance of the cell. Electro catalytic activity is also another important factor of SOFC. Hence by enhancing the porosity of cathode; one can increase the oxygen exchange and diffusion in cathode which results to higher current density in the cell.

Since six years $\text{Ba}_{0.5}\text{Sr}_{0.5}\text{Co}_{0.8}\text{Fe}_{0.2}$, $\text{Ba}_{0.5}\text{Sr}_{0.5}\text{Co}_{0.2}\text{Fe}_{0.8}$ and other combinations have got much attention for intermediate temperature of SOFCs but, this reduction in temperature below 800°C causes an increase of the over potential of SOFC cathode. Therefore, to obtain the low potential cathode materials, the material $\text{Ba}_{0.5}\text{Sr}_{0.5}[\text{Co}_{0.8}\text{Fe}_{0.2}]_{1-x}\text{Ti}_x\text{O}_{3-\delta}$ is designed, { where 'x' is Various Compositions and 'δ' is deficiency of oxygen (0 or 0.1) }. By taking such doping of titanium with BSCF, the weight % was found to be 70%. Compared to SDC (Samaria- doped Ceria) this is 30% defective.

The structure is of $\text{ABO}_{3-\delta}$, Perovskite. In this, oxygen ion vacancies will be pronounced along $(\text{B}^{3+/4+})-\text{O}^{2-}-\text{B}^{3+/4+}$, due to overlapping of the oxygen p-orbital and transition-metal d-orbital. When Ba is in site 'A', it increases the amount of B^{4+} ions and increases oxygen vacancies. In recent works, Ito et al. has shown the presence of both covalent and ionic bonds in $\text{BSCFO}_{3-\delta}$ and increases electron and oxygen ion conductivity at fairly low values of temperature. This clearly explains that the stoichiometry of oxygen decreases with increase in temperature and hence reduction in cations in site 'B' [17, 18]. SOFC performance will purely depend on sample preparation, its reactants, chemicals, stoichiometry, calcinations, sintering, acids, and bases. In recent papers, it is suggested that, by varying x and δ values in the $\text{BSCFO}_{3-\delta}$ [5582] would affect the performance. Hence, in the present study, investigation has been studied with $\text{Ba}_{0.5}\text{Sr}_{0.5}[\text{Co}_{0.8}\text{Fe}_{0.2}]_{1-x}\text{Ti}_x\text{O}_{3-\delta}$ by taking δ=0, δ=1 and x=0.10, 0.15, 0.20, 0.25 with different thermal properties. BSCF has proved as a good material for solid oxide fuel cell cathode [20,

21, and 22]. These have properties like ionic conductivity, Ferro electricity.

There are various models developed to explain reaction mechanisms off cathodes, of SOFCs [9, 10]. But, BSCF cathode preparation doped with Titanium and the over potential study is an important factor. A cathode should possess high electro catalytic activity for the reduction of oxygen. A good cathode should have high oxygen exchange and diffusivity so that, there will be rapid movement of oxygen ions [23]. This can also be obtained by increasing the porosity of BSCF cathode [24] which provide more dispersion of gases increasing reduction of oxygen. Hence, the current density will be generated within the cell.

II. EXPERIMENTAL

2.1 Materials:

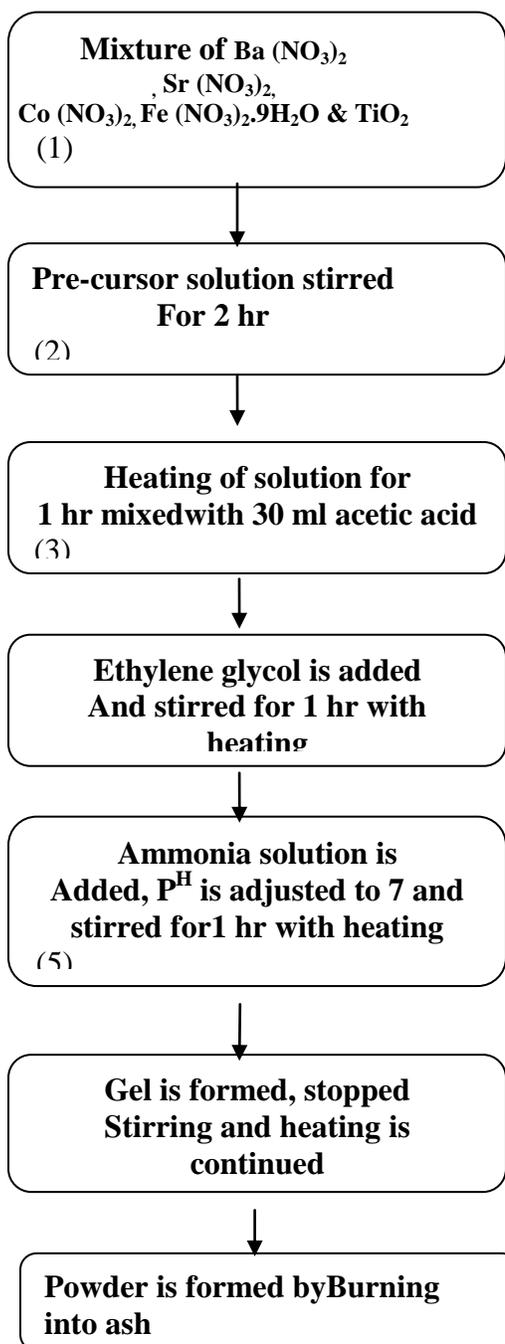
Commercial powders of AR grade of Aldrich Company were used in this work for the preparation of Cathode. Ethylene di-amine-tetra-acetic acid (EDTA), ethylene glycol, anhydrous citric acid, nitrate salts of Barium, Strontium, Cobalt, Iron and Zirconium oxide were purchased from Sigma Aldrich, USA. Ammonia solution is used as base. In the present observations, the Nano crystalline cathode material of $\text{Ba}_{0.5}\text{Sr}_{0.5}(\text{Co}_{0.8}\text{Fe}_{0.2})_{1-x}\text{Ti}_x\text{O}_{3-\delta}$ by varying x values, (x = 0, 0.10, 0.15, 0.20) BSCF $\text{Ti}(5582\text{Ti})$ powders were prepared by sol-gel process as it is one of the economical ways and characterizations like SEM/ EDAX of LEO SUPRA 55 VD ultra high resolution of ZEISS, XRD TGA/DTA, Impedance Analyzer were used. Barium nitrate $\text{Ba}(\text{NO}_3)_2$, Strontium Nitrate $\text{Sr}(\text{NO}_3)_2$, Cobaltous Nitrate $\text{Co}(\text{NO}_3)_2$, Fluka $\text{Fe}(\text{NO}_3)_2 \cdot 9\text{H}_2\text{O}$ & TiO_2 , are used.

2.2 Method

Precursor solution is prepared by taking above powders by appropriate formula and mixing, aqueous solution of the above chemicals in molar ratio of 0.5:0.5 and 0.8:0.2 & required Acetic acid, ammonia, are added and $\frac{c}{n}$ ratio is maintained as 0.5. The solution is taken in borosil glass beaker and placed on a Magnetic stirrer with hot plate for about 4 hr with appropriate speed and by heating at about 80°C to 100°C , the solution is stirred till it becomes like a gel and then, the speed is reduced, heated for some time till it becomes ash after burning. The ash is calcined first at 750°C , for 8 hr, and at 900°C , for 8 hr and at 1050°C , 1100°C each for 2 hr and after preparing pellets, their densities are calculated as in table.1

Table. 1 Density calculations

Sam ple	Wt. in air (g)	Wt. in xylene (g)	$\rho_{Exp} = \frac{Wt.inair}{Loss\ of\ wt.in\ xylene}$	a (Lattice constant)	$\rho_{Th} = \frac{nM}{a^3 N_A}$	Density $\% = \left\{ \frac{\rho_{Exp}}{\rho_{Th}} \right\} * 100$
Ti ₁	0.2376	0.1943	5.5169	4.0016 Å	5.6419	97%
Ti ₂	0.3773	0.3108	5.4120	3.9793 Å	5.7727	95%
Ti ₃	0.3866	0.3167	5.5198	3.9804 Å	5.6051	97%
Ti ₄	0.3183	0.2594	5.4179	3.9873 Å	5.6620	96%



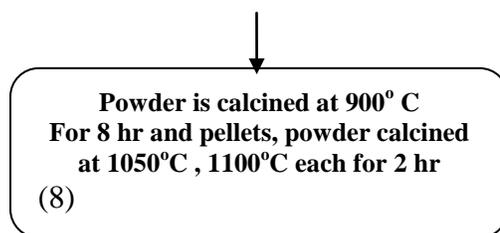


Figure 1. Flow chart of method of preparation of sample

III. CHARACTERIZATION

3.1 XRD Characterization:

X-ray diffraction (XRD) analysis of the sintered samples is carried out with XRD at 40 KV and 30mA, using $\text{CuK}\alpha$ radiation with diffraction angle (2θ) range from 20° to 80° and particle size is determined by line broadening technique as shown below for all four samples. It is clear that, powder is partially amorphous and it is observed that calcined powders were of Perovskite structure and these were found to be similar with other XRD's of various authors. It is observed that, at higher temperatures, the noise in XRD is reduced and still it can be reduced by calcining the samples at about 1100°C , 1150°C or more.

Table 2. Cathode (BSCFTi – 5582Ti) powders by sol-gel process.

Sample	Cathode powder	c/n
Ti_1	$\text{Ba}_{0.5}\text{Sr}_{0.5}[\text{Co}_{0.8}\text{Fe}_{0.2}]_{0.9}\text{Ti}_{0.1}\text{O}_2$	0.5
Ti_2	$\text{Ba}_{0.5}\text{Sr}_{0.5}[\text{Co}_{0.8}\text{Fe}_{0.2}]_{0.85}\text{Ti}_{0.15}\text{O}_2$	0.5

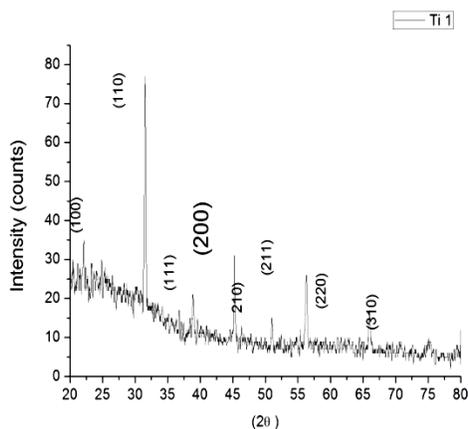


Figure.2 XRD Patterns of $\text{Ba}_{0.5}\text{Sr}_{0.5}(\text{Co}_{0.8}\text{Fe}_{0.2})_{L-x}\text{Ti}_x\text{O}_{3-\delta}$ with $\delta=0$, $x=0.1$ calcined at 1050°C

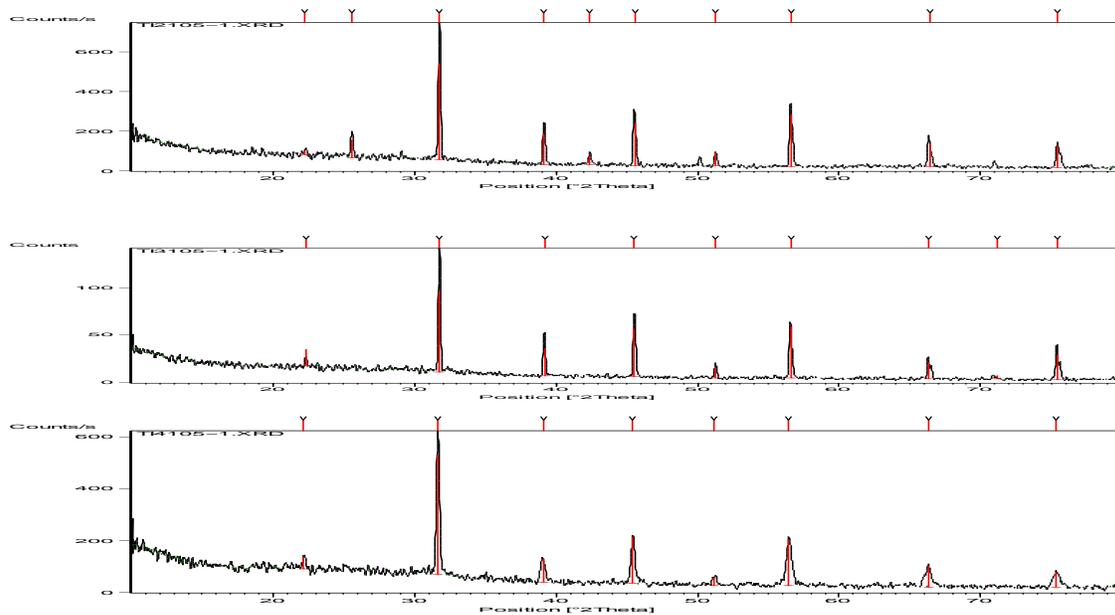


Figure3. XRDs of Ti₁, Ti₂, Ti₃ samples

Table. 3XRD data for determining the average particle size of BSCF Ti (5582 Ti)

Sample	2θ	θ	Cos θ	FMHM (β)	Particle size $D = \frac{0.9\lambda}{\beta \cos\theta}$ nm
Ti ₁	31.571	15.786	0.96228	0.4723	30.506
Ti ₂	31.7698	15.8849	0.9618	0.3149	45.77
Ti ₃	31.6666	15.8333	0.9620	0.4723	30.51

Where $\lambda = 1.540510$ nm

3.2 SEM with EDAX Characterization:

Few amounts of powders and pellets were used for study of compounds present in the samples after calcinations using ZEISS Scanning Electron microscope and images of various magnifications were done. The sem photographs of Ti₁ & Ti₂ samples are as under.

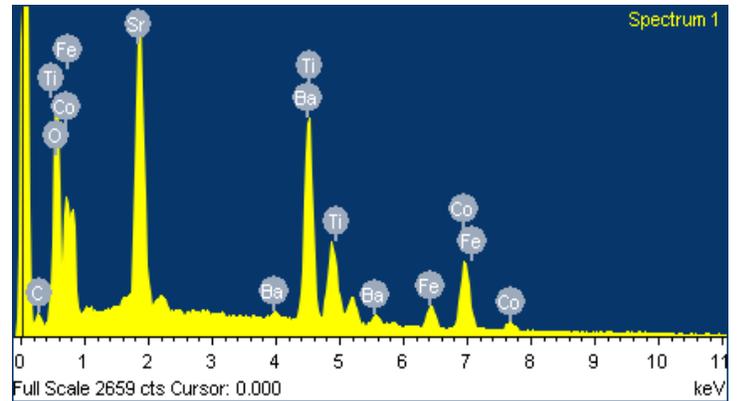
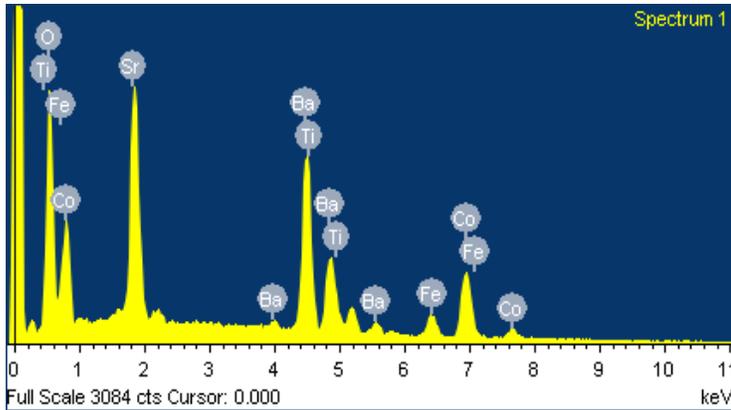


Figure.3 (a) SEM with EDAX of Ti1 sample

Figure. 3(b) SEM with EDAX of Ti 2 sample

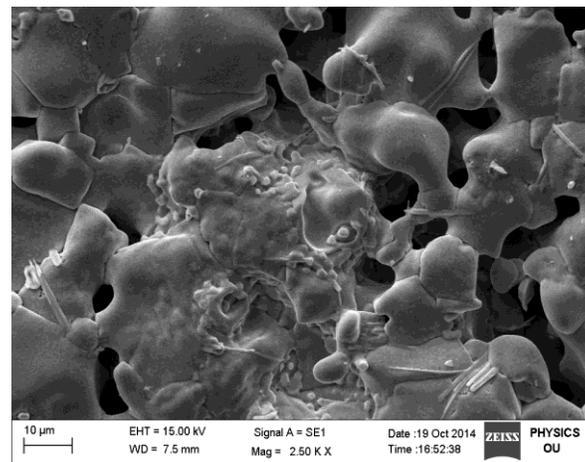
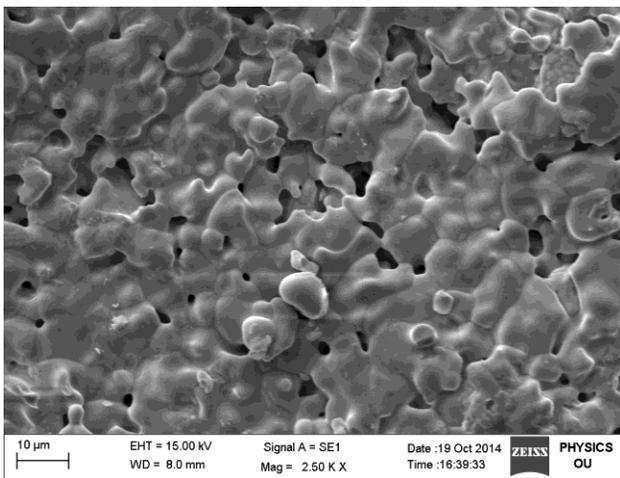


Figure .4(a) SEM Photograph of sample 1

Figure. 4(b) SEM Photograph of sample 2

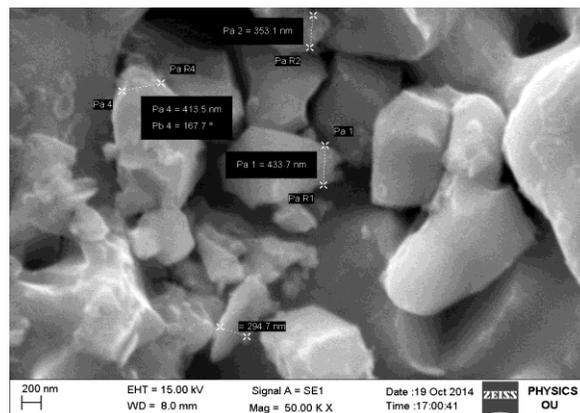
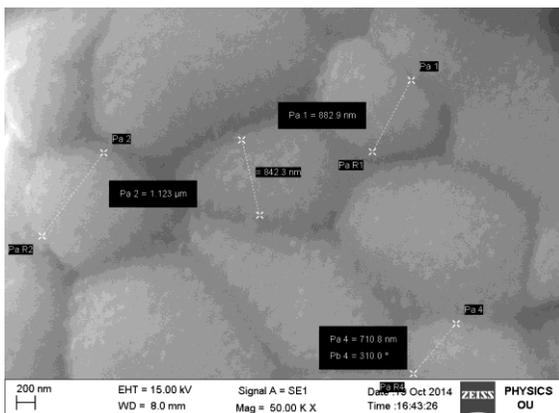


Figure.5 (a) SEM of sample 1, particle size

Figure. 5(c) SEM of sample 2, particle size

From SEM with EDAX analysis, it is found that the atomic percentage as taken in the formula is not changed and all atoms are present in the same proportion, particle sizes are noted which are in good coincidence with the data taken from XRD. The figures show the presence of Ba, Sr, Co, Fe, Ti, C peaks. The appearance of c may be due to usage of acetic acid. This C % has been reduced by calcining the samples at a higher temperature.

IV. TGA/DTA CHARACTERIZATION

The curves in figures 6(a) and 6(b) correspond to TGA (weight loss in % Vs temperature) and DTA (Rate of losing weight Vs temperature) for Ti_1 and Ti_2 samples. DTA is studied with difference in temperature and flow of heat between the sample and a reference. Moisture, Thermal stability and composition are studied simultaneously by TG/DTA referring Exothermic and Endothermic processes.

Powder samples of Ti_1 of 10.6 mg and Ti_2 of 7.6 mg are taken and corresponding curves are recorded. The weight loss is observed for three times in TGA first at 78°C due to evaporation of moisture, second at 490°C due to evaporation of nitrates and third at 640°C due to evaporation of other impurities because of usage of acids, bases etc.,. The melting point can be at about 1070°C or 1100°C .

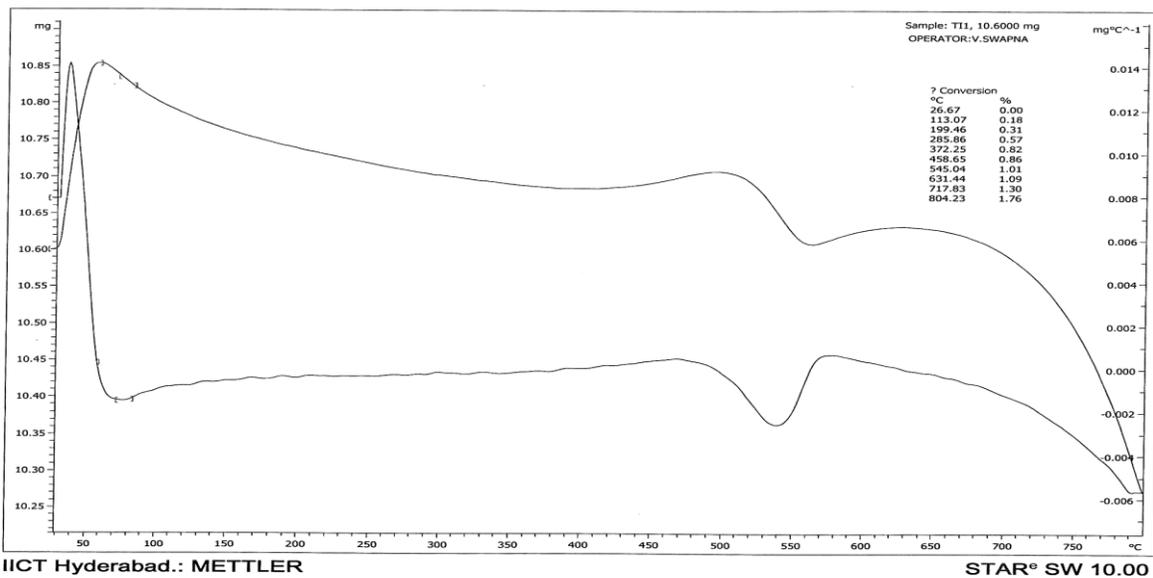


Figure. 6(a) TGA/DTA Curves for $Ba_{0.5}Sr_{0.5}[Co_{0.8}Fe_{0.2}]_{0.9}Ti_{0.1}O_2$

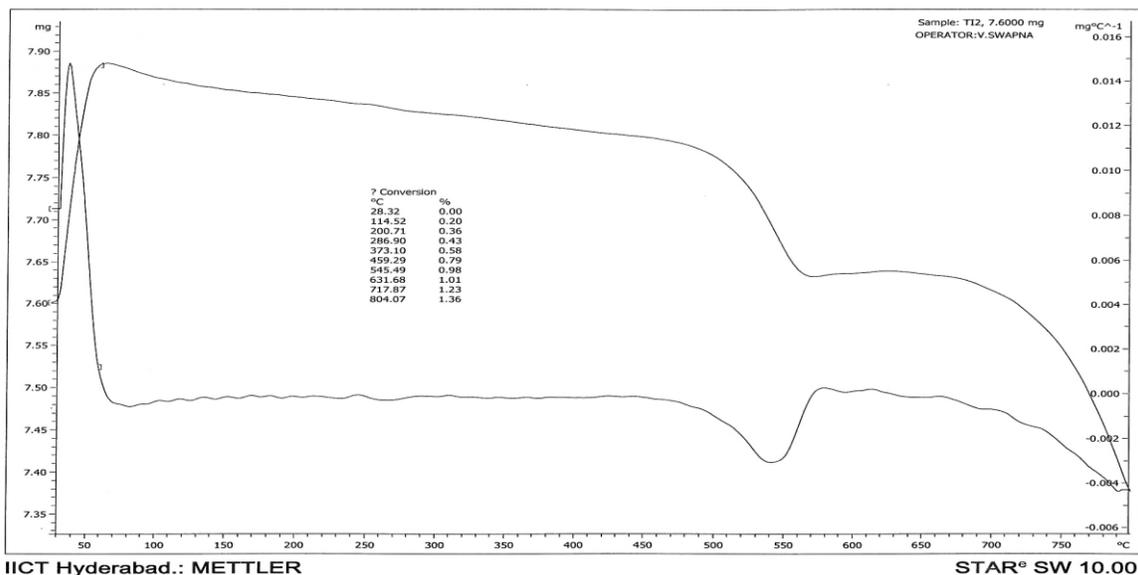


Fig. 6(b) TGA/DTA Curves for $Ba_{0.5}Sr_{0.5}[Co_{0.8}Fe_{0.2}]_{0.85}Ti_{0.15}O_2$

V. IMPEDANCE ANALYSIS

To characterize the electrochemical properties of BSCFTi cathode, AC impedance values are measured and characterization of cathode processes is based on the application of ac potential

$E(t) = E_0 \cos(\omega t + \phi)$ of minimum amplitude such that ac current $I(t) = I_0 \cos(\omega t - \phi)$ is obtained.

$$\frac{E(t)}{I(t)}$$

Impedance, $Z = \frac{E(t)}{I(t)}$ is calculated at different AC frequencies up to few MHz

The impedance measurements characterizes the physical and chemical processes related to time constants ,electron transfer at high frequencies and mass transfer at low frequencies. In the present study, the impedance measurements were carried from room temperature to 150⁰ C and Nyquist plots ($Z_s = Z_{Real} + Z_{Imaginary}$) are drawn as shown in figures 7(a) for Ti₁ sample and 8(a) for Ti₂ sample respectively.

The cole-cole plots are observed for Z^I and Z^{II} plots as depicted in figures 7(a) & 8(a) .

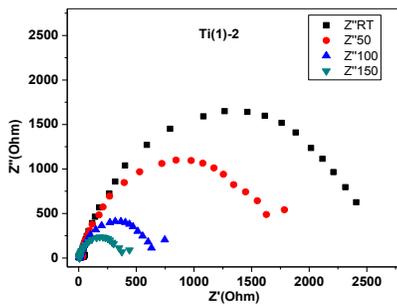


Figure .7(a) COLE-COLE plot of Ti sample

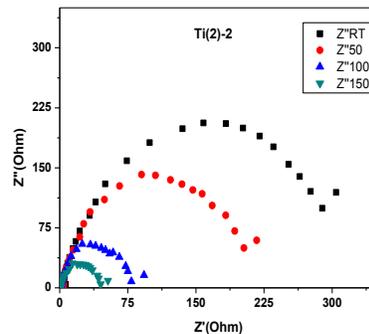


Figure .8(a) COLE-COLE plot of Ti2 sample

The stable behavior of the real part of the impedance with frequency is observed with increase of temperature as shown in figures 7(b) & 8(b).

From cole-cole plots one can calculate the grain resistance (R_g) and grain boundary (R_{gb}) resistance and their temperature dependence can be observed.

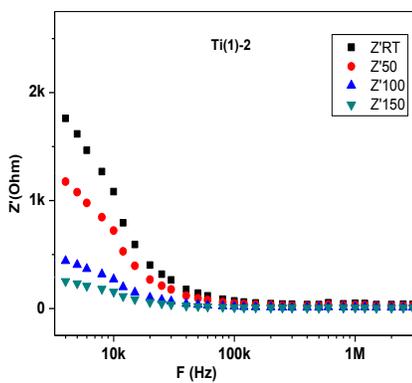


Figure.7 (b) Z Vs Frequency curves

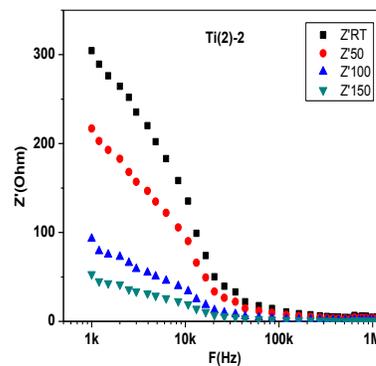


Figure.8 (b) Z Vs Frequency curves

It is observed from figures 7 (c) and 8(c) that, the peak frequencies were shifted for imaginary component of Z^{II} Vs Frequency as temperature increases. This is the relaxed behavior of a sample.

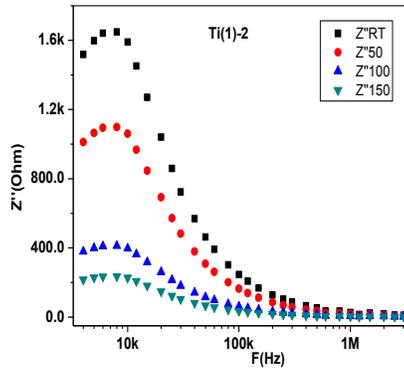


Figure.7(c) Z Vs Frequency curves

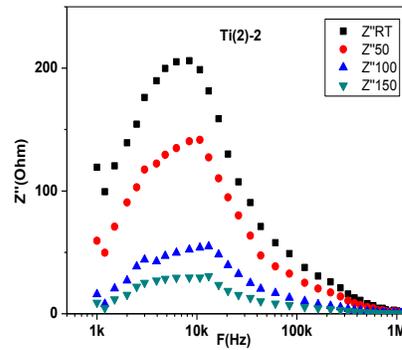


Figure.8(c) Z Vs Frequency curves

AC conductivity is calculated by the formula $\sigma_{AC}^l = \frac{Y^l t}{A}$ where Y^l is called admittance, t is thickness of the sample and A is area of the sample. The conductivities are tabulated in tables 4 & 5 below.

Table 4. Calculation of conductivities through AC Impedance of Ti 1 Sample:

S.No	Temperature	Resistance (ohm)	Admittance (Y)	Area(A) m ²	Thickness(t) m	$\sigma_{AC}^l = \frac{Y^l t}{A}$
1	Room temp.	1750	5.714×10^{-4}	69.42×10^{-6}	1.46×10^{-3}	0.0120
2	50	1200	8.33×10^{-4}	69.42×10^{-6}	1.46×10^{-3}	0.01752
3	100	500	2×10^{-3}	69.42×10^{-6}	1.46×10^{-3}	0.042059
4	150	250	4×10^{-3}	69.42×10^{-6}	1.46×10^{-3}	0.08411

Table 5. Calculation of conductivities through AC Impedance of Ti 2 Sample:

S.No	Temperature	Resistance	Admittance	Area(A) m ²	Thickness(t) m	$\sigma_{AC}^l = \frac{Y^l t}{A}$
1	Room temp.	320	3.125×10^{-3}	70.6124×10^{-6}	1.42×10^{-3}	0.06284
2	50	230	4.347×10^{-3}	70.6124×10^{-6}	1.42×10^{-3}	0.087417
3	100	95	0.01052	70.6124×10^{-6}	1.42×10^{-3}	0.211556
4	150	50	0.02	70.6124×10^{-6}	1.42×10^{-3}	0.402197

VI. RESULTS AND DISCUSSIONS

- In this study, the composition of the material was adjusted in such a way that the dopant will take its place on site B of Perovskite structure ABO_3 .
- The X-Ray diffraction patterns of powdered BSCFTi were studied at different temperatures starting from 750 °C but diffraction peaks were found to be broad and very weak and sample was not crystallized perfectly. But, as the sintering temperature is increased, the impurity peaks due to Fe_3O_4Co . The sharp lines in XRD pattern depicts a well crystallized structure is formed. All the peaks indicate a Perovskite structure and correspond to planes (100), (110), (111), (200), (210), (211), (220), (300), (221), (310). There are some other peaks are also observed due to impurities and these were also disappeared as the

doping of Ti is increased and sintering is done for much time accordingly. The particle sizes of samples are calculated using XRD data.

- Densities of samples are calculated and for all samples the density percentage is above 90% .
- SEM images of BSCFTi proved the presence of porous spherical particles of nano size for the powders calcined above 850°C.
- From TGA plots it can be concluded that there is no weight loss after about 540°C which indicates that combustion process is completed at this temperature and oxide phases are formed.
- As the concentration of Ti increases the resistance of the sample has been decreased and conductivity increased.
- AC conductivities of all samples are calculated and observed that, as temperature is increased, resistance is decreased and conductivity is increased and the graph

between ac conductivity (along y-axis) and $1000/T$ along x-axis will be a linear graph.

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