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Structural and Electrical Properties of Nickel Oxide - Gadolinium Doped Ceria Composite Cermet Anode Materials for Low Temperature Solid Oxide Fuel Cells

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Abstract: In the present research work, for solid oxide fuel cell, nano composite anode materials were synthesized using nickel oxide and gadolinium doped ceria (GDC20) and. Anode materials prepared for low temperature operating solid oxide fuel cells, from metallic NiO and ceramic GDC20 powders. These are mechanically mixed as X (NiO) +1-X ($Ce_{0.8}Gd_{0.2}O_{2-\delta}$) where (X = 30, 40 ,45,50,55,60 wt. %). The mixed powders were ground and pelletized with respect to X. All the samples were characterized after sintered at the temperature 1300^{o}C. Systematically studied the structure, purity, phase and structural parameters of as-synthesized NiO-GDC20 anode samples were carried out by XRD and SEM. Electrical characterizations such as A.C Conductivities and D.C conductivities were carried out using impedance spectroscopy and four probe D.C conductivity measurements respectively. The activation energies of the anode samples were estimated from 300- 500 ^{o}C. The activation energies of A.C conductivities are observed that decrease with increase in frequencies.

Keywords: Solid Oxide Fuel Cell, Nano Composite Anode, D.C Conductivity, A.C Conductivity, Activation Energy.

I. INTRODUCTION

Solid oxide fuel cells (SOFC) are eco-friendly energy conversion system, which directly convert the chemical fuel to electrical energy via an electro chemical reaction [1] ,[2]. In recent years, researchers have great attention to develop the intermediate or low temperature (IT/LT) operated SOFCs. Therefore, reduction of operation temperature has the potential to reduce the cost of cell fabrication and also overcome the critical issues related to thermal aging and thermal expansion coefficient mismatch within the cell components at high temperatures.

The find a suitable kind of an anode material for SOFC is important to operate at Low or Intermediate temperatures(LT/IT) i.e. from 773^{o}K to 973^{o}K, which improve the performance, reliability and long operational life and reduce the cost of the system [2],[3]. The anode component performance is depending on its composition and the resulting microstructure and hence, it has great interest to investigate the proper composition of anode material [4]. The anode must have high electronic and ionic conductivity, good catalytic activity for fuel oxidation, proper porosity at the microstructure in reduced environment (during fed of hydrogen fuel at anode) at operating temperature [3]-[6]. Presently NiO-YSZ cermet anodes are using in the SOFC's, but they are operating at high temperature range 800^{O}C to 1000^{O}C [8]. Many of the investigators recommended that the GDC electrolyte is best candidate for low or intermediate operating temperatures than YSZ (Yttrium stabilized Zirconia).Hence NiO–GDC (or CeO2) composite is a best candidate for anode material and good compatible with GDC electrolytes under operating conditions. In NiO–GDC cermet anode, NiO acts as the best catalyst for oxygen activation and provides good electronic conductivity whereas GDC mainly act as a matrix to support the catalyst and avoid the Ni metal agglomeration under operating conditions and also they are an ideal match with GDC electrolyte at Operating temperatures [4]-[7]. The main requirement of anode is that, it should be electronically as well as ionically conducting. The cermet anode conductivity arises through two mechanisms that is ionic (through the GDC phase) and electronic (through the metal nickel phase)[9]- [12]. In the present study, synthesized the NiO - GDC20 ($Ce_{0.8}Gd_{0.2}O_{2-\delta}$) composites using mechanical mixing method with the variation of NiO. Here, fine Nano powders are used for homogeneous, crystalline and un-agglomerated multi component oxide ceramics for anode composite structure. Since the anode performance is dependent strongly on its composition, fabrication process and the resulting microstructure [8][19]

II. EXPERIMENTAL

In this research work, NiO nano powder was prepared by sol-gel citrate method. Commercially available Gadolinium doped Ceria powder $Ce_{0.8}Gd_{0.2}O_{2-\delta}$ (GDC20), (Cottor International, India) with a particle size of 200 nm was used for the processing of the specimens. To obtain fine nano powders, both NiO and GDC20 ($Ce_{0.8}Gd_{0.2}O_{2-\delta}$) powders were separately ball milled . In the process of ball milling , powder was mixed with 2wt% of methyl cellulose as a binder and de-ionized water as solvent(slurry) and it is allowed for ball billing using zirconia balls as grinding media for 12 hrs. Thus obtained slurry is dried in an oven and the resultant powder is granulated. For various compositions of cermet anode samples, NiO and GDC20 nano powders of crystallite size 30 nm,35nm respectively, were mixed as X (NiO) + (1-X) $Ce_{0.8}Gd_{0.2}O_{2-\delta}$ (where X = 30, 40,45,50,55 and 60 wt.%). As mixed powders were ground in agate mortar pestle for 3hrs with adding acetone and ethyl alcohol for homogeneous mixing. The homogenized anode composite powders were calcined at 750^{o}C for 4hrs and these powders were pressed into cylindrical pellets having 10mm-diameter and 1.5- 2.5 mm thickness using hydraulic press machine under pressure of 60Mpa. The pellets were sintered at 1300^{o}C for 4 hrs. in air with the heating rate of 5^{o}C/min from RT to 800^{0}C and from 800^{0}C to 1300^{o}C heating rate of 2^{o}C/min [13].

The crystal structure, lattice parameters and crystallite size of the anode samples were determined using X- ray diffraction (Phillips-3710 powder XRD) using CuKα1 radiation (λ=1.5406 A^{o}) over the range 20-80^{o} of 2θ with the scanning rate 5^{o}C/min. The relative densities of anode samples A1, A2, A3, A4,A5 and A6 are 90.1%, 92%, 93.4%, 94% ,93% and 92% respectively at sintering temperature 1300^{O}C, calculated using Archimedes method. The structure, phase and surface morphology of the samples were studied by using XRD and SEM (ZEISS) respectively [9].

The green pellets were coated with silver paste on its surface for electrical characterizations. Electrical properties of anode composites such as ac conductivity and dc conductivity were carried out using Impedance spectroscopy and dc four probe conductivity measurements from the probable operating temperature range 300^{O}C to 500^{O}c (LT) and subsequently estimated the activation energies .

The impedance spectroscopy is an ideal and well developed tool for studying the electrical properties and to separate out the bulk (grain), grain boundary and grain-electrode contributions to the total conductivity. Impedance measurement is used to compute the ac electrical conductivity for that ccomplex impedance data on anode composite samples were recorded using impedance analyzer in the temperature range of RT to 500^{O}C and the frequency range 1Hz To 5MHz.

D.C four probe conductivity instrument was used for accurate measurement of conductivity with respect to temperature. Anode compositions variation with respect X are coded (here in after called) as shown in Table (1).

TABLE 1 : Sample identification

S.No.	Composition X (NiO) + (1-X) $Ce_{0.8}Gd_{0.2}O_{2-\delta}$	Anode composites code
1	X=30 Wt%	A1
2	X=40 Wt%	A2
3	X=45 Wt%	A3
4	X=50 Wt%	A4
5	X=55 Wt%	A5
6	X=60 Wt%	A6

III. EXPERIMENTAL RESULTS AND DISCUSSION

A. *XRD Analysis*

XRD patterns of NiO –GDC20 composites are shown in Fig.1. These patterns are compared indexed accordingly with the standard JCPDS file no# 01-075-0162 for GDC20 and JCPDS file no.# 01-089-7130 for NiO. Obviously, XRD patterns reveals that all the composites (A1 to A6) has the multiphase , here GDC has fluorite (cubic) structure (Fm-3m group)and NiO has the cubic structure (Fm-3m group) [10],[14].

From the XRD data, lattice parameters of NiO and GDC20 phase were computed from the anode composites as shown in Fig.1 and observed that lattice parameters are independent of ‘x’ and are 4.178± 0.005A^{O} and 5.426 ±0.008A^{O} respectively for NiO and GDC20 [14].

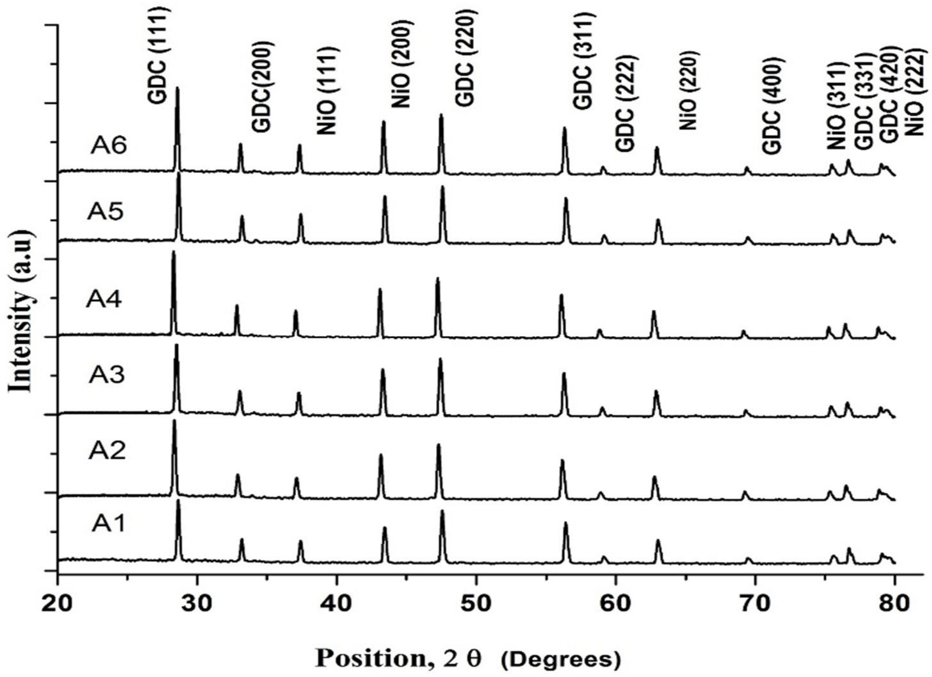

Fig.1. XRD Patterns of Anode Composites A1 to A6 Sintered at 1300^{O}C

As observed that the NiO content increasing in anode compositions, density of the samples increasing and decrease in lattice parameters has been observed in the sintered anode composites from 1100 ^{o}C to 1300^{o}C. Since Ni atoms are smaller than the Gadolinium (Gd) and Cerium (Ce), it occupies interstitial positions of crystal structure.

The avg. crystallite size of the anode composites were calculated by Scherrer's formula

$$\mathbf{D} = \frac{0.9\lambda}{\beta\, cos\theta} \qquad ----(1)$$

Where D is the crystallite size in nm, λ is the radiation wavelength Cu Kα = 0.15406nm,
θ is diffraction angle and β is the full width half maximum.

The average crystallite size of NiO and GDC20 phase in the anode composites were computed using equation (1) shown in Table 2.

TABLE 2: Crystallite Sizes of Anode Composites A1 to A6

Anode samples	Crystallite size (in nm)	
	NiO	GDC20
A1	40.85	47.42
A2	42.28	47.73
A3	46.07	49.86
A4	50.69	52.68
A5	44.51	48.40
A6	41.26	48.05

As observed that, crystallite sizes of NiO and GDC increased a few nm with the NiO content up to x = 50wt. % and slightly decreasing trend above x = 50wt. % at 1300^{O}C.

B. Electrical Properties

1) *DC conductivity:* The dc electrical conduction is a thermally activated process , dc electrical conductivities of all cermet anode composites sintered at 1300^{o}C were carried out using four probe dc- electrical conductivity measurement in air from the temperature range 300^{o}C - 500^{o}C (or 573 - 773^{O}K), which is the probable operating temperature range of the anode supported low temperature SOFC's.

The dc conductivities of all samples fit linearly into the Arrhenius relation. The variations of ln (σ_{dc}) vs. 1000/T i.e Arrhenius curves of anode composites A1, A2, A3, A4 , A5 and A6 are shown in Fig.2.

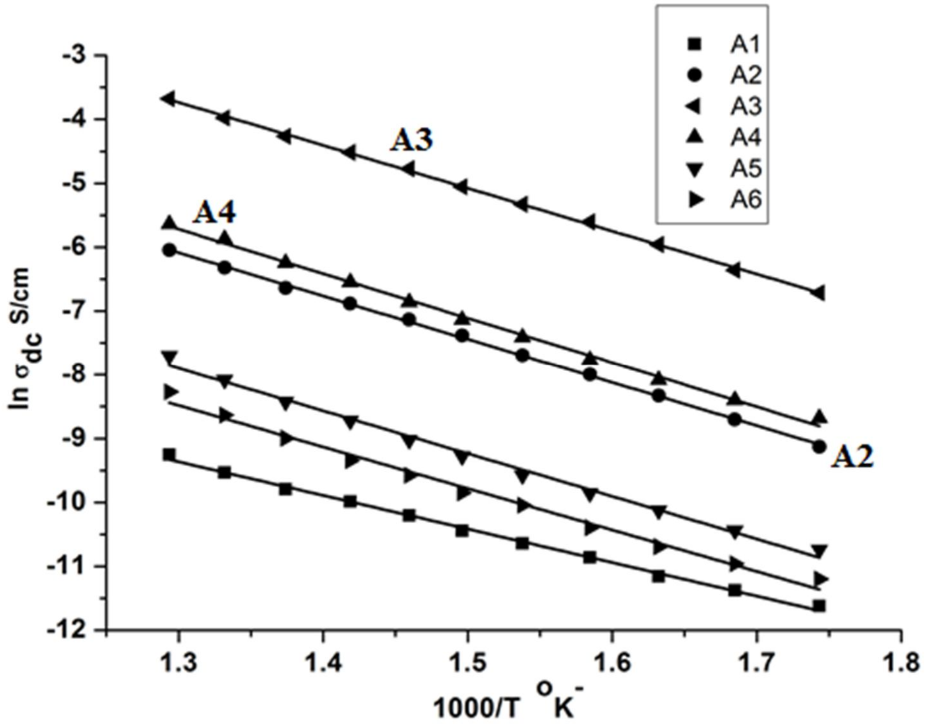

Fig. 2 : D.C Conductivity, Arrhenius plots of Anode Composites A1 to A6

The slope of the linear fit was calculated for the activation energy and the activation energies (E_a) of the anode composites are given by

$$\sigma_{dc} = \sigma_o \exp\left(\frac{-E_a}{K_B T}\right) \qquad (2)$$

Where σ_o is the pre-exponential factor, E_a is the activation energy, T is the absolute temperature and K is the Boltzmann constant. The dc conductivities of anode samples A1-A6 at the temperature 400 and 500°C and calculated activation energies from the temperature range 300-500°C are tabulated in the Table 3.

TABLE 3: DC Conductivities and Activation energies

Anode sample	DC conductivity (S/cm) at temperature		Activation energy(E_a) in ev
	400°C	**500°C**	
A1	2.92×10^{-5}	9.60×10^{-5}	0.93
A2	6.22×10^{-4}	2.38×10^{-3}	0.82
A3	7.54×10^{-3}	2.53×10^{-2}	0.74
A4	7.17×10^{-4}	3.57×10^{-3}	0.79
A5	6.14×10^{-5}	4.51×10^{-4}	0.84
A6	5.28×10^{-5}	2.56×10^{-4}	0.87

The maximum in conductivity or a minimum in activation energy of the anode samples are caused by the interaction of the substitutional rare earth ions and the oxygen vacancy or point defects[16]. Hence, the electrical conduction with respect to temperature in the cermet anodes is controlled by the charge-carrying defects. As observed conductivities of all the anode samples (A1 to A6), NiO concentrations below X= 45wt. %, the conductivity is predominantly ionic and above 45wt. %, it is predominantly electrical (typical of metals). Anode composites have low NiO content, NiO is distinctly distributed throughout the composite and

NiO-GDC20 connectivity is poor, as a result ionic conductivity is dominated over the electronic. But the activation energy less than 1eV implies that NiO also contributed to the electronic conductivity [15].

As the requirement of the anode for SOFC is that, it should be electronically as well as ionically conducting. The conductivity almost saturates above 45wt. % of NiO in the cermet's of NiO-GDC20, this shows the percolation limit [20] . Therefore, anode composite A3 attains maximum dc-conductivity of 2.53X10^{-2} S/cm at 500^{O}C. It is also expected that NiO-GDC20 contacts would increase leading conduction path along the interface between NiO and GDC, so that conductivities in the composites increased at x = 45wt. % and 50wt. %.

2) *A.C conductivity:* A.C conductivity of samples were measured by using impedance spectroscopy. The impedance spectroscopy is a non-destructive highly sophisticated experimental technique for the electrical characterization and microstructural properties of solid ionic materials or cermet's. The a.c conductivity is thermally activated and frequency dependent process and a.c conductivities of all the anode compositions were measured in the temperature range 300^{O}C to 500^{O}C and the frequencies from 1Hz to 5MHz. The ac conductivity arises as a result of charge hopping from one site to another site of the cermet's crystal and also due to the oxygen vacancies of anode composites. The material is sintered at high temperatures and may cause the oxygen to escape, but during the subsequent cooling process, the oxygen re-enters into the crystal, normally it may not completely compensate the oxygen loss. This may cause the oxygen vacancies apart from the vacancies created from the thermodynamic reasons. The hopping conduction in localized state gives rise to frequency dependent ac conductivity of ceramics and mixed conduction is because of hopping at the ionic migration through vacancies both oxygen as well as Ni vacancies.

The a.c electrical conductivity was obtained by the following relation

$$\sigma_{ac} = \frac{l}{SZ^1} \qquad (3)$$

Where l is the thickness, S is the surface area of the sample and Z^1 = real part of impedance.

A.C conductivities were obtained with respect to frequency and temperature and are shown in Table 4, conductivities at maximum temperature 500^{O}C at a few frequencies.

The thickness of the samples also plays the critical role in impeding the electrical performance of samples. At lower temperatures, the complexes formed are immobile ions and doesn't contribute much to the conductivity. As the temperature increases, impurity-defect complexes start to dissociate there by contributing to conductivity.

The total AC conductivities of all the samples have increasing trend with respect to frequency and obviously, observed that A3 has maximum conductivity among the samples i.e the maximum conductivity of A3 has at 500^{O}C is 7.84 X 10^{-2} S/cm and at the frequency 5MHz .

TABLE: 4 A.C conductivities of Anode Composites at different frequencies

Frequency	A.C conductivity S/cm at 500^{O}c					
	A1	A2	A3	A4	A5	A6
5 MHz	1.71 x 10^{-3}	2.70 x 10^{-3}	7.84 x 10^{-2}	6.54 x 10^{-3}	2.58 x 10^{-3}	2.30 x 10^{-3}
1.33MHz	1.43 x 10^{-3}	2.64 x 10^{-3}	7.53 x 10^{-2}	4.42 x 10^{-3}	2.36 x 10^{-3}	2.02 x 10^{-3}
485KHz	7.92 x 10^{-4}	1.86 x 10^{-3}	6.71x 10^{-2}	2.38 x 10^{-3}	1.71 x 10^{-3}	1.61 x 10^{-3}
1.1KHz	2.66 x 10^{-4}	8.10 x 10^{-4}	1.56 x 10^{-2}	9.20 x 10^{-4}	8.90 x 10^{-4}	8.60 x 10^{-4}
800Hz	2.62 x 10^{-4}	8.02 x 10^{-4}	1.20 x 10^{-2}	9.08 x 10^{-4}	8.29 x 10^{-4}	8.31 x 10^{-4}
1Hz	2.40 x 10^{-4}	7.03 x 10^{-4}	7.83 x 10^{-3}	7.49 x 10^{-4}	6.50 x 10^{-4}	6.32 x 10^{-4}

The total ac conductivity of the cermet anode is the sum of ionic and electronic conductivity. The conductivity increases with increasing NiO content up to 45wt% and after that shown decreasing trend up to 60wt% , above 45% of NiO in the anode composites metallic nature dominated and hence conductivity may decreasing trend at the temperature range 300 -500^{O}C . Therefore better candidate of anode material must have both ionic and electronic conductivity in compliance with that A3 has better composition candidate for anode material.

All the samples ac conductivities are linearly fit according to Arrhenius relation in the range of temperature 300 - 500^{O}C. The variation of real part of a.c conductivity(σ'_{ac}) with the $\frac{1000}{T}$ ^{0}K is shown in Fig.3, Fig.4 and Fig.5 at different frequencies. The nature of variation shows almost linear over wide range temperatures obeys the Arrhenius relation.

$$\sigma'_{ac} = \sigma_0 \exp\left(\frac{-E_a}{KT}\right) \qquad \text{-------------} \qquad (3)$$

Where E_a is the activation energy of conduction

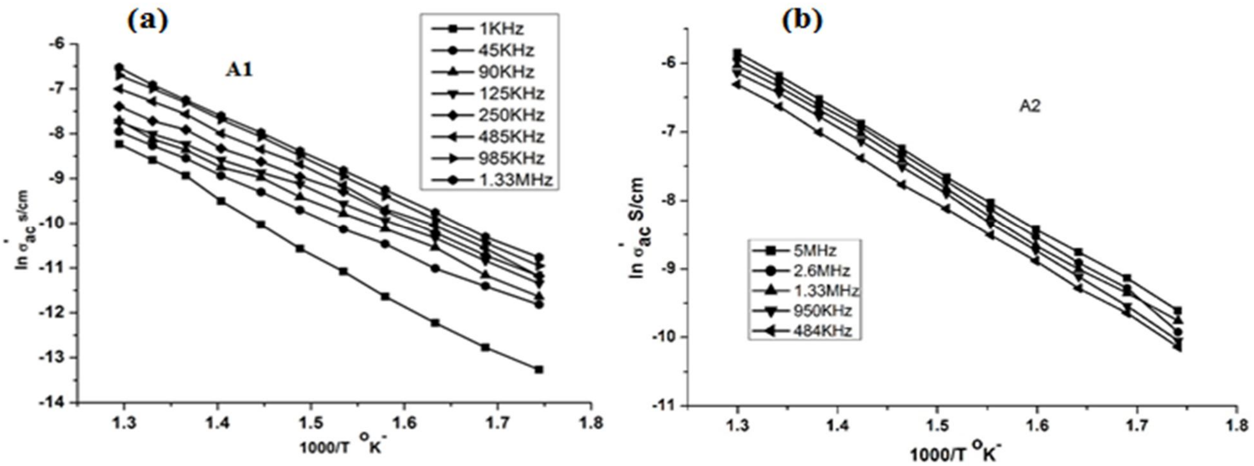

Fig. 3 : Arrhenius plots of anode composites at different frequencies a) A1 (X = 30 wt%) and b) A2 (X = 40 wt%) .

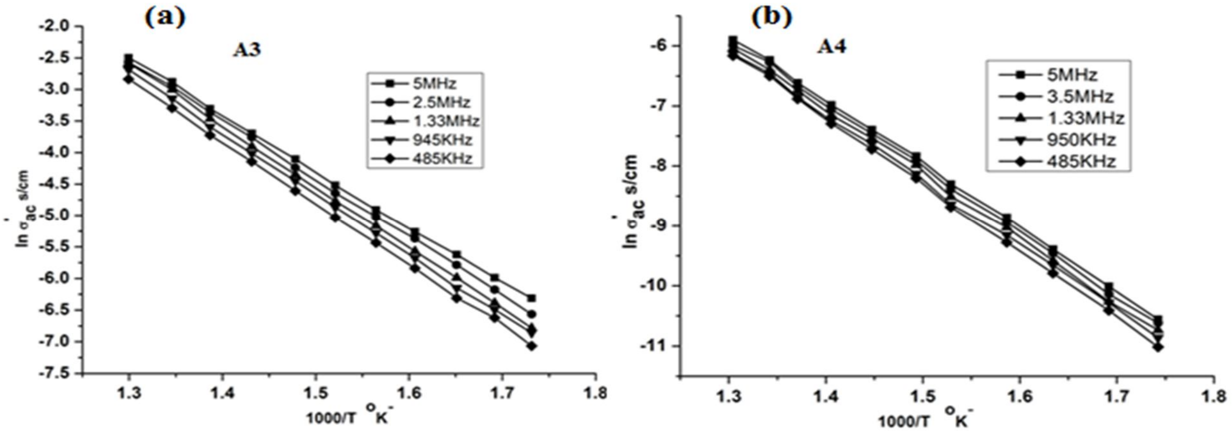

Fig.4: Arrhenius plots of A.C conductivity at different frequencies (a) A3 (x=45 wt.%) (b) A4 (x=50wt.%)

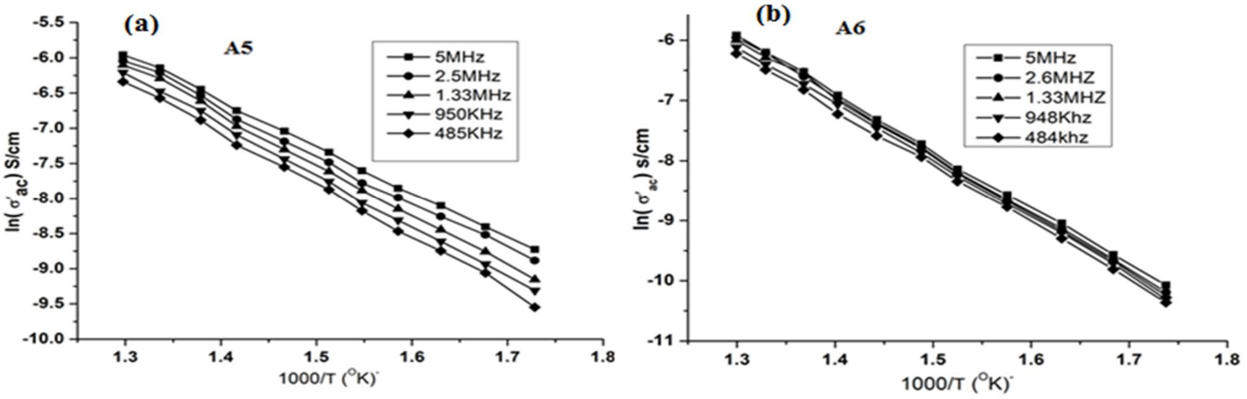

Fig.5: Arrhenius plots of A.C conductivity at different frequencies (a) A5 (x=55 wt.%) (b) A6 (x=60wt. %)

The slope of the ln (σ) Vs. 1000/T of all the samples were calculated for estimation of activation energies from the temperature range 300 to 500^{O}C and the frequency range 1Hz to 5MHz. The temperature above 300^{0}C may be the intrinsic region and the calculated activation energies are tabulated in table 5.

The activation energy for total conduction of samples comes from three sources that is the enthalpy of migration of oxygen ions (ΔH_m) ,the association enthalpy of complex defects(ΔH_a) and the activation energy for the grain boundary conduction . But in the low temperature range (< 400^{0}c) these three sources simultaneously limit the total conductivity.

TABLE: 5 Activation Energies of AC Conductivities

Frequency	Activation energies in eV					
	A1	A2	A3	A4	A5	A6
1 – 5 MHz	0.9±0.02	0.72±0.02	0.64±0.01	0.70±0.02	0.76±0.02	0.81±0.01
100- 950KHz	0.94±0.05	0.73±0.02	0.68±0.02	0.75±0.03	0.81±0.02	0.83±0.02
1 - 90KHz	1.1±0.06	0.80±0.03	0.73±0.03	0.84±0.04	0.84±0.01	0.85±0.02
100- 800Hz	1.15±0.05	0.85±0.03	0.78±0.02	0.91±0.02	0.91±0.04	0.93±0.038
1-100Hz	1.22±0.02	0.88±0.02	0.83±0.03	0.93±0.02	0.96±0.05	0.99±0.04

For low NiO content, NiO is distinctly distributed throughout the composite and NiO, GDC connectivity is poor. Hence ionic conductivity is dominated over the electronic, but the activation energy less than 1eV implies that NiO contributed to the electronic conductivity, for further NiO additions, NiO and GDC are homogeneously distributed thereby forming three dimensional connectivity and the activation energies are decreased with increase in frequency. The composite A3 has lower activation energy among the samples and it has 0.63 eV at 5MHz frequency.

The anode composites A3 and A4 have the better conductivity and activation energies among the samples .The conclusion of conductivity remains the optimum variation of NiO and hence the analysis of further research i.e at reducing environment under operating conditions of symmetric cell , which may be decisive of suitable anode composite for anode supported SOFC's .

IV. CONCLUSION

The XRD patterns reveals the purity of the anode composite and phase formation is cubic , crystallite sizes of NiO and GDC increased a few nm with the NiO content up to x = 50wt. % and slightly decreasing trend above x = 50wt. % .

The DC an AC conductivity measurements in the temperature range 300^{O}C-500^{O}C were given that, the better candidate of anode composites are A3 and A4. The maximum DC conductivity obtained cermet composition among all the anode composites is A3 which has 2.53 X 10^{-2} S/cm at 500^{O}C and its activation energy is 0.74 eV . Similarly, AC conductivity measurements also given that the maximum ac conductivity composition is A3 at the frequency range 1Hz to 5MHz. The maximum ac conductivity is 7.84 X 10^{-2} S/cm at the frequency 5MHz and temperature at 500 ^{o}C. The activation energies of anode A3 and A4 are 0.63 and 0.68 eV at 5MHz. The conclusion of conductivity remains the optimum with the variation of NiO and hence the analysis of further characterizations i.e symmetric cell characterization of anode composites and working at reducing environment under operating conditions. which may be decisive of suitable anode composite i.e A3 or A4 for anode supported SOFC's.

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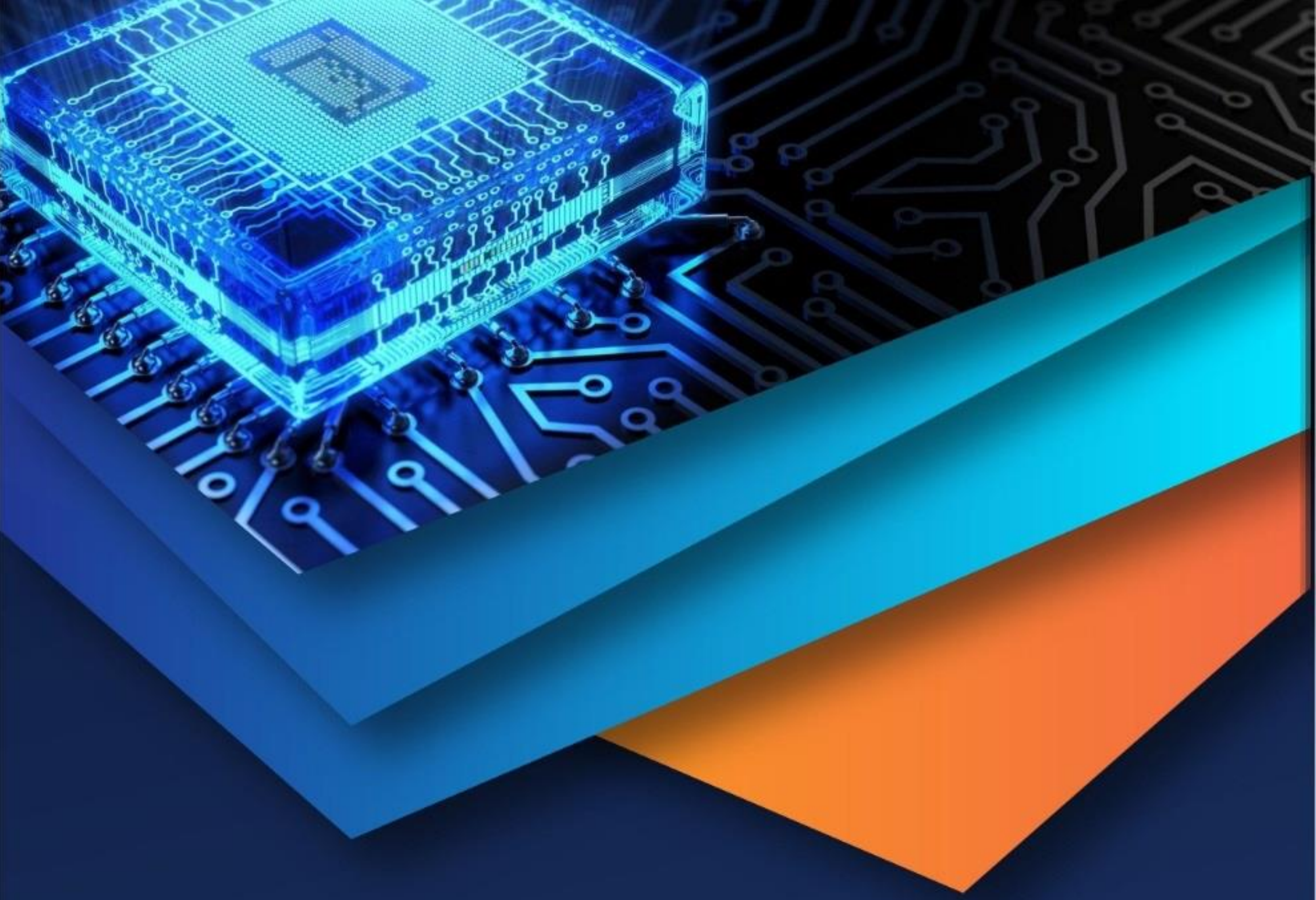

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