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Electrical Parameters of $(SnO_2)_{1-x}(ZnO)_x$ nanocomposites prepared by solvothermal method

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Abstract

$(SnO_2)_{1-x}(ZnO)_x$ (with x values 0.0,0.2,0.4,0.5,0.6,0.8,1.0) nanocomposite was prepared by a simple microwave assisted solvothermal method using ethylene glycol as solvent. The as-prepared samples are calcinated at 500^0C. They were characterized using powder X-ray diffraction (PXRD) and the AC electrical parameter was studied with in the temperature range of 40-150^0C. Both the dielectric loss factor and dielectric constant decreases with increase in frequency and increases with increase in temperature. It is due to the occurrence of space charge polarization. Increase in AC electrical conductivity with increase in frequency and temperature was noted and it is due to hopping of electrons. The results are reported here with.

Keywords : *dielectric constant, dielectric loss factor, microwave, nanocomposite, solvothermal.*

I. INTRODUCTION

Tin oxide and Zinc oxide are mostly n- type semiconducting material with interesting features. These materials has a wide band gap of 3.6-3.8 eV and 3.37 eV [1,2] respectively. The preparation techniques for SnO_2, ZnO nanoparticles and their nanocomposites include sol-gel method [3,4,5], solvothermal method [6,7], spray pyrolysis [8,9], sonochemical [10], hydrothermal [11,12,13], microwave assisted method [14,15] and co-precipitation method [16]. SnO_2 and ZnO nanoparticles and their nanocomposites has been widely used in a variety of applications such as sensor [17,18,19], in lithium ion batteries [20], catalyst [21], solar cells [22,23,24], antibacterial agent [25] and photovoltaic applications [26].

Composite is a combination of two or more different materials that are mixed together to blend the best properties of both. The morphology and interfacial characteristics influence the properties of nanocomposites apart from the properties of parent material [27]. In the present work we have synthesized $(SnO_2)_{1-x}(ZnO)_x$ (with x values 0.0,0.2,0.4,0.5,0.6,0.8,1.0) nanocomposites via a simple microwave assisted solvothermal method with ethylene glycol as solvent. The structure and electrical parameters such as dielectric constant, dielectric loss factor and the AC electrical conductivities were analyzed and the results were discussed.

II. EXPERIMENT

A. METHODOLOGY

$(SnO_2)_{1-x}(ZnO)_x$ (with x values 0,0.2,0.4,0.5,0.6,0.8,1) nanocomposites were prepared using the precursors stannous chloride dehydrate, zinc acetate dehydrate, urea and ethylene glycol. All the chemicals used were of analytical grade. SnO_2 (when x= 0) nanoparticles were synthesized by taking stannous chloride and urea in 1:3 molecular ratio. The precursors are rehabilitated as solution by dissolving them separately in 200 ml ethylene glycol solution using a magnetic stirrer. Then the urea solution was added drop-wise into the stannous chloride solution kept under stirring. The final solution was kept in a domestic microwave oven (operated with a frequency of 2.5 GHz and power 800 W). Microwave irradiation was carried out until the solvent gets evapourated and a colloidal precipitate was formed. The colloidal precipitate was washed with double distilled water for about five times and then with acetone to remove the organic impurities present if any. The washed samples were dried in atmospheric air and then preserved as yield. Similarly ZnO (when x= 1) nanoparticles were prepared by taking zinc acetate and urea. The composites of $(SnO_2)_{1-x}(ZnO)_x$ with x = 0.2,0.4,0.5,0.6,0.8 were prepared by taking stannous chloride, zinc acetate and urea. The molecular ratio and the method of preparation were same as that used for SnO_2 nanoparticle preparation. A total of seven samples were prepared and calcinated at a temperature of 500^0C.

B. CHARACTERIZATION

The annealed samples were characterized by powder X-ray diffractometer (XPERT-PRO) using Cu Kα (λ=1.54060Å) radiation. The powdered samples were pelletized using a hydraulic press. Graphite coating was given on the flat surfaces of the pellets for good conductivity. The electrical parameters of these pellets were analyzed using Agilant LCR meter in the temperature range of 40-150^{0}C. All the measurements were done while cooling.

III. RESULT AND DISCUSSION

A. XRD ANALYSIS

The powder XRD pattern of $(SnO_2)_{1-x}(ZnO)_x$ (with x values 0.0,0.2,0.4,0.5,0.6,0.8,1.0) nanocomposite annealed at 500^{0}C are shown in figure 1. The peaks obtained in the pattern SnO_2 and ZnO matches well with tetragonal phase of Tin Oxide with lattice constants a = 4.746 and c = 3.1928 (JCPDS Card NO: 88-0287) and Hexagonal phase of Zinc Oxide with lattice constants a = 3.2525 and c = 5.2098 (JCPDS Card NO: 89-1397). The peaks in the pattern contain both tin oxide and zinc oxide phase. Occurrence of Zn_2SnO_4 and $ZnSnO_3$ phases in ZnO-SnO_2 system was reported by Choi [28]. In our work additional phases such as SnO, Sn_2O_3, Sn_3O_4, ZnO_2, Zn_2SnO_4, $ZnSnO_3$ were absent. Absence of additional phases such as SnO, Zn_2SnO_4, $ZnSnO_3$ was reported by Omar et al [5].The average grain size was calculated using Debye-Scherer formula [29,30].

$$D = K\lambda / \beta\cos\theta$$

Where D is the mean crystallite size, K is the size factor (0.9), λ is the wavelength of incident beam, β is the full width half maximum (in radian) and θ is the Bragg reflection angle. It was observed that the intensity of peaks corresponding to the x values (0.4,0.5,0.6)decreases when compared to end members. This was mainly due to the increase in the concentration of tin and zinc in the formation of nanocomposites. Similar decrease in the intensity of peaks was observed on the addition of Sn into ZnO nanopowders by Prasopporn et al [16].The average crystallite size was found as 7-31nm.

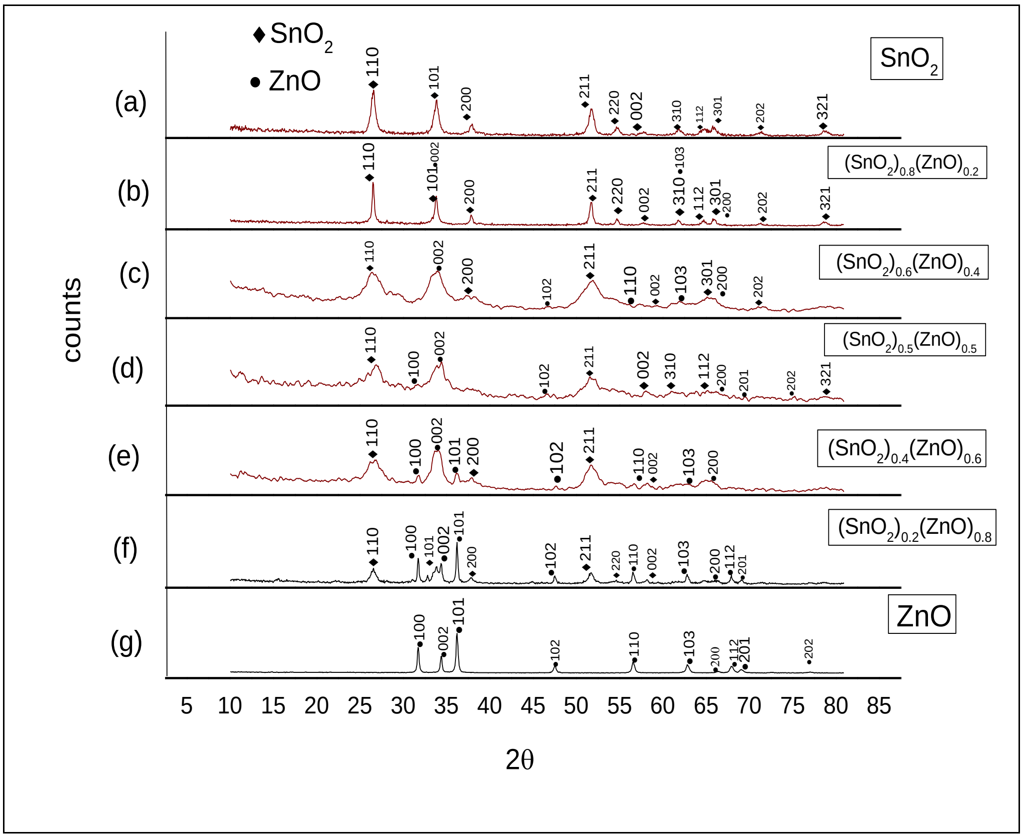


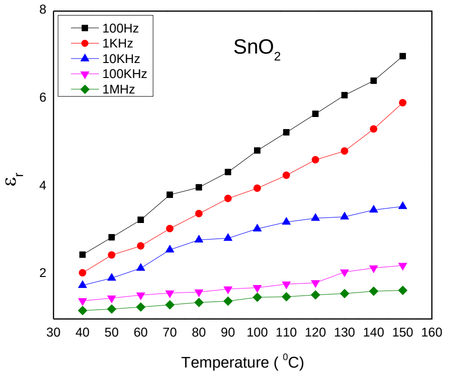
Figure: 1 The PXRD pattern of $(SnO_2)_{1-x}(ZnO)_x$ nanocomposites

B. ELECTRICAL PARAMETERS

The behaviour of electrical parameters such as dielectric constant, dielectric loss factor and the AC electrical conductivity of all the samples with respect to temperature and frequency were shown in figure 2-22. The dielectric constants was calculated using the relation $\varepsilon_r = C_c/C_a$. The AC electrical conductivity (σ_{ac}) was calculated using the formula,

$$\sigma_{ac} = \varepsilon_0 \varepsilon_r \omega \tan\delta.$$

Where ε_0 is the permittivity of free space (8.85 x 10^{-12} $C^2N^{-1}m^{-2}$) and ω is the angular frequency (ω = 2πf, where f is the frequency). The variation of dielectric constant with frequency and temperature were shown in figure 2-8. It was observed that at low temperature the value of dielectric constant for the end members was less when compared to composites, except for the composite with x value 0.4. At higher temperatures and low frequencies the dielectric constant value was high for the end members than that for the composites.



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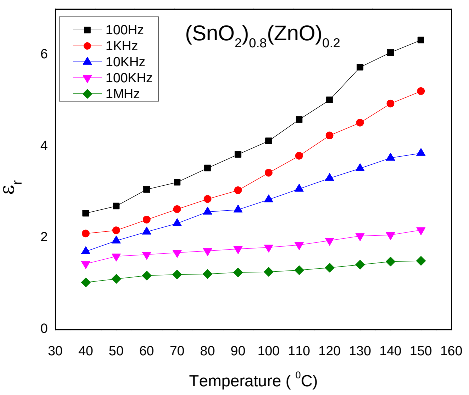

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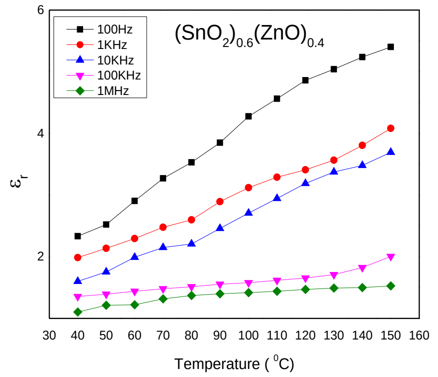

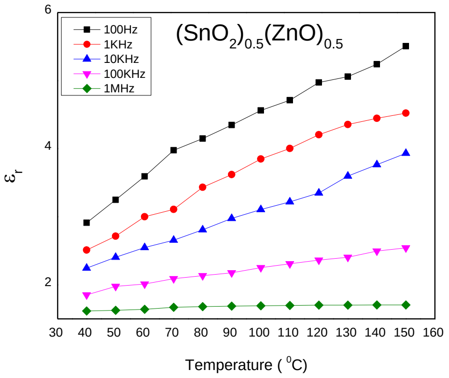

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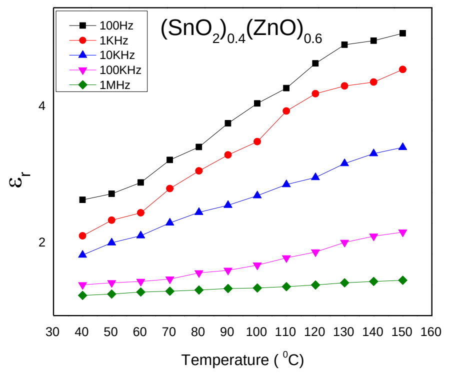

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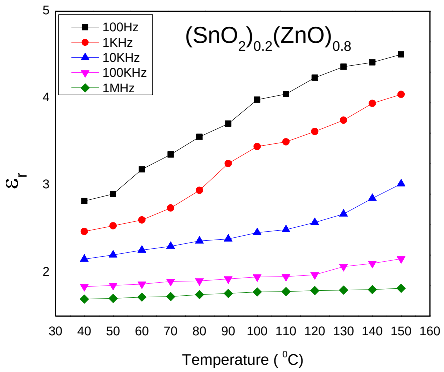

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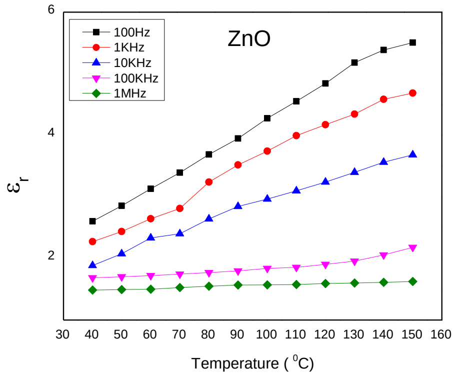
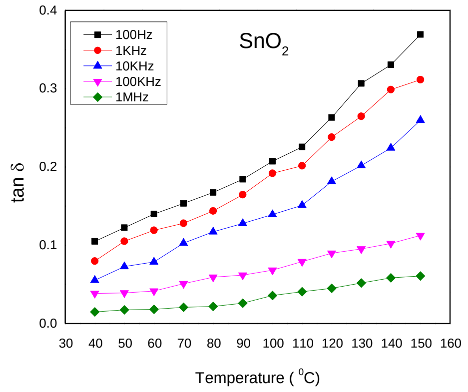


Figure: 8

Figure: 2 – 8 shows the variation of dielectric constant with frequency and temperature for $(SnO_2)_{1-x}(ZnO)_x$ nanocomposites.

The variation of dielectric loss with frequency and temperature are shown in figure 9-15. A low value of dielectric loss factor was observed for the end members than that for the composites except the composite with x value 0.2. It was observed that the dielectric constant, dielectric loss factor increase with increase in temperature and decreases with increase in frequency. The charge transport relaxation time was responsible for the variation of dielectric constant with applied field [31]. In heterogeneous structures, the space charge polarization was predominant and it results in the decrease of dielectric constant with increase in frequency [32].



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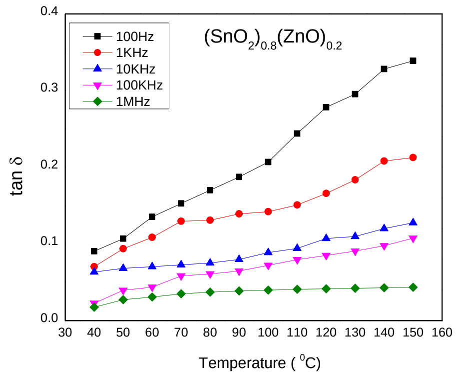

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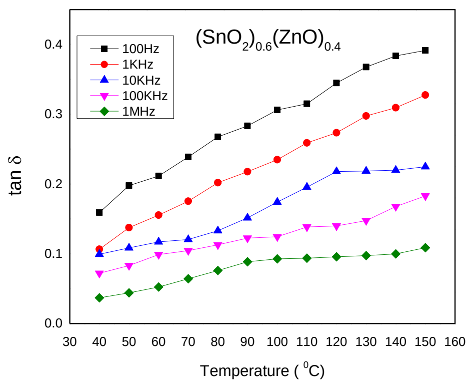

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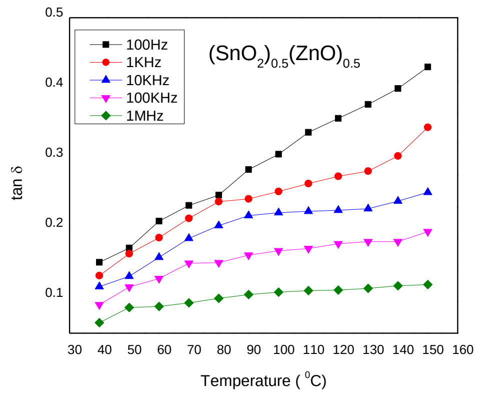

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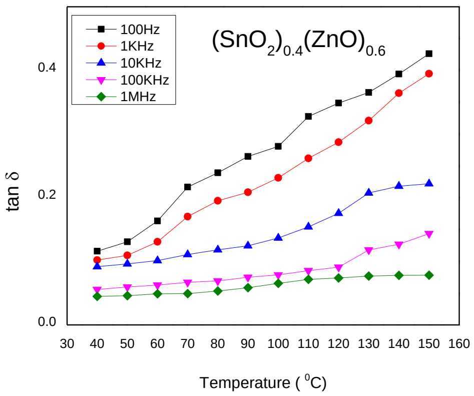

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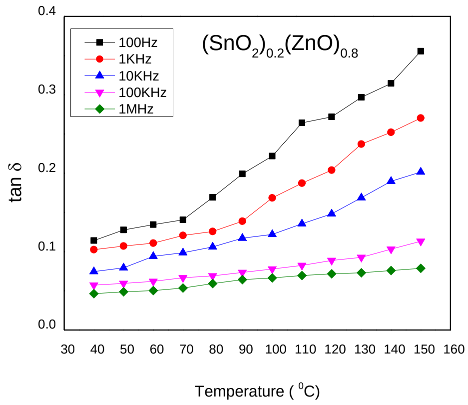

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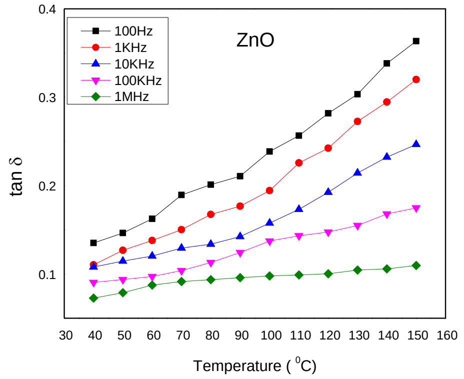
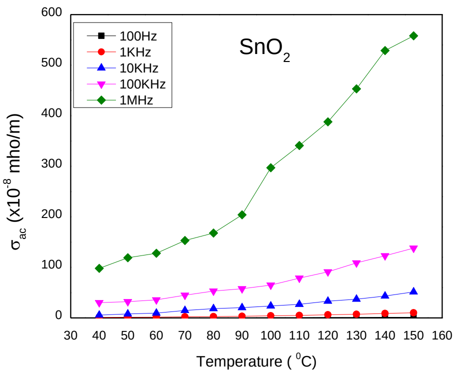


Figure: 15

Figure: 9 – 15 shows the variation of dielectric loss factor with frequency and temperature for $(SnO_2)_{1-x}(ZnO)_x$ nanocomposites.

The variation of AC electrical conductivity with frequency and temperature are shown in figure 16-22. At low frequencies the value of AC electrical conductivity for the end members was found to be less than that for the composites, except for the composite with x value 0.2.



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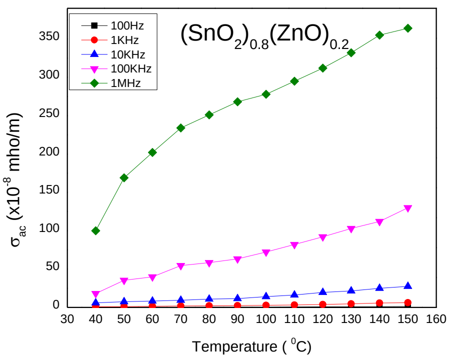

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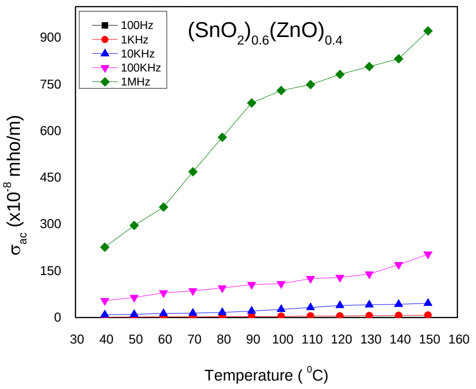

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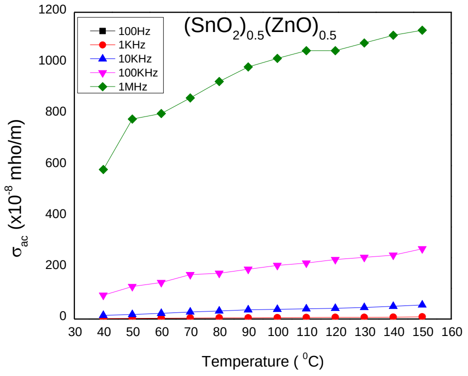

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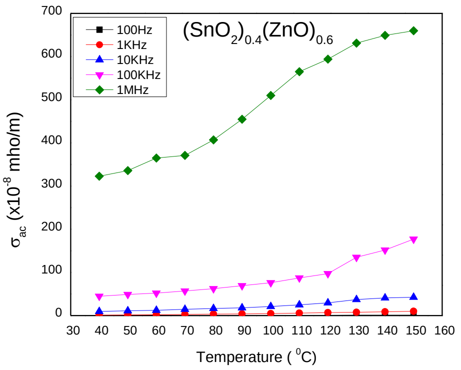

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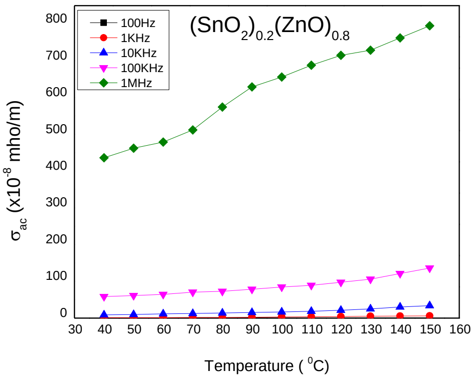

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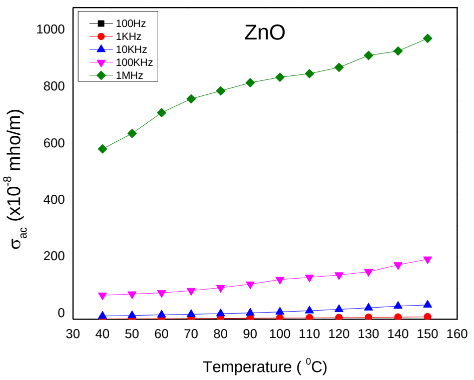

Figure: 22

Figure: 16 – 22 shows the variation of AC electrical conductivity with frequency and temperature for $(SnO_2)_{1-x}(ZnO)_x$ nanocomposites.

Occurrence of defects such as vacancy clusters, microporosites and dangling bonds causes change of positive and negative space charge distributions at the interfaces. Dipole moments are produced when these charge distributions are trapped at defect sites. This causes the occurrence of space charge polarization [33]. At lower temperatures dipoles remain fixed and at higher temperatures they are free to move and hence it can respond to the applied electric field. Thus an increase in dielectric constant and dielectric loss factor was observed with increase in temperature. The electronic polarization occurs due to hopping of electrons. The hopping is mainly due to the mobility of charge carries and it results in increase in the AC electrical conductivity with increase in temperature [34].

IV. CONCLUSION

$(SnO_2)_{1-x}(ZnO)_x$ (with x values 0,0.2,0.4,0.5,0.6,0.8,1) nanocomposite were successfully prepared by a simple microwave assisted solvothermal method using ethylene glycol as solvent. The average particle size was found as 7-31nm. It was observed that the dielectric loss factor, dielectric constant decreases with increase in frequency and increases with increase in temperature. The decrease in dielectric loss factor and dielectric constant with increase in frequency was due to the occurrence of space charge polarization. Increase in AC electrical conductivity with increase in frequency and temperature was noted and it was due to hopping of electrons.

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