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Structural and Conductivity studies of BFN-ST40 Nanoceramics Processed by Ball milling

Ritesh Kumar[1], Amarendra Narayan[2], N.K.Singh[3]
[1]Research Scholar, University Dept. of Physics, V KS University, Ara-802301, Bihar, India
[2]Dept. of Physics, Patna University, Patna, Bihar, India
[3]University Dept. of Physics, V K S University, Ara-802301, Bihar, India

***Abstract:** 0.60 Ba($Fe_{0.5}Nb_{0.5}$)O_3 -0.40 $SrTiO_3$ nanoceramics sample was prepared by ball milling induced solid-state reaction method after annealing at high temperature 1200°C.The phase purity and crystallinity have been studied by using the X-ray diffraction (XRD) technique. The micro structural analysis has been carried out by field emission scanning electron microscopy (FESEM). Conductivity studies have done on the basis of experimental data obtained by impedance analyzer. Variation in Activation energies with the increase in frequency exhibits that at lower frequency the long range hopping involves to overcome the high barrier width while the lower activation energy at high frequency involves the short range or localized hopping. Jump relaxation model (JRM) and Universal dielectric response (UDR) law have been used to explain conductivity behavior of present sample.*

***Key terms:** Nanoceramics, Ball milling, XRD, FESEM, Impedance analyzer, Jump relaxation model ,UDR law*

I. INTRODUCTION

Polycrystalline nanoceramics, which have high dielectric constant (HDC) are of enormous importance to the electronic industry due to their wide applications as memory devices, multilayer capacitors, power transmission devices, sensors, high energy storage devices, etc [1,2]. Many researchers studied following lead based HDC materials as Pb($Fe_{1/2}Nb_{1/2}$)O_3 [3,4], Pb($Fe_{1/2}Ta_{1/2}$)O_3 [5], Pb($Mg_{1/3}Nb_{2/3}$)O_3 [6], Pb($Mg_{1/3}Nb_{2/3}$)O_3–$PbTiO_3$ [7–9], 0.94Pb($Fe_{1/2}Nb_{1/2}$)O_3–0.06$PbTiO_3$ [10] etc. It has remarkable truth that lead causes environmental pollution due to its toxic nature. Therefore, it is necessary to make constant attempt to search alternative lead free compounds which should have either comparable or superior dielectric properties. Researchers constantly tried to improve the dielectric nature of material by way of controlling grain growth and designing microstructure inhomogeneity [11]. Many research findings have suggested the BaFe0.5Nb0.5O3 based systems can be used as HDC materials, However, dielectric loss(~1) of $BaFe_{0.5}Nb_{0.5}O_3$ ceramic is high at room temperature [12,13]. Electric properties of BFN system can be improved by doping or by forming solid solutions with other systems. In order to decrease dielectric loss some dopant or solid solution were added into $BaFe_{0.5}Nb_{0.5}O_3$ ceramic Eitssayeam et al. [14] synthesized the $Ba_{0.9}Sr_{0.1}[Ti_{1-x}(Fe_{0.5}Nb_{0.5})_x]O_3$; x=0 to 0.50 ceramics and studied their dielectric properties. Singh et al. [15] reported $BaTiO_3$ and $SrTiO_3$ doped $BaFe_{0.5}Nb_{0.5}O_3$ ceramics and found diffuse type of phase transition. In this paper we have studied 0.60 Ba($Fe_{0.5}Nb_{0.5}$)O_3 -0.40 $SrTiO_3$ nanoceramic sample was prepared by solid-state reaction method through Ball milling.

II. EXPERIMENTAL PROCEDURES

Complex perovskite oxides 0.60Ba($Fe_{0.5}Nb_{0.5}$)O_3-0.40$SrTiO_3$ hereafter abbreviation as BFN-ST40 was prepared by a mixed oxide preparation route. $BaCO_3$(Merck,99.101%),$SrCO_3$(Lobachemie 98%),Fe_2O_3(Merck ≥90%),TiO_2(Merck ≥98.5%) and Nb_2O_5(Lobachemie,99.9%). were used for the preparation of BFN-ST40 ceramics. These chemicals were taken in stoichiometric ratio, and mixed in the presence of air for 2 hrs. and in acetone for 6 h using mortar and pastle. The finely mixed powder of BFN-ST40 was calcined at 1200°C for 8 h. The calcined powder of above mentioned ceramics were regrinded through 250 rpm for 20 Hrs using Retsch Ball Mill in ethanol medium to obtain nano-ceramics. The structure and quality of the obtained powder was checked with The X-ray powder diffraction pattern of the sample is taken at room temperature using a X-ray powder diffractometer using Cukα radiation (λ = 1.5418 Å) in a wide range of Bragg angles 2θ (20° ≤ 2θ ≤ 80°) with scanning rate 2°/min.Field emission scanning electron microscopy (FESEM) has used to study morphological feature of the sample. Powder is used to make pellet of diameter ~10 mm and thickness 1.2 mm using polyvinyl alcohol as binder. The pellets were sintered at 1250°C for 5 h and then brought to room temperature under controlled cooling. Dielectric behaviour of sample have investigated by using Impedance analyzerin the temperature range from 40°C to 400°C and in the frequency range from 1 KHz to 1 MHz .

III.STRUCTURAL ANALYSIS

Figure.1 shows the XRD patterns of the $0.60Ba(Fe_{0.5}Nb_{0.5})O_3$-$0.40SrTiO_3$ i.e. BFN-ST40 nanoceramics powder conventionally sintered at 1200°C for 8 hrs. Sharp and distinct XRD peaks were obtained for the given sample, which suggest the good crystalline and homogenous nature of the BFN-ST40 nanoceramic samples.

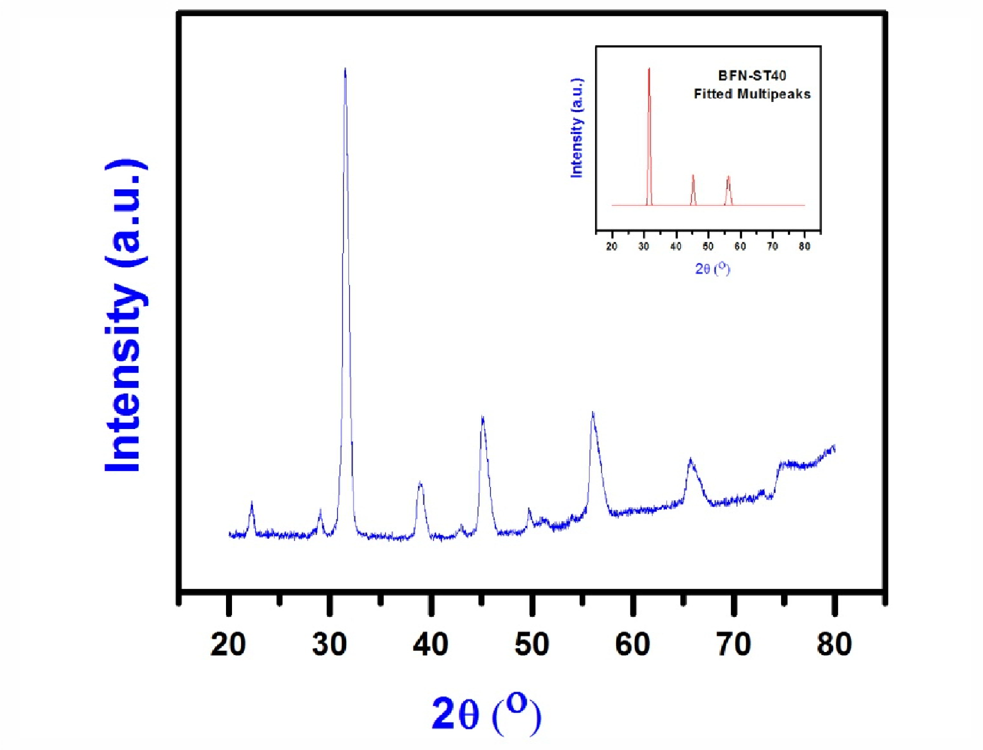

Figure .1 XRD pattern of 0.60 $Ba(Fe_{0.5}Nb_{0.5})O_3$ -0.40 $SrTiO_3$Nanoceramic system

III. MORPHOLOGICAL ANALYSIS

Figure.2 shows the FESEM micrographs of the sintered BFN-ST40 nanoceramics. The sintered BFN-ST40 nanoceramics show dense and almost non-uniform microstructures. There is good crystallinity can be confirm with help of FESEM image present here.

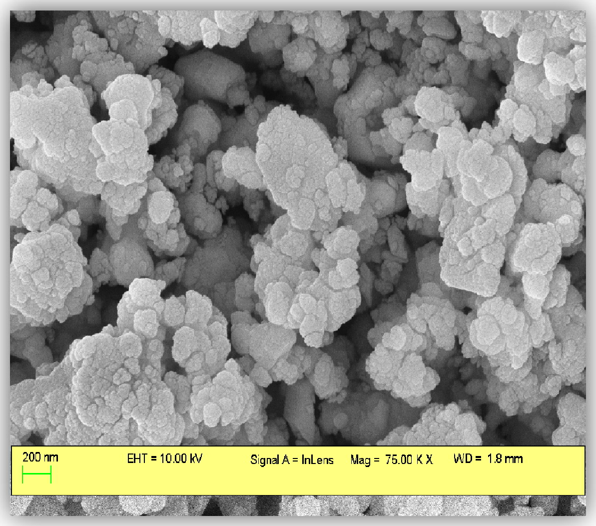

Figure.2 FESEM image of 0.60 $Ba(Fe_{0.5}Nb_{0.5})O_3$ -0.40 $SrTiO_3$Nanoceramic system

IV. CONDUCTIVITY STUDIES

Figure-3 shows frequency – dependent ac conductivity data at different temperatures in the BFN-ST40 nanoceramics sample. At low temperature, ac conductivity of present sample increases sharply with increase in frequency. This implies the presence of dispersion in conductivity with respect to frequency. At higher temperatures all the curves for different frequencies show a tendency to merge down, this shows that the dispersion of AC conductivity has narrowed down. Conductivity of the present sample increases with increase in frequency at higher temperature.

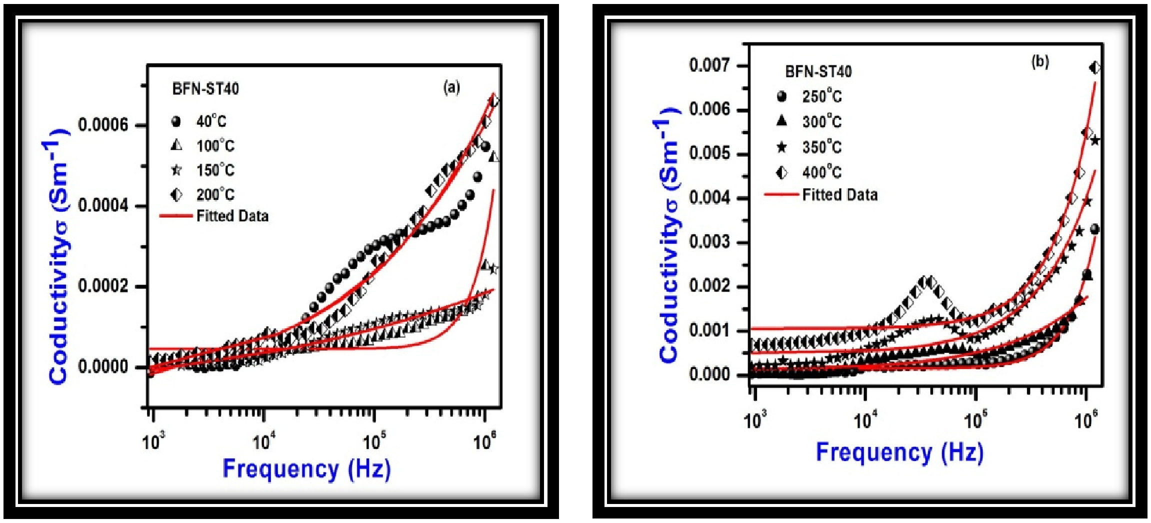

Figure 3 (a) and (b) shows Frequency dependence behavior of Conductivity at different temperatures.

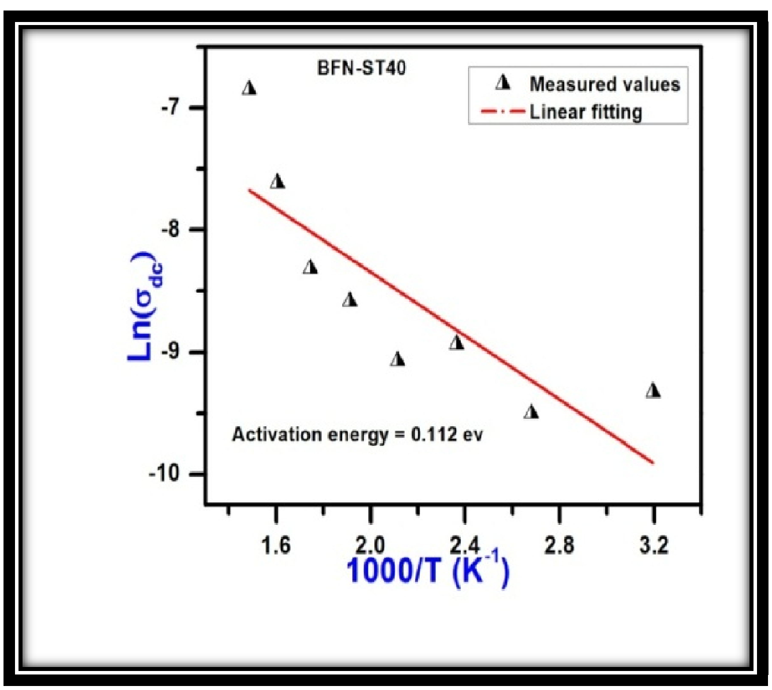

Figure 4.shows the activation energy calculated from linear fit of frequency dependent AC conductivity (lnσ) variation with reciprocal temperature (1000/T) for BFN-ST4 nanoceramic.

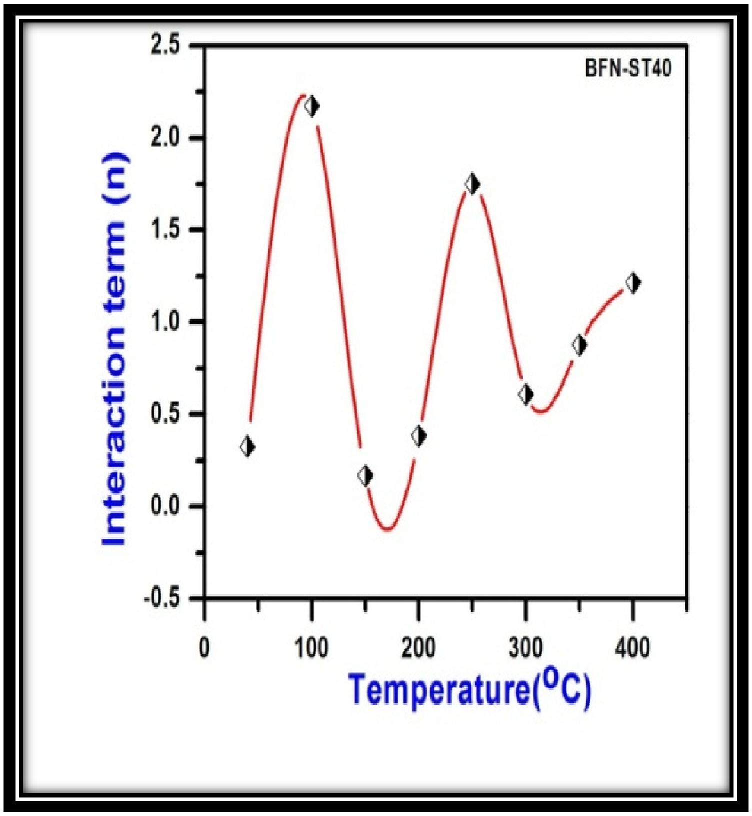

Figure 5.shows the interaction term calculated from linear fit of frequency dependent AC conductivity (lnσ) variation with reciprocal temperature (1000/T) for BFN-ST40 nanoceramics.

Table-1 indicates that the conductivity of the BFN-ST40 nanoceramics sample increases with increase in temperature. As applicable in various materials, Jump relaxation model (JRM) can account for this conductivity behavior with increase in temperature. DC conductivity is independent of frequency which can be attributed to long range movement of charge carriers [16]. Universal dielectric response (UDR) law can be a possible cause in this case of ac conductivity variation with frequency [17].

The increase in ac conductivity with increasing temperature may be considered as a charge compensation process occurring due to oxygen vacancies created by the loss of oxygen (usually during sintering) which follows Kröger-Vink equation [18].The activation energy of BFN-ST40 nanoceramics sample has found to 0.112 eV. Long range hopping of oxygen ion between neighboring sites can be a possible cause of this type of relaxation [19].

Table 1.Different parameters calculated from Universal dielectric response (UDR) law

Temperature (^{o}C)	σ_{dc}	A	n
40	-8.91211E-5	7.79758E-6	0.32472
100	4.60451E-5	2.37137E-17	2.17439
150	-9.04653E-5	2.65461E-5	0.16934
200	-4.55431E-5	3.35053E-6	0.38421
250	1.52648E-4	6.70244E-14	1.751
300	1.09369E-4	3.59519E-7	0.61031
350	4.92221E-4	1.92546E-8	0.87699
400	0.00106	2.23209E-10	1.21644

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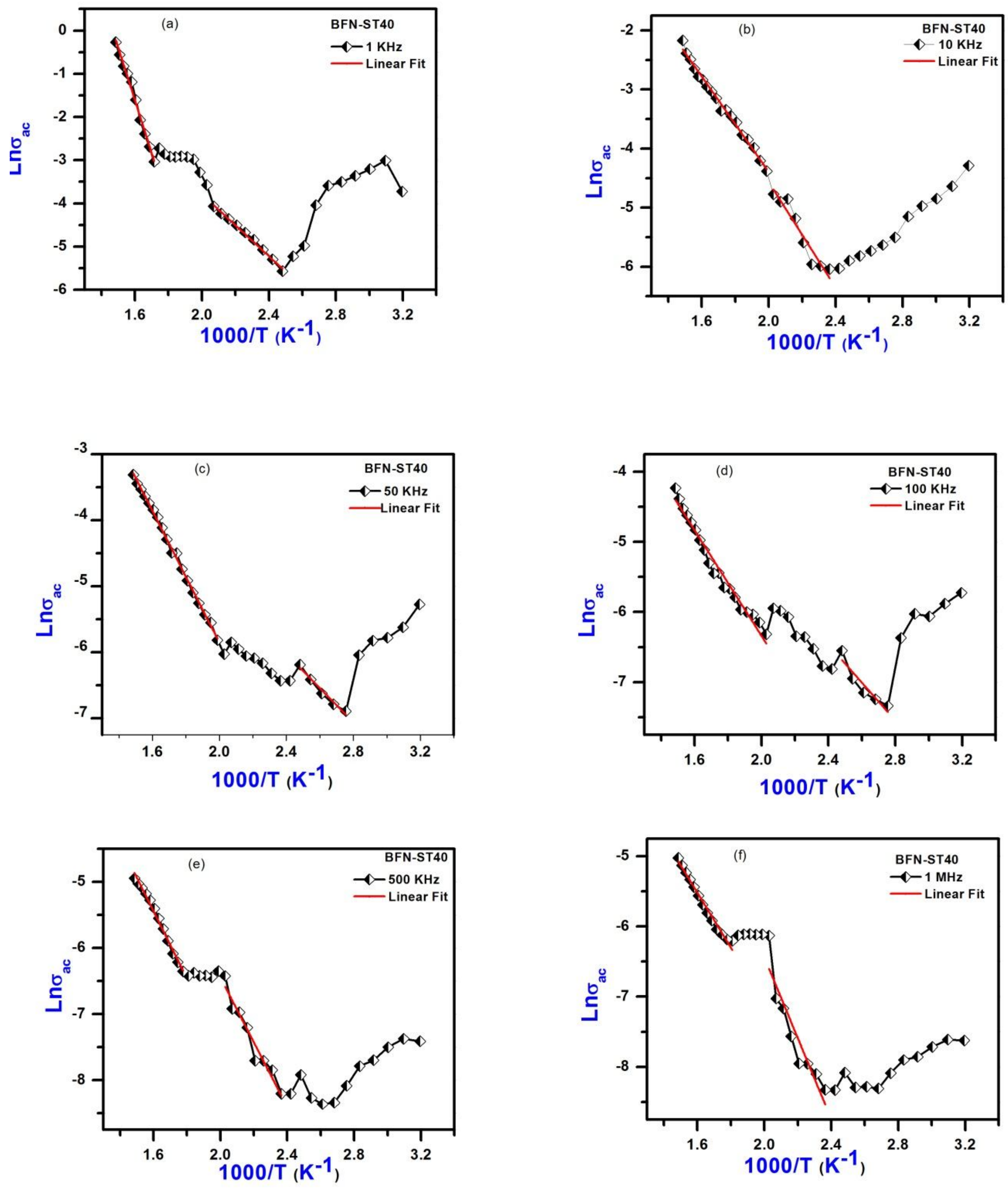

Figure 5 . AC conductivity ln(σ_{ac}) variation with reciprocal of temperature (1000/T) at different frequencies for BFN-ST40 nanoceramics

The temperature dependence of σ_{ac}(T), at different frequencies for the BFN-ST40 nanoceramics was measured in the temperature range of 40-400 ^{o}C. The results obtained are shown in Figure 5, in whichln (σ_{ac}) is plotted as a function of (1000/T). It is clear from the figure that σ_{ac}(T) for the BFN-ST40 nanoceramics sample shows a frequency and temperature dependence over the whole range of frequencies from 1 KHz to 1 MHz. Figure 6, describes the frequency variation of activation energy for different temperature regions.

Table 2.Activation energy values for BFN-ST40 nanoceramics.

Frequency (KHz)	E_a(eV) for T_H region	E_a(eV) for T_L region
1	1.053	0.30
10	0.65	0.38
50	0.43	0.22
100	0.32	0.23
500	0.43	0.42
1000	0.33	0.49

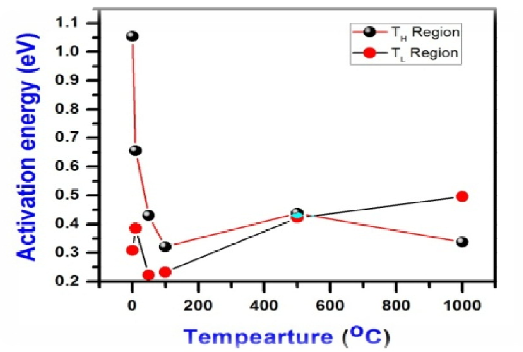

Figure 6.Frequency dependent variation in Activation energy for different conduction regions

Table-2 shows that in High temperature region (T_H) the activation energy lies in the range of 1.05 to 0.33 eV, whereas in Low temperature region (T_L), the activation energy lies in the range of 0.30 to 0.49 eV. Variation in Activation energies with the increase in frequency exhibits that at lower frequency the long range hopping involves to overcome the high barrier width while the lower activation energy at high frequency involves the short range or localized hopping.

V. CONCLUSION

Polycrystalline BFN-ST40 nanoceramic has been successfully synthesized by Ball milling induced solid sate reaction technique. The present sample shows the good crystalline and homogenous nature. At low temperature, ac conductivity of BFN-ST40 sample increases sharply with increase in frequency. This implies the presence of dispersion in conductivity with respect to frequency. Conductivity of the present sample increases with increase in frequency at higher temperature. Universal dielectric response (UDR) law can be a possible cause in this case of ac conductivity variation with frequency. Variation in Activation energies with the increase in frequency exhibits that at lower frequency the long range hopping involves to overcome the high barrier width while the lower activation energy at high frequency involves the short range or localized hopping.

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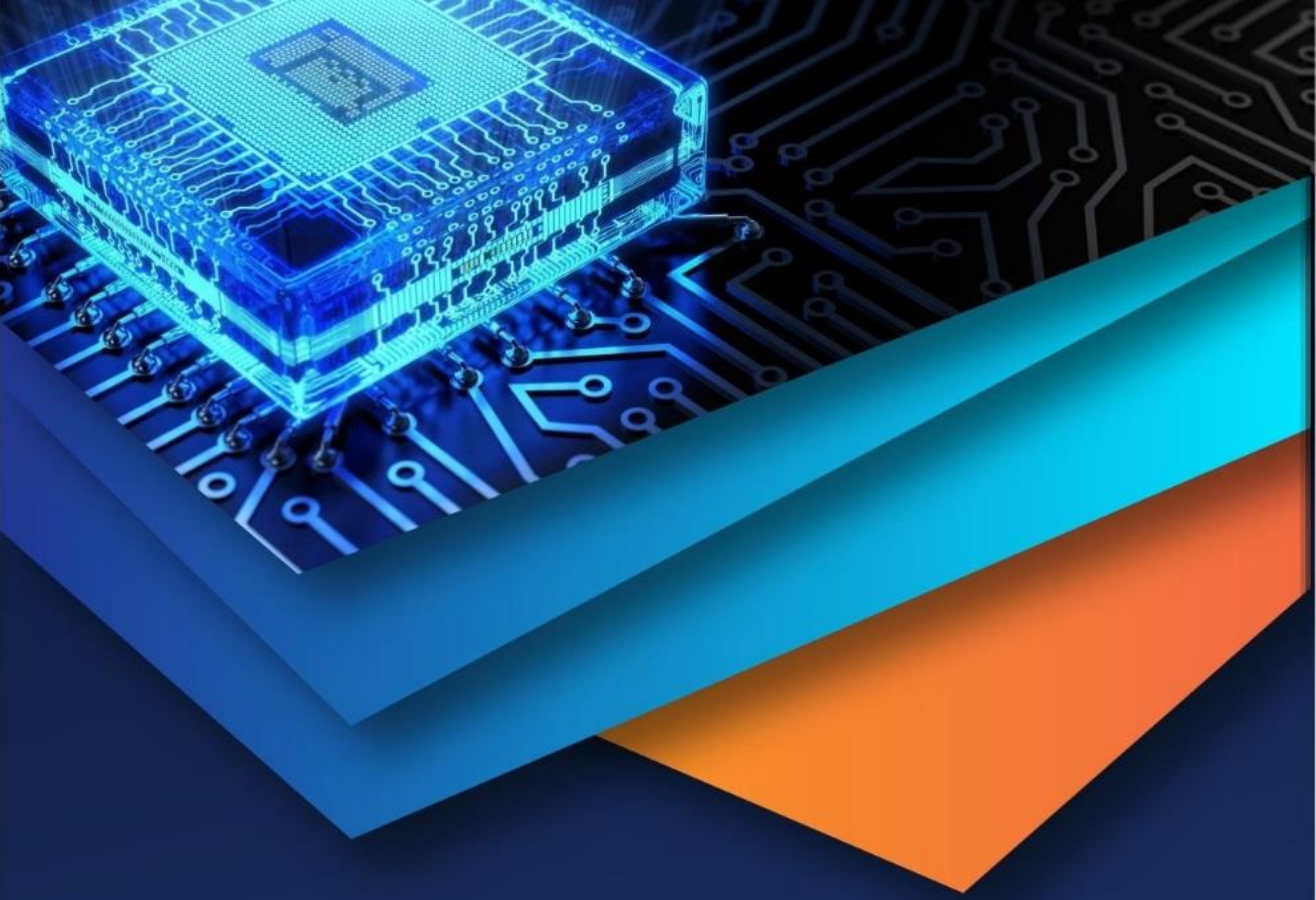

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