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Electrical Properties of Pure and Glycine added KDP-ADP Mixed Crystals

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ABSTRACT: Pure and glycine added $K_{1-x}(NH_4)_xPO_4$ single crystals have been grown at room temperature by the free evaporation method. AC electrical measurements were made by the parallel plate capacitor method on all the 15 grown crystals at various temperatures ranging from 40 to 140 °C with 5 different frequencies, viz. 100Hz, 1 kHz, 10 kHz, 100 kHz and 1MHz. Results obtained indicate that all the grown crystals exhibit a normal dielectric behavior. The AC electrical conductivity data obtained are found to obey the Arrhenius relation as well as the power law. The activation energies were also determined. The frequency, temperature and mixed composition dependences of dielectric parameters obtained are explained.

KEYWORDS: Crystal growth, KDP-ADP mixed crystals, Doped crystals, Dielectric constants, Electrical conductivities, Dielectric properties

I. INTRODUCTION

KH_2PO_4 (Potassium dihydrogen phosphate, KDP) and $(NH_4)H_2PO_4$ (Ammonium dihydrogen phosphate, ADP) are good examples of hydrogen bonded crystals exhibiting ferroelectric and antiferroelectric properties respectively at low temperatures. At room temperature, these belong to the tetragonal crystal system (The JCPDS file numbers are: For ADP: 76-0414 and for KDP: 84-0328) and are isomorphous to each other exhibiting paraelectric property. Both of them have attracted extensive attention in the investigation of hydrogen bonding behaviors in crystal and the relationship between crystal structure and their properties [1–5]. In the past few decades, many efforts have also been made to promote the crystal's quality and increase the growth rate to meet the requirements of inertial confinement fusion [6].

Mixed crystals of KDP and ADP have attracted researchers' attention because of their potential applications in quantum electronics and optoelectronics [7,8]. These mixed systems are rarely investigated because obtaining crystals with a given isomorphous composition is problematic [9,10]. However, KDP-ADP mixed crystals are considered to be interesting because of the existence of spin glass state in a certain intermediate mixing composition range due to modification of the hydrogen bonding system. So, mixing of KDP and ADP leads to significant change in the properties of the crystals. Moreover, forming hybrid materials by doping (impurity addition) also leads to significant changes in some of their properties [11-14].

Mahadevan and his co-workers, aiming at discovering new materials, have planned a research program and investigated on the growth and characterization of hybrid crystals based on KDP and ADP. Several interesting results have already been reported [15-24]. As a part of the above research program, we have grown by the free evaporation method at room temperature and characterized pure and glycine (a simple but important and interesting organic nonlinear optical material) added (0.005 and 0.010 M are the concentrations considered) KDP-ADP mixed crystals ($K_{1-x}(NH_4)_xH_2PO_4$ with $x = 0.0, 0.25, 0.5, 0.75$ and 1.0). Growth of crystals along with structural and optical properties have already been reported [25]. We report herein the results of electrical characterization and discussed.

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II. MATERIALS AND METHODS

2.1. Crystal growth

Analytical reagent (AR) grade samples of KDP, ADP and glycine along with double distilled water were used for the growth of pure and glycine added KDP-ADP mixed crystals. Single crystals (a total of 15) were grown by the free evaporation of solvent at room temperature as we have reported earlier [25]. All the crystals grown are found to be stable in atmospheric air, colorless and transparent. The single crystals grown in the present study can be represented as

KA1 → $K_{(1-x)}(NH_4)_xH_2PO_4$ with $x = 0.0$ (Pure KDP)

KA2 → $K_{(1-x)}(NH_4)_xH_2PO_4$ with $x = 0.25$

KA3 → $K_{(1-x)}(NH_4)_xH_2PO_4$ with $x = 0.50$

KA4 → $K_{(1-x)}(NH_4)_xH_2PO_4$ with $x = 0.75$

KA5 → $K_{(1-x)}(NH_4)_xH_2PO_4$ with $x = 1.0$ (Pure ADP)

KA6 → KA1 added with 0.005M glycine

KA7 → KA2 added with 0.005M glycine

KA8 → KA3 added with 0.005M glycine

KA9 → KA4 added with 0.005M glycine

KA10 → KA5 added with 0.005M glycine

KA11 → KA1 added with 0.010M glycine

KA12 → KA2 added with 0.010M glycine

KA13 → KA3 added with 0.010M glycine

KA14 → KA4 added with 0.010M glycine

KA15 → KA5 added with 0.010M glycine

2.2. Electrical characterization

Crystals without surface defects (i.e. without any pit or crack or scratch on the surface) and of size greater than 3mm were selected and used for the AC electrical (dielectric) measurements. The extended portions of the crystals were removed completely and the opposite faces were polished and coated with good quantity graphite to obtain a good conductive surface layer. The AC electrical conductivity measurements were carried out for all the fifteen grown crystals along both a- and c- directions at various temperatures ranging from 40 - 140°C by the parallel plate capacitor method in a way similar to that followed by Mahadevan and his co-workers [18,19,26-30]. The crystal (with air) capacitance (C_{crys}) and dielectric loss factor ($\tan \delta$) were measured to an accuracy of $\pm 2\%$ using an LCR meter (Agilent 4284A) with five different frequencies, viz. 100Hz, 1 kHz, 10 kHz, 100 kHz and 1MHz. The air capacitance (C_{air}) was also measured, but, only at 40°C since the temperature variation of C_{air} was found to be negligible. The dielectric constant of the crystal (ϵ_r) was calculated using Mahadevan's formula [18, 19, 26-30]:

$$\epsilon_r = [A_{air}/A_{crys}] \{ [C_{crys} - C_{air} [1 - A_{crys}/A_{air}]] / C_{air} \} \quad (1)$$

Here, A_{crys} is the area of the crystal touching the electrode and A_{air} is the area of the electrode. The AC electrical conductivity (σ_{ac}) was calculated using the relation [31]:

$$\sigma_{ac} = \epsilon_0 \epsilon_r \omega \tan \delta \quad (2)$$

Here, ϵ_0 is the permittivity of free space and $\omega (=2\pi f)$ is the angular frequency of the applied field.

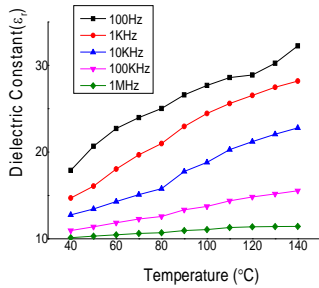
III. RESULTS AND DISCUSSION

Figures 1-3 show the dielectric constants, dielectric loss factors and AC electrical conductivities observed in the present study for the pure and glycine added KDP – ADP mixed crystals along a- and c- directions. Results obtained indicate an increase of dielectric parameters, viz. ϵ_r , $\tan \delta$ and σ_{ac} with the increase in temperature. The ϵ_r and $\tan \delta$ values decrease whereas the σ_{ac} value increases with the increase in frequency. This is considered to be a normal dielectric behavior.

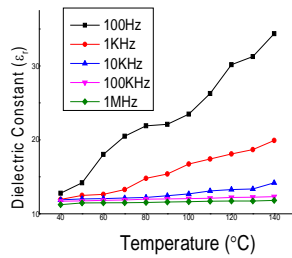
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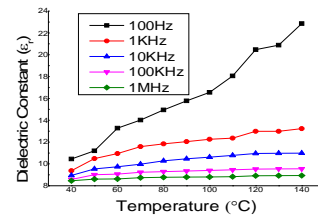
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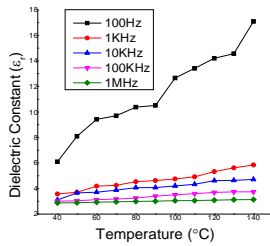
KA1 along a-direction



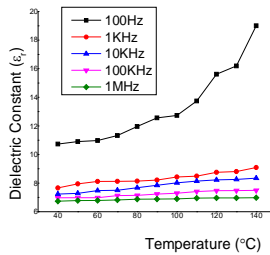
KA2 along a-direction



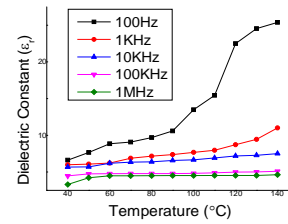
KA3 along a-direction



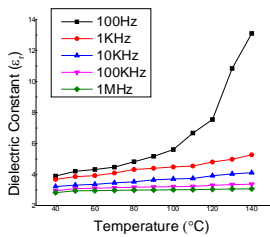
KA4 along a-direction



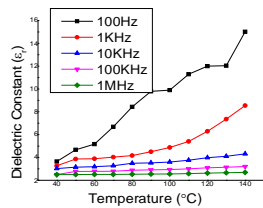
KA5 along a-direction



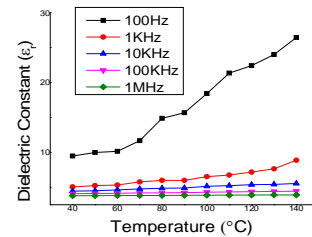
KA6 along a-direction



KA7 along a-direction



KA8 along a-direction



KA9 along a-direction

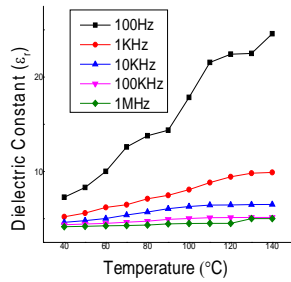
The temperature dependence of dielectric constant is normally attributed to the crystal expansion, the electronic and ionic polarizations and the presence of impurities and crystal defects. For the hydrogen bonded crystals like KDP-ADP mixed crystals, the major contributions to dielectric constant are from electronic and ionic polarizations. As all the fifteen crystals considered in the present study are good hydrogen bonded ionic crystals, ionic polarizability can be considered as the dominating contribution. Also, with the usual electric fields, the charge transported by electrons in ionic crystals is zero due to a large forbidden energy gap. So, the increase in dielectric constant with temperature is essentially due to the variation of ionic polarizability with temperature.

Electrical conductivity of the KDP-group crystals is normally determined by the proton transport within the framework of hydrogen bonds and the conduction is mainly due to the anions, namely, $(\text{H}_2\text{PO}_4)^-$ ions and not the cations $[\text{K}^+$ and $(\text{NH}_4)^+$ ions]. The proton transport can be accounted for by the motion of protons accompanied by a D defect (excess of positive charge). Migration of these defects can modify the electric polarization but not the charge at an electrode [31].

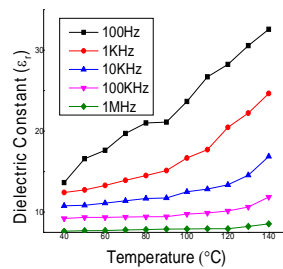
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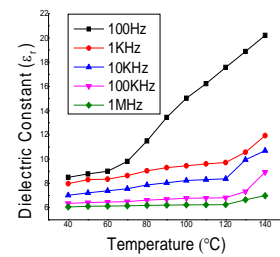
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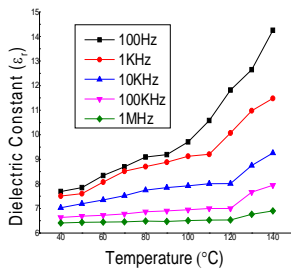
KA10 along a-direction



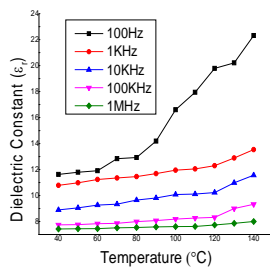
KA11 along a-direction



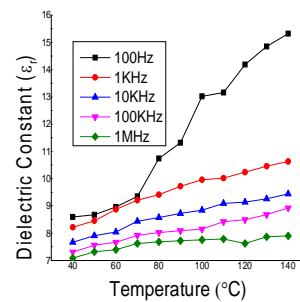
KA12 along a-direction



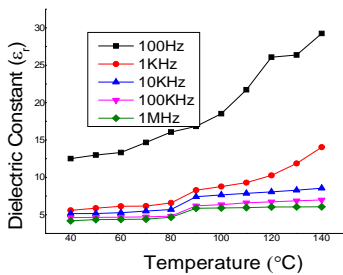
KA13 along a-direction



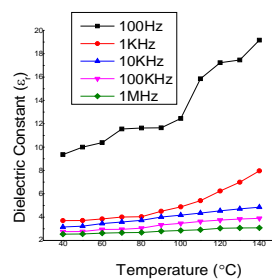
KA14 along a-direction



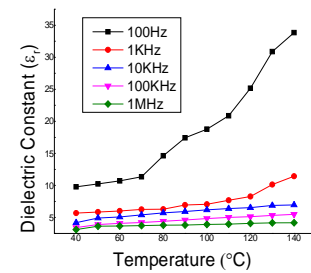
KA15 along a-direction



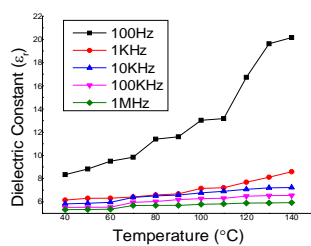
KA1 along c-direction



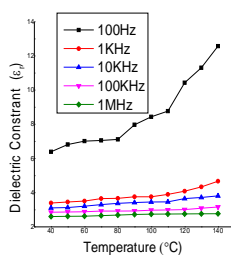
KA2 along c-direction



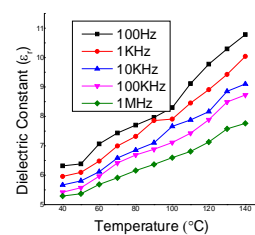
KA3 along c-direction



KA4 along c-direction



KA5 along c-direction

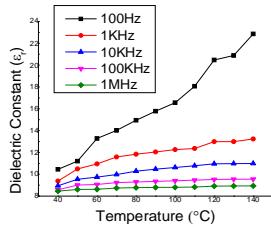


KA6 along c-direction

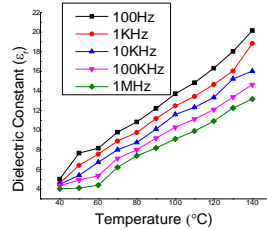
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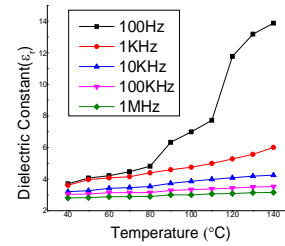
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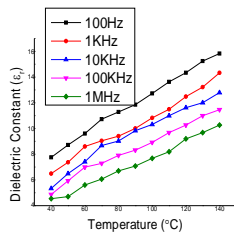
KA7 along c-direction



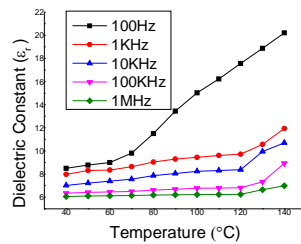
KA8 along c-direction



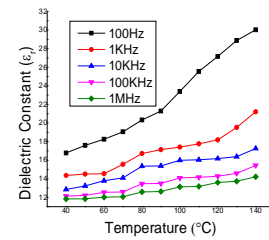
KA9 along c-direction



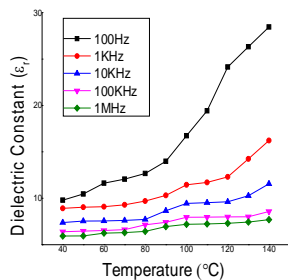
KA10 along c-direction



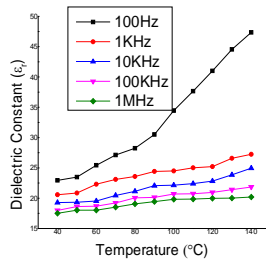
KA11 along c-direction



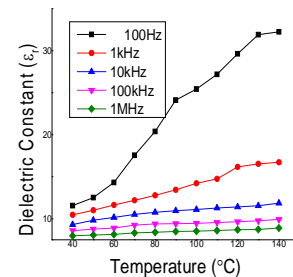
KA12 along c-direction



KA13 along c-direction



KA14 along c-direction



KA15 along c-direction

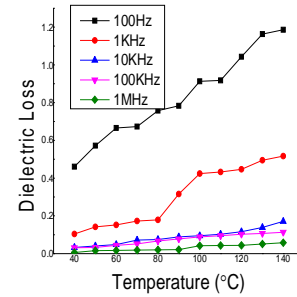
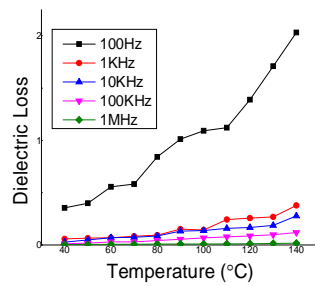
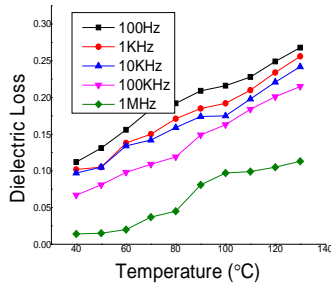
Figure:1 Dielectric constants of the grown crystals

The experimental data (especially the temperature dependence of conductivity) obtained allow us to state that the conductivity of the grown crystals is determined by both the thermally generated L-defects (vacant hydrogen bonds) and the foreign impurities incorporated into the lattice and generating L-defects there [19]. Thus, it can be understood that the proton conduction depends on the generation of L-defects and the increase of conductivity with the increase in temperature observed in the present study can be understood as due to the temperature dependence of the proton transport. Moreover, the conductivity increases smoothly through the temperature range considered in the present study. Further, there is no sharp increase in conductivity that would be characteristic of a superprotonic phase transition [19].

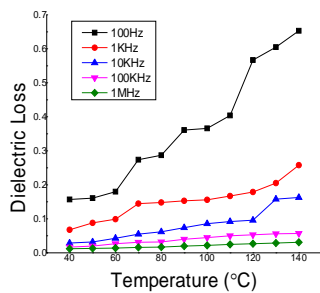
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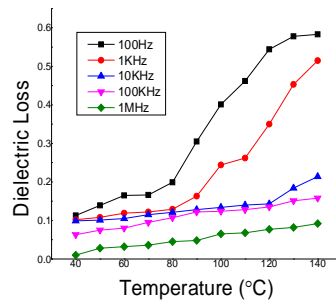
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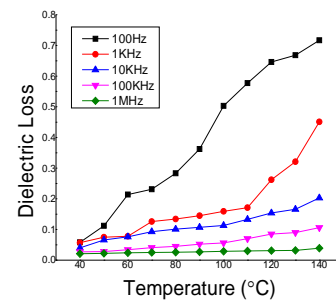
KA1 along a-direction KA2 along a-direction KA3 along a-direction



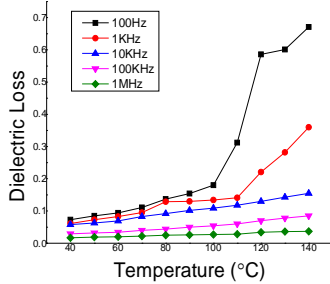
KA4 along a-direction



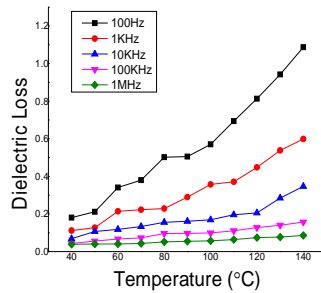
KA5 along a-direction



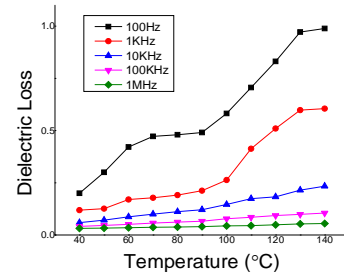
KA6 along a-direction



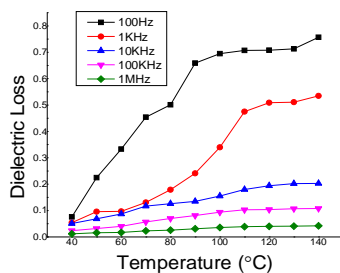
KA7 along a-direction



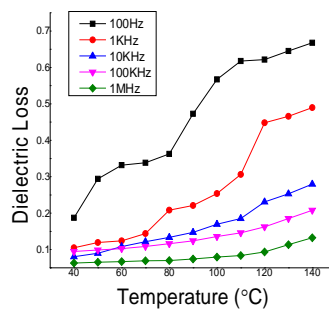
KA8 along a-direction



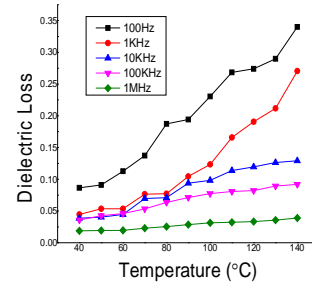
KA9 along a-direction



KA10 along a-direction



KA11 along a-direction



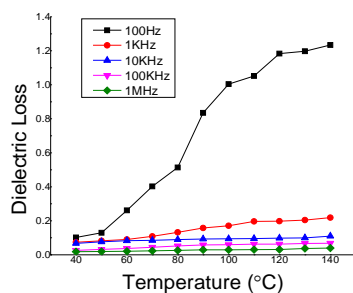
KA12 along a-direction

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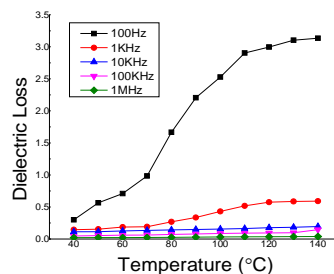
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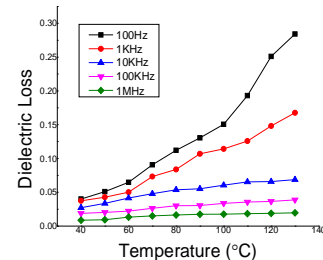
It can be seen that the bulk (mixed) composition has nonlinear influence on the AC electrical conductivity. This is similar to that reported in the literature for alkali halide mixed crystals [32-34] and $Ni_xZn_{1-x}SO_4 \cdot 7H_2O$ single crystals [31]. The nonlinear variation of σ_{ac} with bulk composition observed in the present study for the $K_{1-x}(NH_4)_xPO_4$ single crystals can be explained in a similar way and may be attributed to the enhanced diffusion of charge carriers along dislocation and grain boundaries which are expected to be more in these mixed crystals.



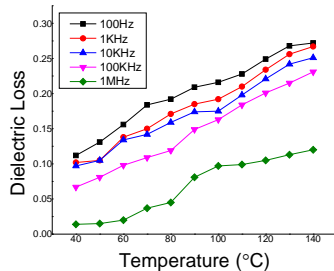
KA13 along a-direction



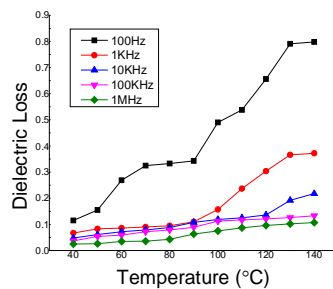
KA14 along a-direction



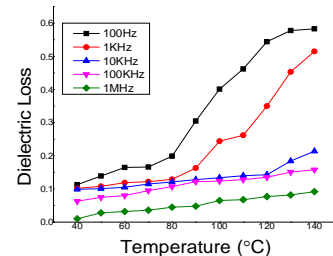
KA15 along a-direction



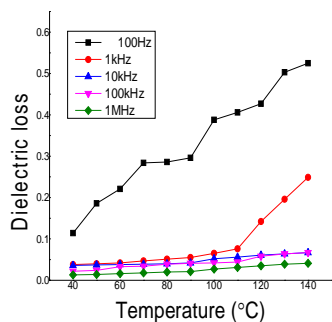
KA1 along c-direction



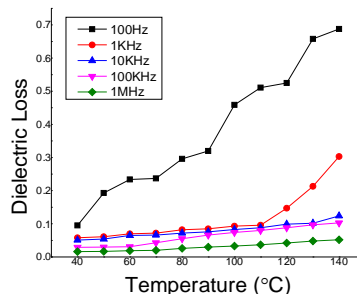
KA2 along c-direction



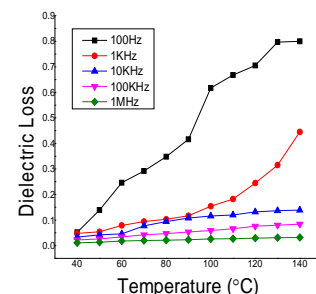
KA3 along c-direction



KA4 along c-direction



KA5 along c-direction



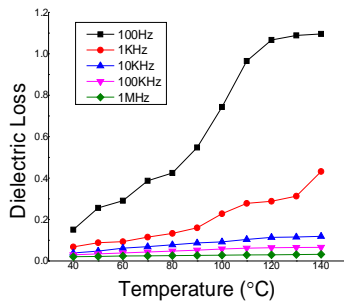
KA6 along c-direction

Glycine is a simple organic substance and the impurity concentrations considered in the present study are small. The glycine molecules are expected to occupy the interstitial positions and to create a disturbance in the hydrogen bonding system.

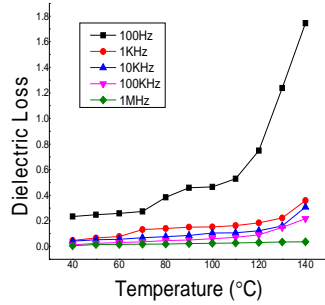
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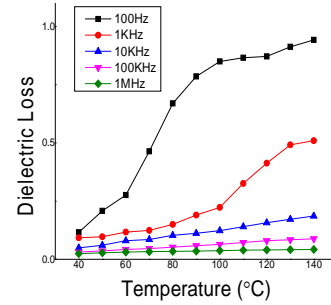
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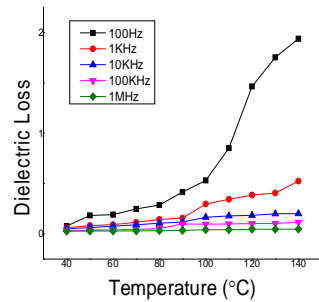
KA7 along c-direction



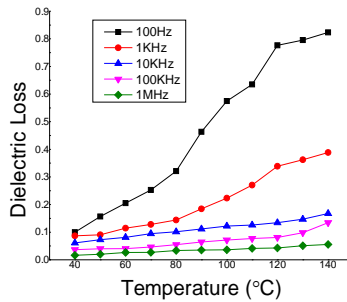
KA8 along c-direction



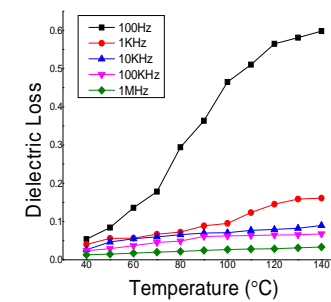
KA9 along c-direction



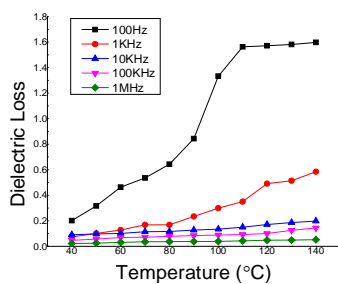
KA10 along c-direction



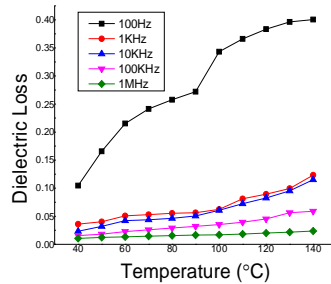
KA11 along c-direction



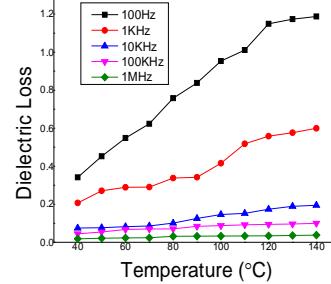
KA12 along c-direction



KA13 along c-direction



KA14 along c-direction



KA15 along c-direction

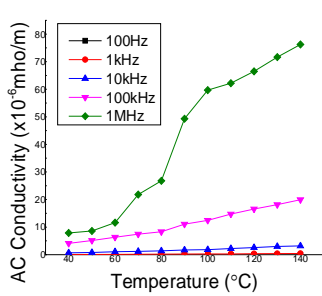
Figure: 2 Dielectric loss factors of the grown crystals

The disturbance in the hydrogen bonding system created by mixing and impurity addition may cause the dielectric parameters not to vary systematically with the impurity concentration as well as the mixed composition. In addition, the direction of measurement (along a or c) is found to have no particular influence on the dielectric parameters.

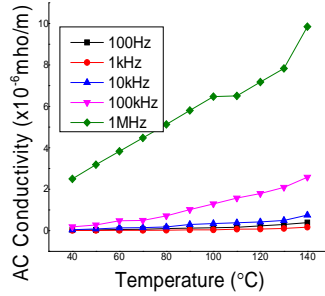
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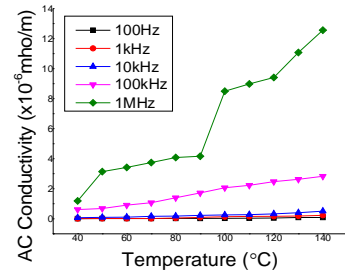
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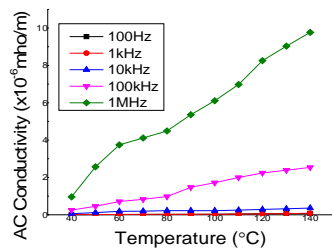
KA1 along a-direction



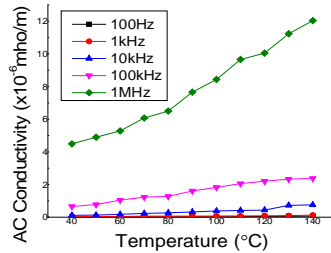
KA2 along a-direction



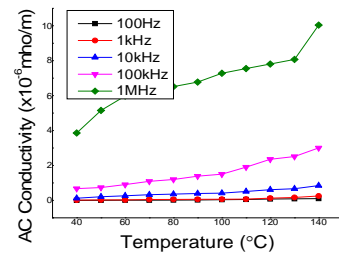
KA3 along a-direction



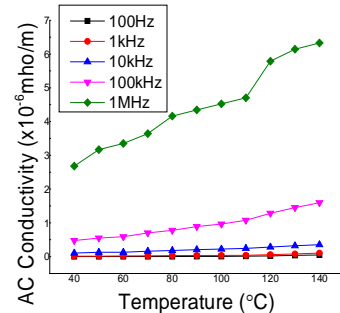
KA4 along a-direction



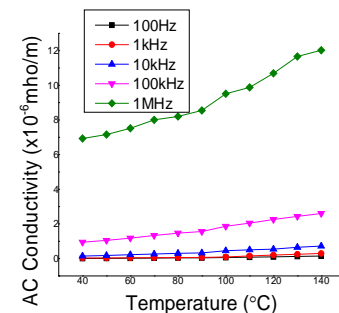
KA5 along a-direction



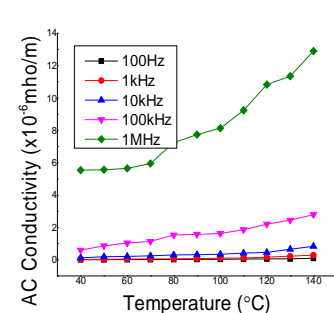
KA6 along a-direction



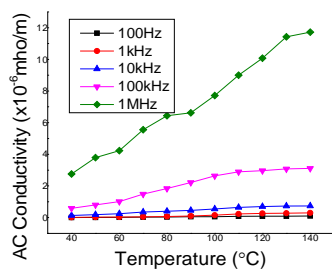
KA7 along a-direction



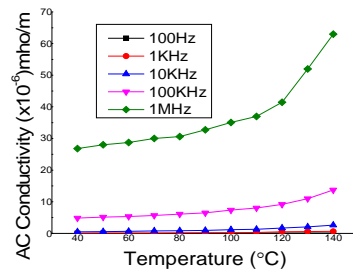
KA8 along a-direction



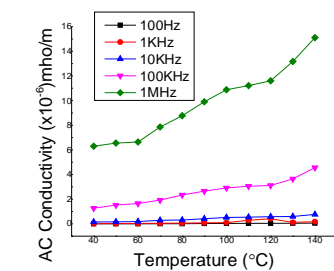
KA9 along a-direction



KA10 along a-direction



KA11 along a-direction



KA12 along a-direction

The plots between $\ln\sigma_{ac}$ and $1/T$ are found to be nearly linear (not shown here) which indicates that the AC electrical conductivity data obtained in the present study can be fitted well into the Arrhenius relation for electrical conductivity [35,36]:

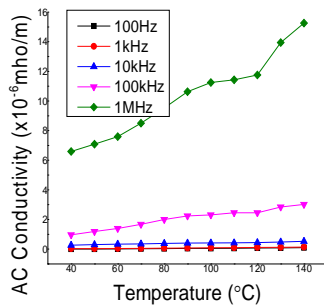
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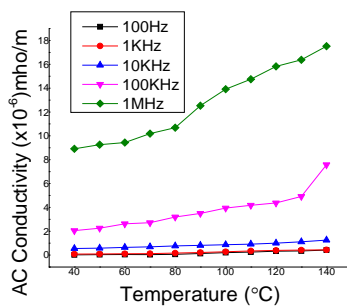
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$$\sigma_{ac} = \sigma_o \exp(-E_{ac}/kT) \quad (3)$$

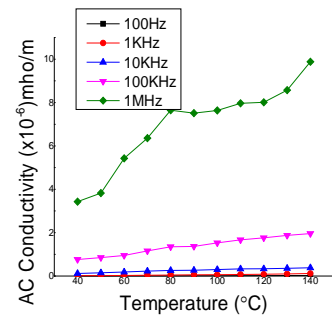
Here, σ_o is the pre-exponent factor (a constant depending on the material having the electrical conductivity unit), E_{ac} is the activation energy for electrical conduction, k is the Boltzmann constant and T is the absolute temperature. The slope of the best-fitted line curve gives $(-E_{ac}/k)$ value from which the activation energy (E_{ac}) for electrical conduction can be evaluated. The estimated AC activation energies are shown in Figure 4.



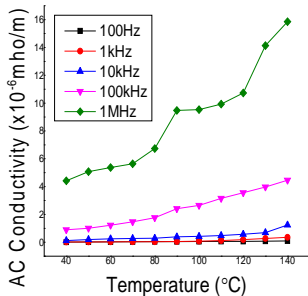
KA13 along a-direction



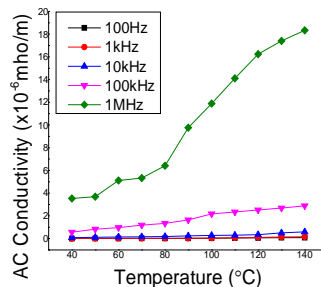
KA14 along a-direction



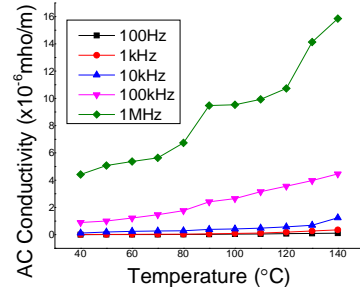
KA15 along a-direction



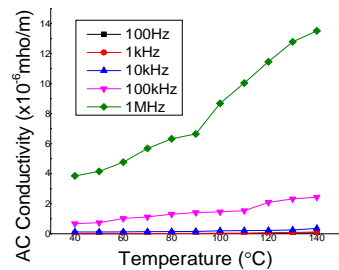
KA1 along c-direction



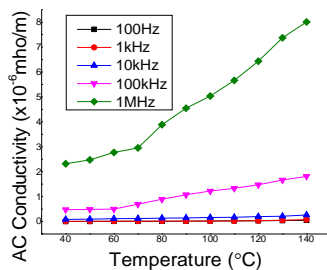
KA2 along c-direction



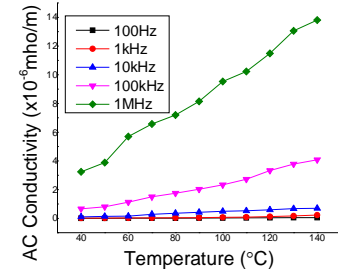
KA3 along c-direction



KA4 along c-direction



KA5 along c-direction



KA6 along c-direction

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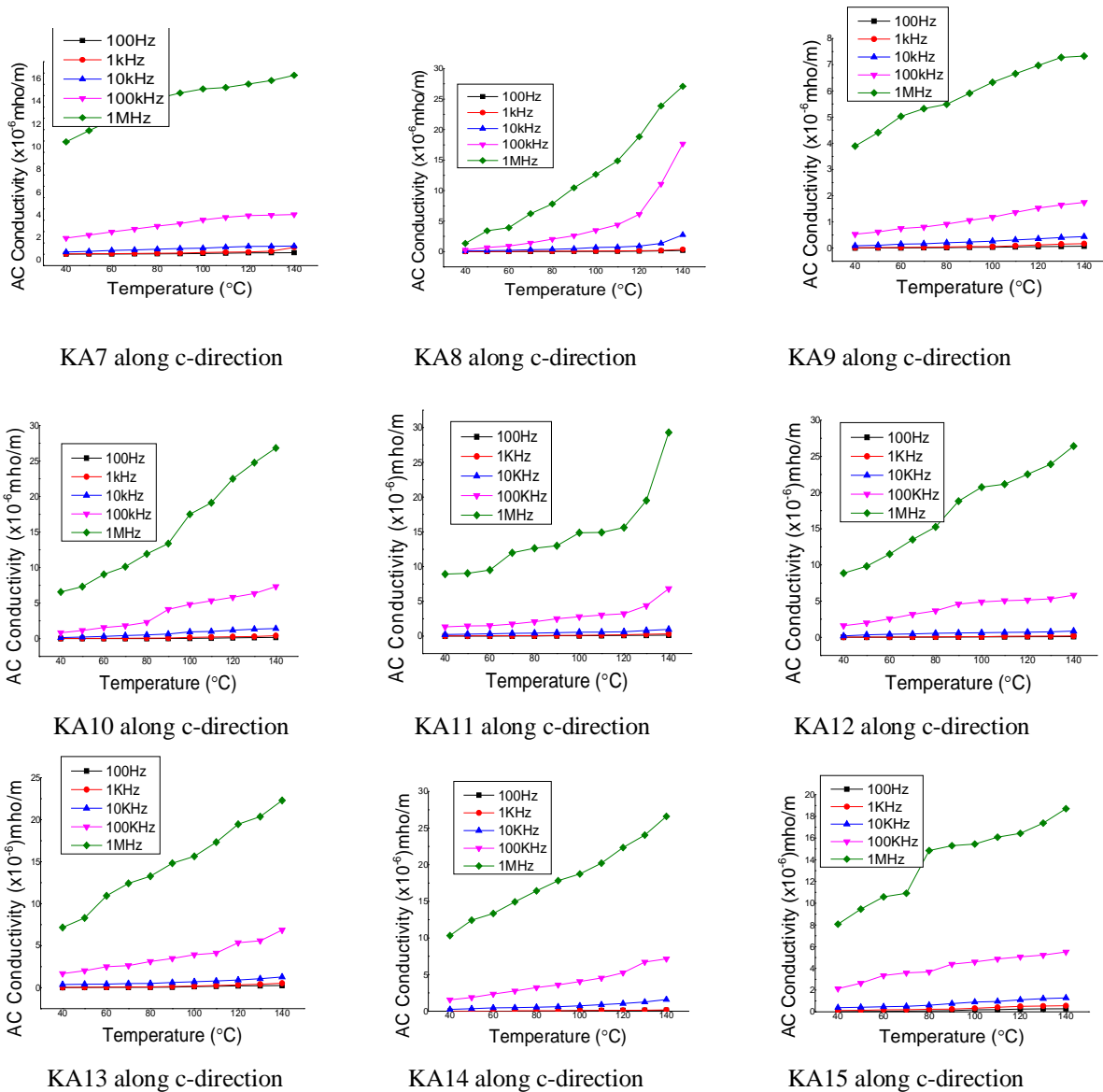


Figure: 3 AC conductivities of the grown crystals

The total electric conduction can be considered as the summation of the band and hopping parts represented by the relation:

$$\sigma_{\text{tot}} = \sigma_{\text{dc}}(T) + \sigma_{\text{ac}}(\omega, T) \tag{4}$$

The first term represents the DC conductivity due to the band conduction which is frequency independent. The second term represents the AC conductivity due to the hopping process which is frequency dependent.

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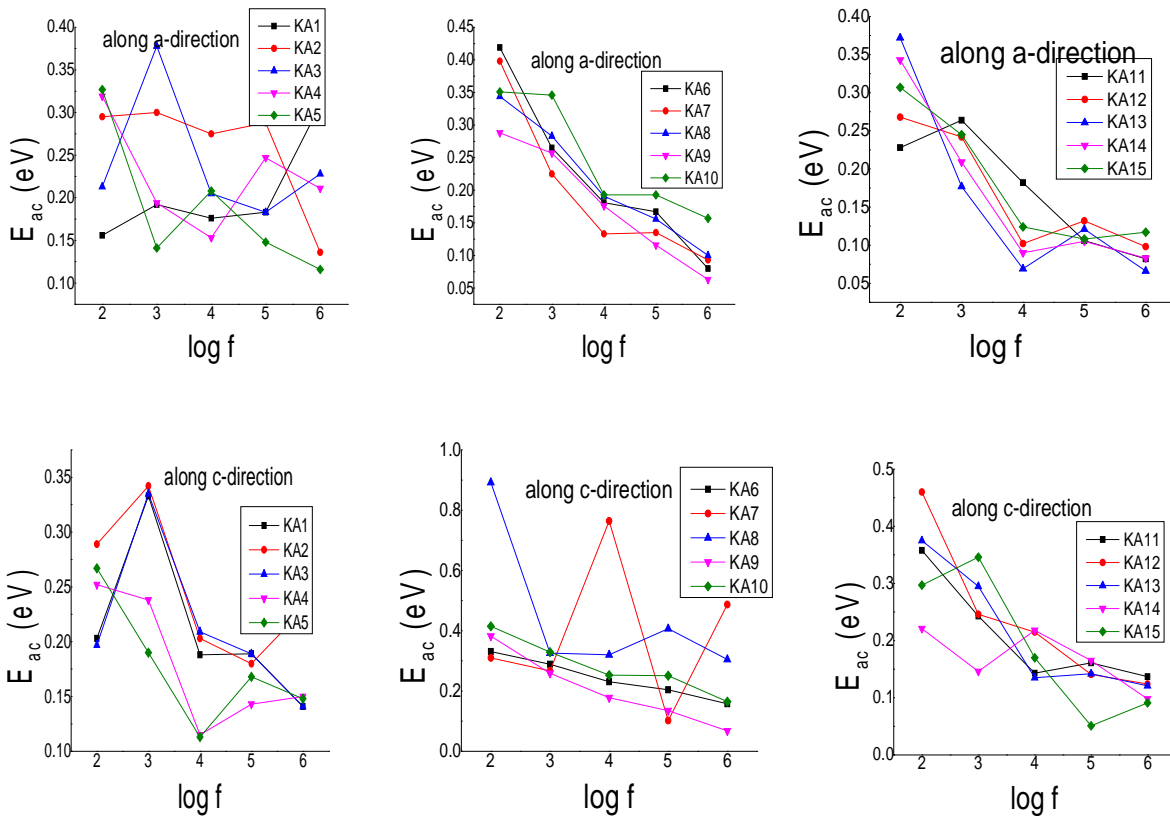
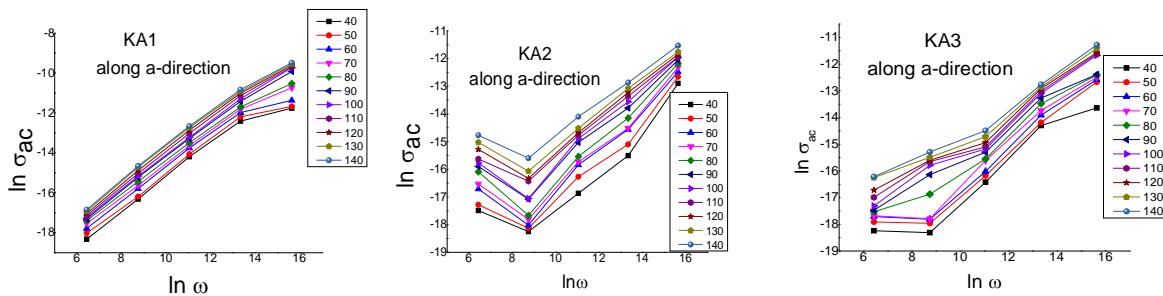


Figure: 4 The estimated AC activation energies

The AC conductivity obeys the empirical formula of the frequency dependence given by the AC power law [37]:

$$\sigma_{ac} = B\omega^m \quad (5)$$

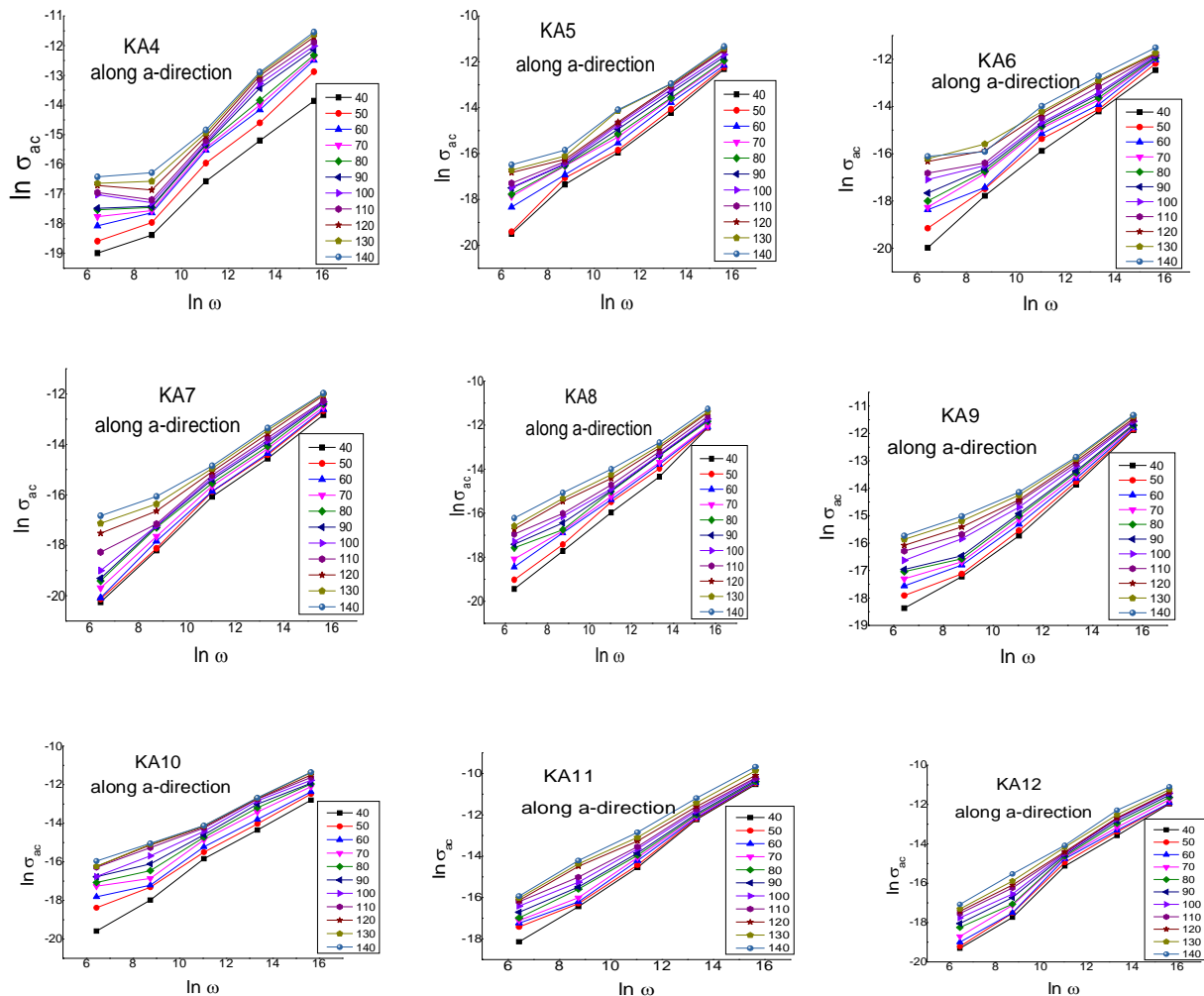
Here, B and m are constants depending on the temperature and material; m is dimensionless and B has the electrical conductivity unit. It is reported in the literature [37] that the value of m ranges between 0 and 1; when $m = 0$, the electrical conduction is frequency independent or DC conduction, but for $0 < m \leq 1$, the conduction is frequency dependent or AC conduction.



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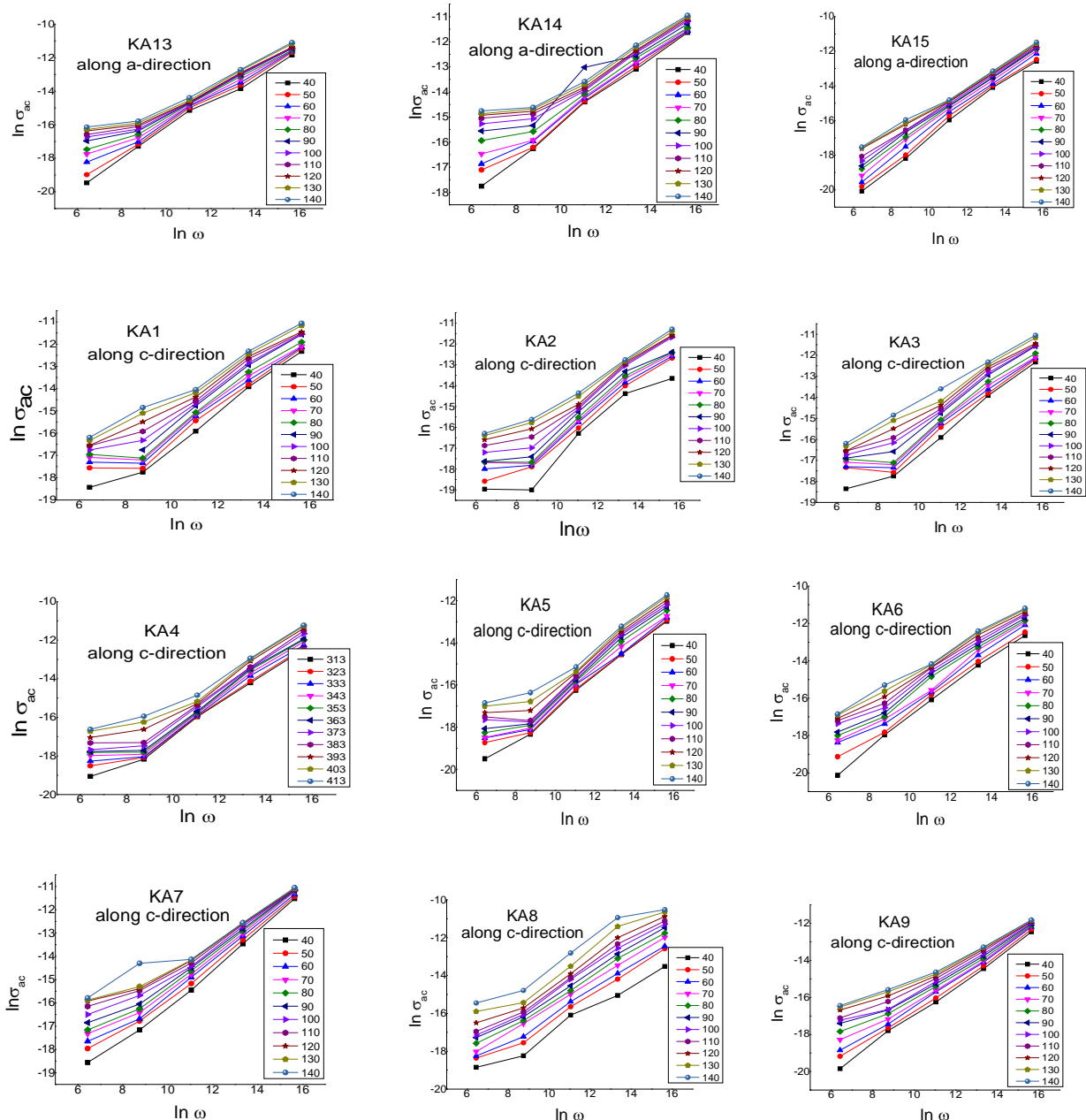


The plots between $\ln \sigma_{ac}$ and $\ln \omega$ (shown in Figure 5) are found to be nearly linear which indicates that the AC electrical conductivity data obtained in the present study could be well fitted into the above power law. The slope and y-intercept of the best-fitted line curve gives respectively the m and B values. The B values obtained in the present study are shown in Figure 6. The m values obtained lie in the range 0.399 – 0.947 which suggests that the conduction phenomenon in all the 15 crystals grown in the present study is AC conduction due to hopping of charges (especially when the frequency is 1000 Hz or more).

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The lower E_{ac} values obtained in the present study indicate oxygen vacancies which would create vacant hydrogen bonds (L-defects).

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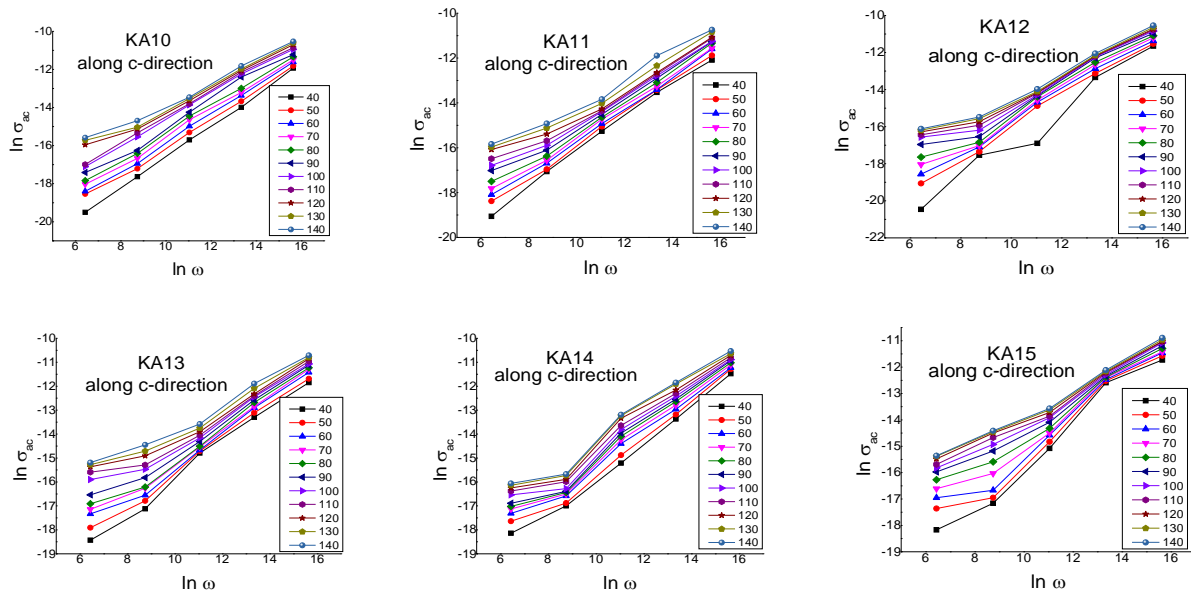


Figure: 5 The plots between $\ln \sigma_{ac}$ and $\ln \omega$

Moreover, the AC electrical conductivities obtained experimentally are found to be more than the DC electrical conductivities (the B values) obtained by dielectric analysis using the power law for electrical conductivity.

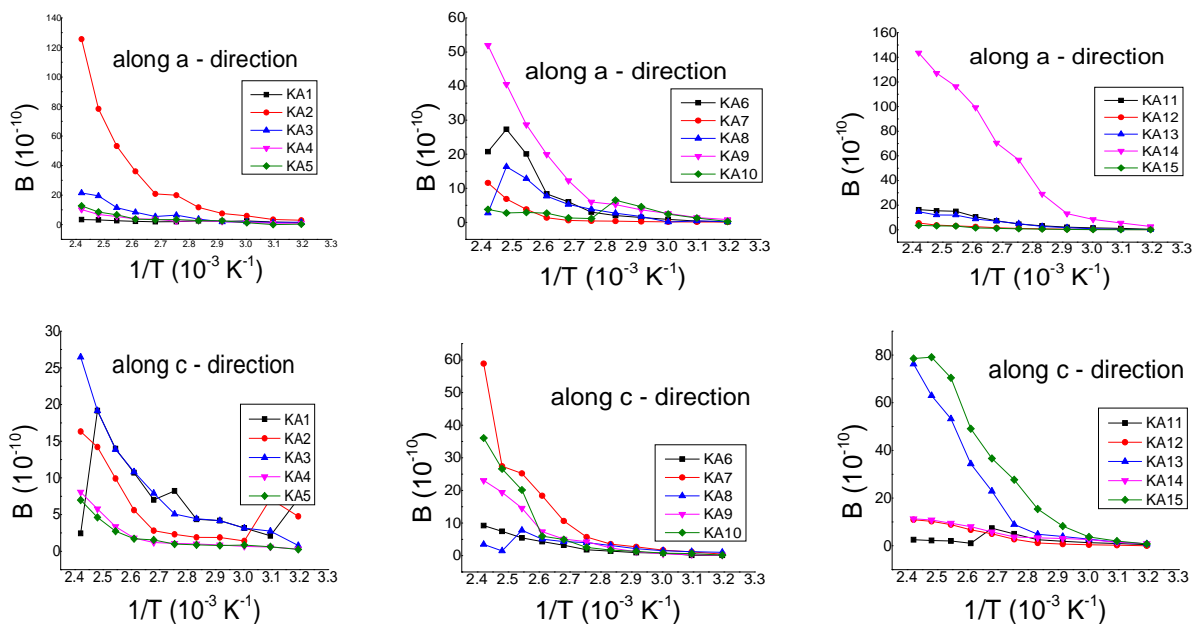


Figure: 6 The plots between B (mho/m) and $1/T$

As this aspect is normally exhibited by the dielectric crystals, the DC conductivity data obtained for the crystals grown in the present study may be considered as valid. Thus, the results of AC electrical measurements made in the present

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study follow well the AC power law. Moreover, the dielectric analysis indicates that the applied field is more or less static when the frequency is lower (less than 1000 Hz) and significantly dynamic when the frequency is higher.

IV. CONCLUSIONS

Pure and glycine added $K_{1-x}(NH_4)_xPO_4$ single crystals (a total of 15) have been successfully grown by the free evaporation method at room temperature. Results of AC electrical measurements made along a- and c- directions by the parallel plate capacitor method (at various temperatures ranging from 40 to 140°C and with 5 different frequencies, viz. 100 Hz, 1 kHz, 10 kHz, 100 kHz and 1 MHz) indicate that the dielectric parameters, viz. ϵ_r , $\tan \delta$ and σ_{ac} increase with the increase in temperature. Also, the ϵ_r and $\tan \delta$ values decrease whereas the σ_{ac} value increases with the increase in frequency indicating that the grown crystals exhibit a normal dielectric behavior. The observed σ_{ac} values could be well-fitted into the Arrhenius equation as well as the power law. The low activation energies obtained indicate the possibility of oxygen vacancies leading to L-defects (vacant hydrogen bonds). The dielectric properties observed for the grown crystals could be explained as mainly due to the ionic polarizability and proton transport and their temperature dependences. Also, the disturbance in the hydrogen bonding system caused due to mixing as well as glycine addition leads the dielectric parameters not to vary systematically with the mixed composition as well as glycine concentration. In addition, no directional influence could be observed.

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