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# Electrical Parameter Mesasurements Of Naxk1-Xbr Single Crystals Grown From Aqueous Solution

Thanuja M N [(a)] & Dr. Neelakanda Pillai N.[(b)]
*(a) B.T. Assistant, GHSS Devarshola, The Nilgiris*
*(b) Associate Professor, Department of Physics, Arignar Anna College, Aralvoimozhi*

***Abstract:*** *Development of lasers revived the interest in alkali halides as materials for optical components. This led to the development of alkali halide-polycystalline materials for use as optical windows. But their uses are limited by their mechanical properties and stability in some cases. Hence there exists the need to strengthen these and improve their stability. In this work, mixed crystals of NaBr and KBr were grown by slow evaporation method. Densities and refractive indices of all the grown crystals were determined and also used for the estimation of the bulk composition in crystal lattice parameters. Dielectric constant and dielectric loss were measured by conventional two probe set up. AC conductivity and activation energy were also determined. The detailed results are reported in this paper.*

## I. Introduction

A mixed crystal is obtained by crystallizing together two isomorphous crystals like NaBr and KBr with comparable lattice constants. Alkali halide crystals are of the completely disordered substitutional type. Haribabu and Subbarao [1] have reviewed the aspects of the growth and characterization of alkali halide mixed crystals. Sirdesh Mukh and Srinivas [2] have reviewed the physical properties. Several more reports are available on binary mixed crystals of alkali halides [3-5]. Ionic conductivity measurements as a function of temperature have been done by a number of researchers on pure alkali halide crystals and also impurity added ones. Various defect parameters such as activation energy for migration, formation energy etc. have been evaluated from these studies. Though an extensive amount of work has been done on pure and impurity added alkali halide crystals, the work on mixed crystals of alkali halides is very limited. Electrical conductivity measurements have been carried out on some binary mixed crystals
like KCl – KBr,KBr – KI, KCl-RbCl, NaCl-NaBr etc [6] Mahadevan and his co-workers measured electrical parameters for their ternary mixed crystals [7-11]. They also found that the dielectric parameter vary non-linearly with compositions.

Measurement of dielectric constant and loss as a function of frequency and temperature is of interest both from theoretical point of view and from the applied aspects. Recently Neelakanda Pillai and his co-workers have measured electrical parameters at various temperatures for various frequencies [12-14] for crystals grown from aqueous solution. They found that, the dielectric constant increases with temperature and decreases with frequency and they have no linear variation with composition.

Large difference in the values of dielectric constant of KCl-KBr system was observed. When compared to the values obtained by Ferritel and Perry. As a cross check Prameela Devi[15] re-determined the dielectric constant of KCl-KBr mixed crystals for various compositions at room

Temperature. Her results favour the values obtained by Kamiyoshi and Nigara and differ considerably from those of Fertel and Ferry. Later Sathaia[3] determined the dielectric constant and dielectric loss at elevated temperature upto about 400$^{o}$C as a function of frequency and also as a function of composition for KCl-KBr and RbCl-RbBr mixed crystals. Also he has analyzed the results semi theoretically.

## II. Experimental Details

### 2.1 Growth Of Single Crystals

NaxK1-xBr single crystals were grown from the aqueous solutions by the slow evaporation method. Anala R grade samples of NaBr and KBr were used as the starting materials and doubly distilled water was used for the growth of the crystals.

Supersaturated solutions were prepared for various values of x ranging from 0.1 to 0.9 including two end member crystals in identical conditions. Crystals were harvested after three weeks.

### 2.2 Density measurements

Densities of all the grown crystals were determined by using the floatation method. Carbon tetra chloride of density 1.594 gm/cc and Bromoform of density 2.890 gm/cc were used as lower and higher density liquids respectively.

**2.3 Refractive Index Measurement**

Refractive index of an under saturated solution of the crystal in distilled water was measured using an Abbe's refractometer.

Refractive index of the crystal was determined by using Gladstone's rule [16] [(n-1)]P= [(n-1)/d1]P1 + [(n2-1)/d2]P2 Where n, n1 and n2 represents the refractive indices of the solution, solvent and solute respectively. d, d2 and d3 are the densities of the solution, solvent and solute respectively. P, P1 and P2 are the percentage weights of the solution, solvent and solute respectively.

**2.4 Estimation of bulk composition**

It has been found that the density values form a linear relationship with composition for the binary mixed crystals[1] as

$$d= xd1 +(1-x)d2$$

Here d1 and d2 represents the densities of NaBr and KBr

**2.5 Lattice Parameter**

X-ray Diffraction data were collected from powdered samples using an automated X-ray powder diffractometer with scintillation counter and monochromated CuKa ($\lambda$ = 1.5418Å) radiation. The reflections were indexed following the procedures of Lipson and Steeple [17]. Analysis of the X-ray diffraction peaks for the binary mixed systems considered in the present study by the available methods shows that the mixed crystals can be indexed with single fcc lattices.

**2.6 Electrical Measurements**

The capacitance and dielectric loss factor (tanδ) measurements were carried out to an accuracy of ±3% using HIOKI 3532-50 HITESTER LCR meter. With a frequeny of 1kHz at various temperature ranging from 30$^o$C to 150$^o$C in a way similar to that followed by Neelakanda Pillai[8]. The observations were carried while cooling the sample. Temperature was controlled to an accuracy of ±1 $^o$C. The grown crystals were polished and opposite faces of the sample crystals were coated with good quality graphite to obtain a good conductive surface layer. They were annealed for two hours at 150$^o$C to remove moisture contents if present. The dielectric constant of the crystal was calculated using the relation (as the crystal area was smaller than the plate area of the cell.) Also, the capacitances were measured for frequencies ranging from 100 Hz to 5 MHz at temperatures 40$^o$C, 70 $^o$C,110 $^o$C and 150 $^o$C.

$$\varepsilon_r = \left(\frac{A_{air}}{A_{crys}}\right)\left(\frac{C_{crys} - C_{air}\left(1 - \left(\frac{A_{crys}}{A_{air}}\right)\right)}{C_{air}}\right)$$

where $C_{crys}$ is the capacitance with crystal (including air),

$C_{air}$ is the capacitance of air,

$A_{crys}$ is the area of crystal touching the electrode and

$A_{air}$ is the area of electrode.

The AC conductivity $\sigma_{ac}$ was calculated using

$$\sigma_{ac} = \omega\ \varepsilon_o\ \varepsilon_r \tan\delta \quad \text{mho m}^{-1},$$

where εo is the permittivity of free space (8.854 x $10^{-12}$ F/m) and ω is the angular frequency (ω = 2πf; f = 1 kHz, 10 kHz, 100 kHz and 1 MHz in the present study).

The σac values were fitted into the general equation for the temperature variation of conductivity given by

$$\sigma_{ac} = \sigma_o \exp\left[\frac{-\Delta E_{ac}}{kT}\right],$$

where Eac is the bandgap energy of the material, T is the absolute temperature of the sample and σo is a constant depending on the material.

## III. Results And Discussions

The binary mixed crystals are found to be transparent and harder than end member crystals. All the grown crystals exhibit cleavage properties which show that the grown crystals are single crystals. The maximum size of crystals grown is 10 mm x 10 mm x 5 mm. Photographs of all the grown crystals are shown in figure 1.

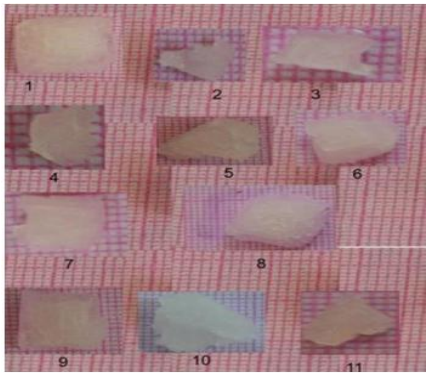



**Figure: 1**

Photograph of pure and mixed NaxK(1-x)Br

various temperatures for NaxK(1-x)Br (x = 0.0, 0.1, (b)Na0.1K0.9Br (c) Na0.2K0.8Br (d) Na0.3K0.7Br
(e) Na0.4K0.6Br (f) Na0.5K0.5Br (g) Na0.6K0.4Br
(h) Na0.7K0.3Br (i) Na0.8K0.2Br (j) Na0.9K0.1Br
(k) NaBr single crystals (a) KBr

The density, refractive index and estimated composition of all the crystals are given in table 1 along with lattice parameter. Observed density and refractive index of the end members compare well with those reported in the literature [1]. Variation of dielectric constant with temperature at 1kHz frequency for all the eleven crystals is shown in figure 2. It is found from the graph that the dielectric constant values increase with increase in temperature. This is similar to that observed for the previous authors [11-13 ]. Variation of εr with temperature is generally attributed to the crystal expansion, the electronic and ionic polarizations and the presence of impurities and crystal defects. The slow variation of εr of at low temperature is mainly due to the expansion of electronic and ionic polarizations. At higher temperatures, the increase is mainly attributed to the thermally generated charge carriers and impurity dipole.

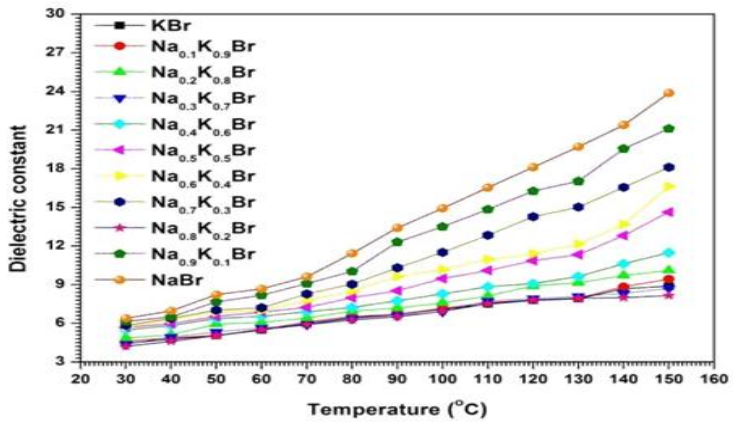



**Figure 2:** Variation of dielectric constant at 0.2, 0.3, 0.4,0.5, 0.6, 0.7, 0.8, 0.9 and 1.0) Single crystals at 1 KHz frequency.

**Table 1: Density, Refractive index, estimated compositions and lattice parameters of all the grown crystals**

| System | Density | Refractive index | Lattice parameter (Å) | Estimated Composition |
|---|---|---|---|---|
| KBr | 2.7254 | 1.554 | 6.5894 | |
| NaBr | 3.1895 | 1.643 | 6.0995 | |
| Na0.1K0.9Br | 2.7532 | 1.568 | 6.5339 | Na0.132K0.868Br |
| Na0.2K0.8Br | 2.7703 | 1.578 | 6.4981 | Na0.271K0.729Br |
| Na0.3K0.7Br | 2.7912 | 1.582 | 6.4538 | Na0.321K0.679Br |
| Na0.4K0.6Br | 2.8356 | 1.591 | 6.3968 | Na0.418K0.582Br |
| Na0.5K0.5Br | 2.8911 | 1.599 | 6.3302 | Na0.509K0.491Br |
| Na0.6K0.4Br | 2.9198 | 1.607 | 6.2867 | Na0.611K0.389Br |
| Na0.7K0.3Br | 2.9274 | 1.623 | 6.2440 | Na0.793K0.207Br |
| Na0.8K0.2Br | 3.0123 | 1.631 | 6.1791 | Na0.868K0.132Br |
| Na0.9K0.1Br | 3.1231 | 1.69 | 6.1319 | Na0.923K0.077Br |

Dielectric constants of all mixed crystals are higher than KBr and it was influenced by the composition of the crystals. The rate of increase with temperature is higher for the mixed crystals than the end members. Varotsos [18] has shown that, out of the two contributions, the electronic polarizability practically remains constant. The increase in dielectric constant with temperature is essentially due to the temperature variation of ionic polarizability.

The variation of dielectric constant with frequency (100 Hz to 5 MHz) for different temperatures viz. 30, 70, 110, 150 ºC is shown in Figures 3. for the

equimolar concentration of NaxK(1-x)Br the figure indicate that the dielectric constant decreases rapidly at lower frequency region (100Hz to 1 KHz) and gradually decreases at moderate frequency region (1 kHz to 10 KHz) while it remains almost constant at high frequency region (above 10 KHz). Dielectric constant is attributed to four types of polarizations [19]. They are electronic, ionic, orientation and space charge polarizations. At lower frequencies at which all four types of polarizations contribute, the rapid increase in dielectric constant is mainly due to space charge and orientation polarizations, which are strongly temperature dependent [20,21]. The variation of dielectric constant at lower

Frequencies is attributed not due to the electronic and ionic contribution but due to the space charge contribution. This space charge polarization occurs up to frequencies of around 1 kHz. The orientation polarization does not exist for ionic crystals. The major contributions to dielectric constant for the alkali halide crystals are from space charge, electronic and ionic polarizations. While with the increase of frequency the ionic and electronic contribution becomes dominant and space charge contribution diminishes gradually and hence dielectric constant decreases with increase of frequency and attains almost constant value at higher frequencies. In the fourth stage polarization is only due to the electronic and hence the dielectric constant value goes very low beyond 100 KHz. The εr increases with the increasing concentration of Na in KBr lattice at all temperatures considered in the present study. However, εr is obtained minimum for the case of Na0.3K0.7Br and Na0.8K0.2Br crystals at all temperatures and almost all frequencies. As the temperature increases the dielectric constant also increases at all frequencies. Similar behavior was observed for all the pure and mixed NaxK(1-x)Br crystals. This may be due increasing cationic vacancies with respect to increasing temperature. As the temperature increases, the space charge increases, in agreement with the temperature dependences of the conductivity, the dielectric constant, the capacitance, and the dielectric loss of ionic crystals. Annenkov and Malofienko[22] calculated the space charge polarization with respect to the temperature from Venderovich equation. He proved that the space charge polarization increases with increasing temperature as the result of an increase in the carrier concentration of cationic vacancies.

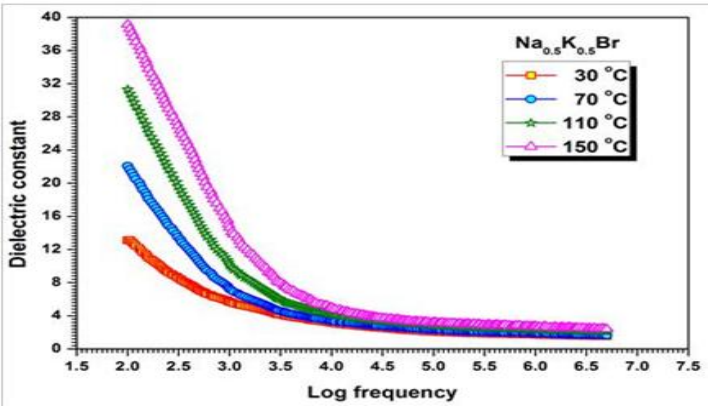



**Figure 3:** Variation of dielectric constant with frequency at various temperatures for Na0.5K0.5Br single crystal

The temperature variation of dielectric loss (tan δ) at 1 kHz is shown in Figure 4. From this figure it is clear that the tan δ increases with the increase of Na content up to x = 0.5 and then tanδ decreases with the increase of Na content x = 1.0. Also the loss increases with increasing temperature due to the increased conduction of thermally activated ions in NaxK(1-x)Br lattices.

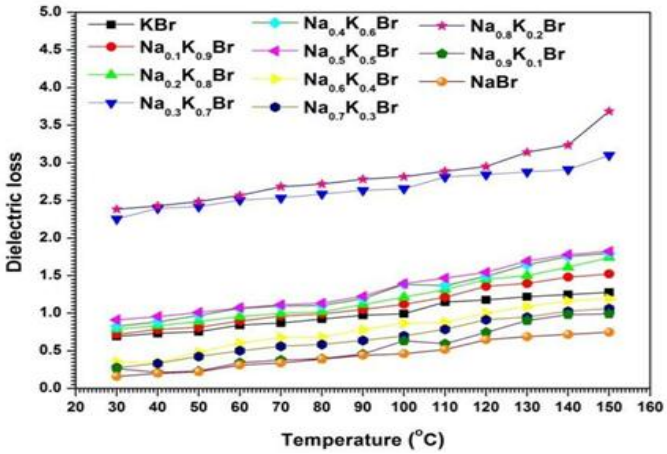



**Figure 4:** Variation of dielectric loss at various temperatures for NaxK(1-x)Br (x = 0.0, 0.1, 0.2, 0.3, 0.4,0.5, 0.6, 0.7, 0.8, 0.9 and 1.0) single crystal at 1 KHz frequency.

The variation of dielectric loss (tan δ) with frequency at different temperatures is shown in Figures 5. The values of tan δ are higher at low frequencies and high temperatures. This behavior is due to the defects arising out of thermally generated charge carriers. The tan δ decreases rapidly in the low frequency region, while the rate of decrease is slow in the middle of the frequency and then at high frequency it becomes almost frequency independent. The tan δ decreases from 1.97 at 100 Hz to a value of 0.14 at 5 MHz at room temperature (30 ºC) for KBr crystal. It can be seen that the loss (tan δ) is low in the region where dielectric constant is independent of frequency. The low loss indicates that the effect of impurities is negligible in frequency independent region at room temperature. High value of εr and tan δ in the low frequency can be attributed to the presence of space charge polarization [23] in the sample. Similar behavior was observed for Na mixed in KBr crystals. Also there is no systematic variation of tan δ was observed with respect to concentration of Na in KBr lattices. However when all the K ions are replaced by Na ions very low dielectric loss was observed. Also low tan δ values was observed for x = 0.7 and x = 0.9 in NaxK(1-x)Br mixed crystals (Na0.7K0.3Br and Na0.9K0.1Br) at all temperatures from 30 to 150 ºC.

From Figure 5, it shows that the material has very less dielectric loss with increase in frequencies. The non-uniform variation of dielectric loss with frequency for all the temperature at lower frequency region may be due to the effect of small variations in polarization with respect to applied filed. It is known that effect of polarization is to reduce the field inside the medium. Therefore, the dielectric constant of a substance may decrease substantially as the frequency is increased. The low value of dielectric loss at high frequency can be taken as a proof for the good optical quality of the crystal [24].

In the present study, it has been observed that the εr value for mixed NaxK(1-x)Br crystals (x = 0.1 to 0.9) is significantly for the pure NaBr crystal. Thus, in effect, the present study indicates that mixed NaxK(1-x)Br crystals are a promising low εr value dielectric material at room temperature.

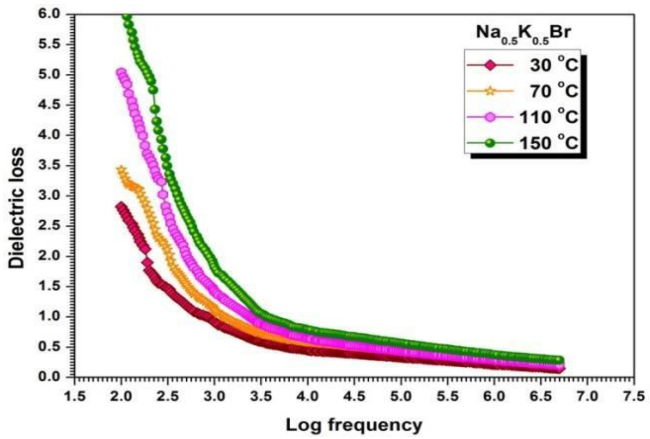
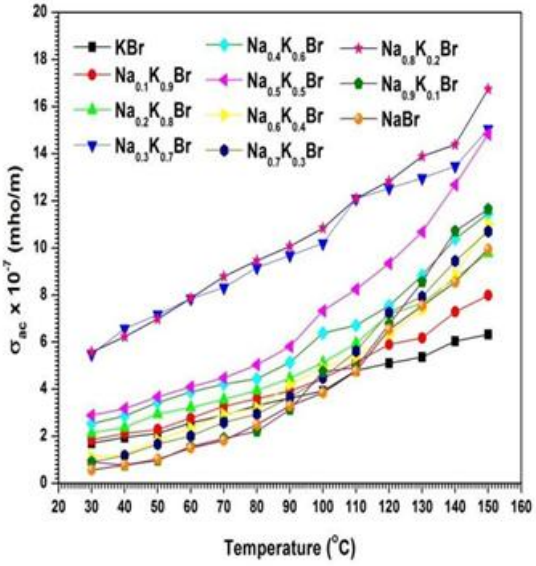



**Figure 5.:** Variation of dielectric loss with frequency at various temperatures for Na0.5K0.5Br single crystal




The variation of AC conductivity of pure and mixed NaxK(1-x)Br crystals as a function of temperature for different compositions of Na in the temperature range of 30–150 ºC at 1 kHz frequency is shown in Figure 6. From the plots, it is clear that the conductivity is found to increase with increase of temperature in pure KBr crystal as well as in all the compositions of Na in NaxK(1-x)Br crystals. The mechanism of electrical conductivity in alkali and silver halide crystals is usually the motion of ions and not the motion of electrons. This has been established by comparing the transport of charge with the transport of mass as measured by the material plated out on electrodes in contact with the crystal [25]. From the Figure 6, it reveals that the AC conductivity of the mixed NaxK(1-x)Br crystals (x = 0.1 to 0.9) are higher than that of pure NaBr and KBr crystals. However there is no systematic variation of conductivity with respect to concentration of Na in KBr lattices.

observed such a nonlinear composition dependence of activation energy for KCl-KBr system and attributed this to the result of enhanced diffusion of charge carriers along dislocations [28]. This could probably account for the variation observed in εr, tan δ and ΔEac for the present grown mixed crystals.

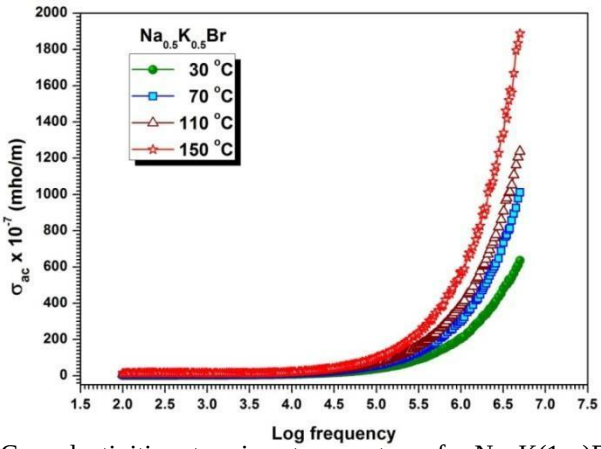



**Figure 6:** Variation of AC conductivities at various temperatures for NaxK(1-x)Br (x = 0.0, 0.1, 0.2, 0.3, 0.4,0.5, 0.6, 0.7, 0.8, 0.9 and 1.0) single crystal at 1 KHz frequency.

The variation of AC conductivity (σac) with frequency at different temperatures (30, 70, 110 and 150 ºC) for pure and mixed NaxK(1-x)Br crystal is shown in Figures 7. The conductivity is high for higher frequencies at a given temperature confirming polaran hopping in the crystals[26,27]. It is clear from the figure that conductivity increases as the temperature increases. This increase in conductivity could be due to the reduction in the space charge polarization at higher frequencies.

The Ac activation energy values of pure and mixed crystals are provided in table2. The variation of AC activation energy (ΔEac) with respect to frequency for NaxK(1-x)Br single crystals are shown in Figure 8. The activation energy obtained for pure and mixed crystals considered in present study at different frequencies do not show any systematic variation with Na composition in KBr lattices. Babu and Rao

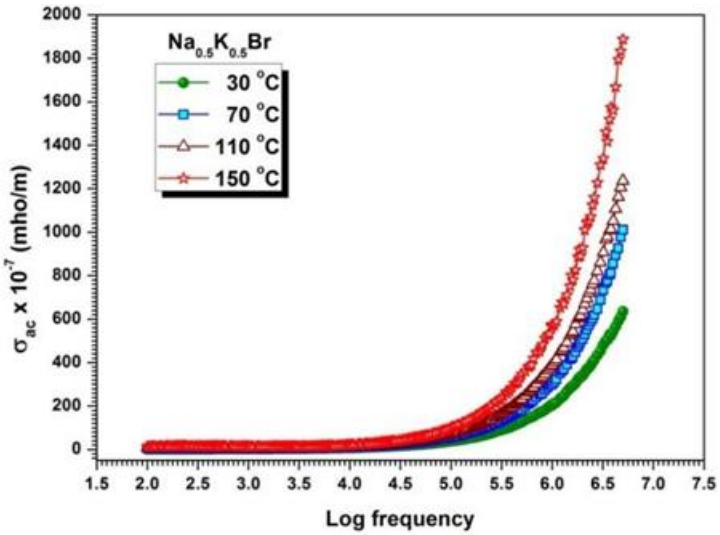



**Figure 7:** Variation of AC conductivity with frequency at various temperatures for Na0.5K0.5Br single crystal

From Figure 7, it reveals that the variation of ΔEac with respect to frequency for the pure and mixed crystal decreases linearly at low frequency region (up to 100 KHz) and increases or shows nonlinear variation at high frequencies (> 100 kHz). Similar behavior was observed for all the pure and mixed NaxK(1-x)Br single crystals. However Na rich (x = 0.8 and 0.9) and K rich (x = 0.1 and 0.2) mixed NaxK(1-x)Br single crystals the variation of ΔEac linearly decreases with frequency. This may be due to the influence of anharmonicity of lattice vibration is lesser than that of other composition crystals. When the composition of Na increases in KBr lattices, anharmonic contribution is enhanced in the crystal lattices similar behavior is observed when the composition of K increases in NaBr lattices too. Similar observations have been studied by Sathaiah for KCl-KBr and RbCl-RbBr mixed crystals. Sathaiah has shown that the anharmonic contribution is enhanced for intermediate compositions.

100 Hz,1 kHz, 10 kHz, 100 kHz, 1 MHz and 5 MHz frequencies.

**Table 5.4:** Calculated AC activation energy values of pure and mixed NaxK(1-x)Br single crystals for different frequencies

| Sl. No. | System | AC activation energy (ΔEac) for different frequencies | | | | | |
|---|---|---|---|---|---|---|---|
| | | 100 Hz | 1 kHz | 10 kHz | 100 kHz | 1 MHz | 5 MHz |
| 1 | KBr | 0.1175 | 0.1191 | 0.0754 | 0.0693 | 0.073 | 0.0687 |
| 2 | Na0.1K0.9Br | 0.1458 | 0.134 | 0.0972 | 0.0385 | 0.031 | 0.0377 |
| 3 | Na0.2K0.8Br | 0.1521 | 0.1392 | 0.062 | 0.0894 | 0.0862 | 0.089 |
| 4 | Na0.3K0.7Br | – | 0.0945 | - | 0.0509 | 0.0505 | 0.0545 |
| 5 | Na0.6K0.4Br | 0.1787 | 0.1347 | 0.0714 | 0.0538 | 0.0398 | 0.0338 |
| 6 | Na0.5K0.5Br | 0.1787 | 0.1493 | 0.0926 | 0.0866 | 0.0928 | 0.0953 |
| 7 | Na0.6K0.4Br | 0.2152 | 0.2053 | 0.1291 | 0.1275 | 0.1756 | 0.2085 |
| 8 | Na0.7K0.3Br | 0.2616 | 0.2265 | 0.1017 | 0.0617 | 0.0538 | 0.0537 |
| 9 | Na0.8K0.2Br | 0.1175 | 0.0998 | 0.0539 | 0.0398 | 0.0297 | 0.0255 |
| 10 | Na0.9K0.1Br | 0.2603 | 0.2348 | 0.1463 | 0.1268 | 0.1357 | 0.1382 |
| 11 | NaBr | 0.2777 | 0.2627 | 0.151 | 0.1232 | 0.1048 | 0.1005 |

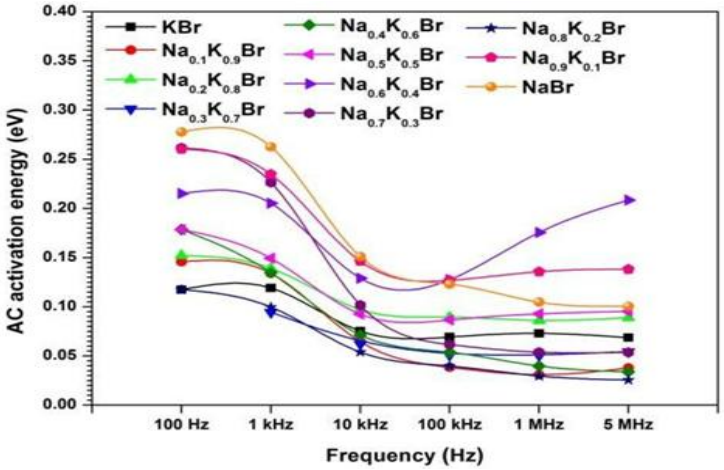



**Figure 7:** Variation of AC activation energy at various frequencies for NaxK(1-x)Br (x = 0.0, 0.1, 0.2, 0.3, 0.4,0.5, 0.6, 0.7, 0.8, 0.9 and 1.0) single crystal.

## IV. Conclusion

Binary mixed crystals of NaBr and KBr were grown from the aqueous solution by slow evaporation method. Compositions of the grown crystals were determined using the measured density and refractive index values by assuming the additive rule, satisfying for them. The dielectric constant and the dielectric loss increases with increase in temperature as observed by the previous authors. The AC conductivity increases with both temperature and frequency. But there is no systematic variation with composition for all the electrical parameters like dielectric constant, dielectric loss, AC conductivity and activation energy

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