

## Mechanical Properties of Pure ZnS Doped (NaCl)<sub>x</sub>(NaI)<sub>y-x</sub>(KI)<sub>1-y</sub> Ternary Mixed Crystals

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**Abstract:** *In the present research work, pure and ZnS doped ternary mixed crystals of alkali halides (NaCl)<sub>x</sub>(NaI)<sub>y-x</sub>(KI)<sub>1-y</sub> of different compositions were grown by slow evaporation technique and studies of microhardness and dielectric constant have been carried out. To estimate mechanical properties by using Vicker's microhardness measurement then the stability and measure the electrical properties by using DC and AC measurement due to growing crystal. The mechanical parameters like Meyer's work hardening co-efficient, Stiffness constant, Second order elastic constant, Young's modulus, Yield strength, Fracture toughness and Brittleness index were calculated by using available relations.*

**Keywords:** *Alkali halides, Activation energies, Density; Vicker's microhardness, Elastic stiffness constant, Second order elastic constant, Young's modulus, Yield strength, Fracture toughness, Brittleness index.*

### 1. INTRODUCTION

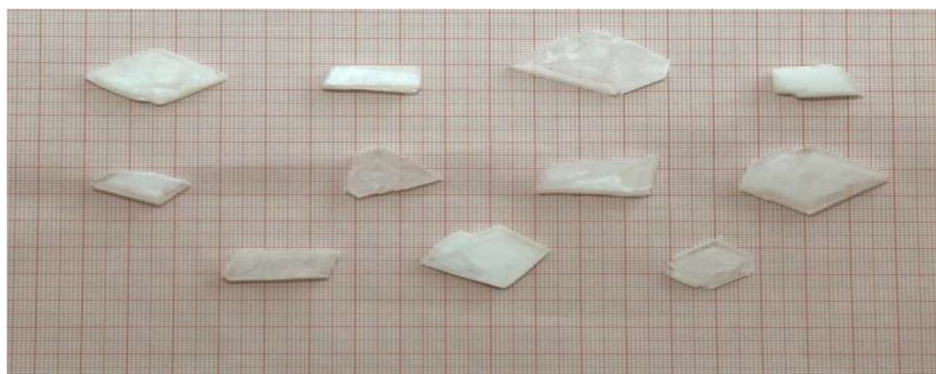
Alkali halide mixed crystals are widely used as laser window materials, neutron monochromators, infrared transmitters etc. The mixed and impurity added crystals of alkali halides are found to be harder than the end members and so they are more useful in these applications [1]. In view of this, it becomes necessary and useful to prepare binary and ternary mixed crystals regardless of miscibility problem and to characterize them by studying their physical properties. In order to investigate the nature of defects, dielectric loss and dielectric constant measurements have been done which showed that the mixed crystals are more defective, containing a high concentration of dislocations, low angle grain boundaries and vacancies as compared to the end products. The alkali halide crystals have importance in past six decades. They have been "model crystals" for testing many solid-state theories. This research work proposes the study of mechanical properties of mixed crystals. The study of mechanical properties is vital especially for real time applications. The mixed alkali halides

find their applications in optical, opto-electronic, and electronic devices. Alkali halide mixed crystals are of the completely disordered substitutional type [2]. Ionic conductivity measurements as a function of temperature have been done by on pure alkali halide crystals and impurity (anionic as well as cationic). Various defect parameters such as activation energy for migration, formation energy, etc. have been evaluated from these studies. The 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 [3]. The measurement of dielectric constant and loss as a function of frequency and temperature upto 400° C and KCl-KBr, RbCl-RbBr mixed crystals from the applied aspects. The static dielectric constant of the KCl-KBr system from Kramers–Kronig analysis of infrared reflectivity data [4]. The immersion method the dielectric constant of five mixed crystal systems, namely, KCl-KBr, KBr-KI, NaCl-NaBr, RbCl-RbBr, KCl-RbCl, and KI-RbI. Crystals were grown and characterized by density, refractive index, composition and lattice parameter determination, and DC and AC electrical measurements.

## 2. MATERIALS AND METHODS

### Growth of Sample Crystals

Analytical Reagent (AR) grade NaCl, NaI and KI doubly distilled water were taken for the growth. Supersaturated solutions of  $\text{NaCl}_x(\text{NaI})_{y-x}(\text{KI})_{1-y}$  were prepared for various values of x and y. The end member crystals were grown for comparison purposes. A crystals have grown as shown in the photograph. All the crystals are less transparent, white in color, hard and stable. The photographs of all the grown crystal are depicted in figure 1.



**Figure.1** Photograph of all the grown crystals

### **Microhardness Measurement**

The Vicker's micro hardness measurement were carried out using ZWICK 3212 hardness tester fitted with Vickers diamond pyramidal indenter. The Vicker's hardness test method consists of indenting the test material with a diamond indenter, in the form of pyramid with a square base and angle of  $136^\circ$  between opposite faces subjected to different loads. Total of three pure and four pure mixed, three doped end number ZnS added Ternary mixed crystals of  $(\text{NaCl})_x(\text{NaI})_{y-x}(\text{KI})_{1-y}$  were grown by slow evaporation technique from aqueous solution using Analog grade samples of NaClNaI and KI as the starting materials for the growth of the crystals. Grown crystals were cleaved and subjected to microhardness and dielectric constant measurements.

In order to determine the value of  $n$ , for the present system, the data is fitted by the least square procedure to the following equation

$$\Delta H_v = kc^n \quad (1)$$

The Vickers hardness number HV of Diamond Pyramid Number (DPN) is defined as defined as:

$$H_v = 2 P \sin(\alpha/2)/d^2 \quad (2)$$

Where,  $\alpha$  is the apex angle of the indenter ( $\alpha=136^\circ$ ).

$$d = L_1 + L_2/2 \quad (3)$$

The micro hardness is calculated using the expression

$$H_v = 1.8544 P/d^2 \text{ Kgmm}^{-2} \quad (4)$$

Where 'P' is the applied load in kg and 'd' is the average diagonal length of the Vicker's impression in mm after unloading. . The applied load was varied as 25, 50 and 100 g for a constant indentation period of 10 sec. All the indentation measurements were carried out on the freshly cleaved samples. The indented impressions were approximately square.

Micro hardness is defined as the ratio of the load applied to the surface area of the indentation The Meyers work hardening co-efficient 'n' can be determined by plotting  $\log P$  vs  $\log d$ , the variation of  $\log P$  vs  $\log d$  for the all ten samples is fig 3 for illustration. The slope of the best linear fit graph gives 'n' value. In the present study total of ten crystals (three pure mixed crystals, four mixed crystals, three doped end members) for various values of  $x$  and  $y$  were grown in identical conditions. The hardness of a material is defined as the resistance it offers to the

motion of dislocations, deformation or damage under an applied stress. Earlier microhardness was measured by Mohs scale [5] which is 10 for the diamond (hardest material). Vicker's indentation tests have been used by several investigators to study the glide deformations, isotropy or anisotropy, cracks, grain boundary hardening, irradiation effect and environment of dislocation mobility of various crystals [6]. The Vicker's hardness test method consists of indenting the test material with a diamond indenter, in the form of pyramid with a square base and angle of  $136^\circ$  between opposite faces subjected to different loads.

Hardness in Mohs scale ( $H_M$ ) can be determined from the Vicker's microhardness number ( $H_v$ ) in  $\text{kgmm}^{-2}$  by using the relation [7]

$$H_M = 0.675 (H_v)^{1/3} \quad (5)$$

It is noted that Mohs scale has no units. The Meyer's work hardening coefficient or Mayer's index number can be determined by from the Meyer's relation which relates the load an indentation diagonal length

$$P = Kd^n \quad (6)$$

$$\log P = \log K + n \log d \quad (7)$$

Where,  $k$  is material constant,  $n$  is a work hardening coefficient. Plot a curve between  $\log P$  and  $\log d$ . By taking  $\log P$  along x-axis,  $\log d$  along y-axis. The  $1/\text{slope}$  of the line from the best fit of the curve gives the work hardening coefficient ( $n$ ). Vicker's microhardness number ( $H_v$ ) can be expressed in GPa by multiplying  $H_v$  in  $\text{kgmm}^{-2}$  by a factor  $9.806 \times 10^{-3}$  [8].

The elastic stiffness constant ( $C_{11}$ ) for all the grown crystals have been estimated by using Wooster's empirical formula

$$C_{11} = H_v^{7/4} \quad (8)$$

And the second order elastic constant ( $C_{12}$ ) can be determined from the stiffness constant ( $C_{11}$ ) value by using the relation

$$C_{12} = \frac{3H_v - C_{11}}{2} \quad (9)$$

Where  $H_v$  is in GPa.

Young's Modulus ( $E$ ) and the Yield strength( $Y$ ) can be determined from the Vicker's microhardness number ( $H_v$ ) by using the relation

$$E = 81.9635 H_v \quad (10)$$

$$Y = \frac{Hv}{3} \quad (11)$$

Fracture toughness (kc) and Brittleness index (Bi) is determined by using the formula

$$kc = \frac{P}{\beta l^{\frac{3}{2}}} \quad (12)$$

Where P is the applied load, l is the crack length,  $\beta$  is the geometrical constant and  $\beta = 7$  for Vickers diamond indenter

$$B_i = \frac{Hv}{kc} \quad (13)$$

### 3. Results and discussion

#### Microhardness Studies

In the present case, hardness steadily increases then decreases for higher loads known as reverse indentation size effect (RISE). This behaviour of independence of microhardness at higher loads can be explained on the basis of slip planes producing dislocations. At small loads, the indenter penetrates only the surface layers, dislocations are nucleated in slip planes close to the indented surface. NaCl, NaI, KI are an ionic compound which consists of Sodium cation ( $Na^+$ ), Potassium cation ( $K^+$ ), Chloride anion ( $Cl^-$ ) Iodide anion ( $I^-$ ). Pauling ionic radii in  $\text{Å}^0$  and the ionic size of ions such as sodium ( $Na^+$ ), potassium ( $K^+$ ), chloride ( $Cl^-$ ) Iodide ( $I^-$ ) atoms are  $0.95 \text{ Å}^0, 1.33 \text{ Å}^0, 1.81 \text{ Å}^0$  and  $2.16 \text{ Å}^0$  respectively [10].

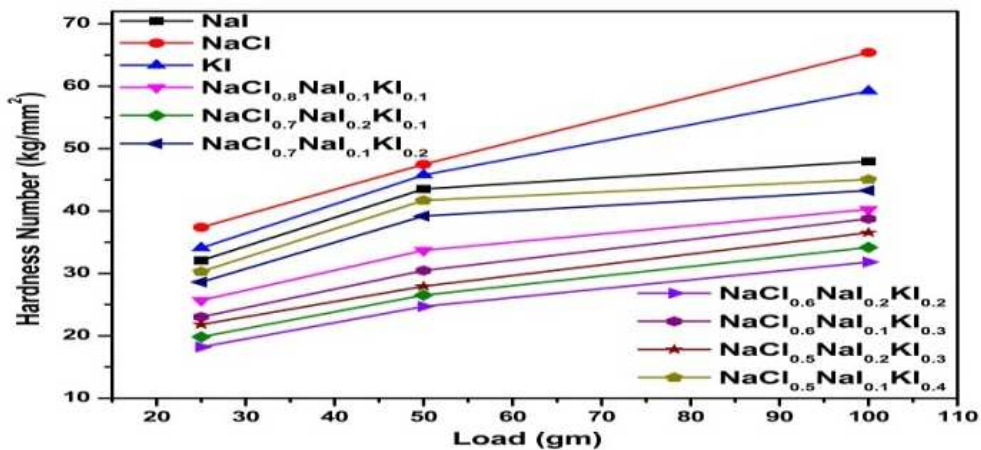


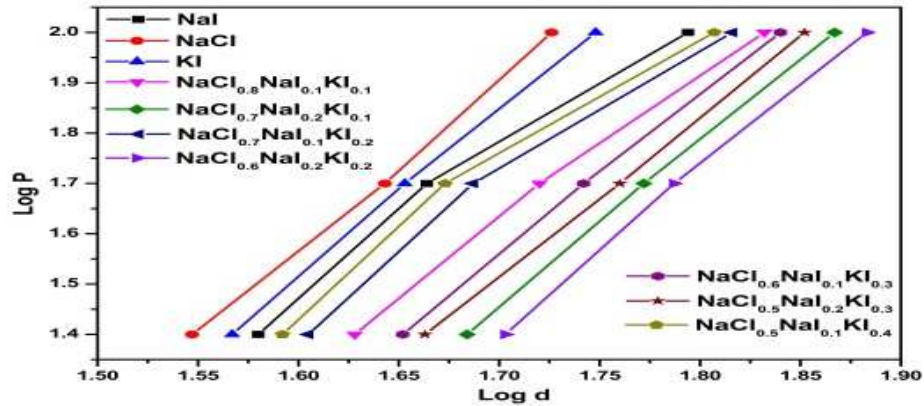
Figure2. Variation of hardness value mixed crystals.

The variation of micro hardness with composition for  $(\text{NaCl})_x(\text{NaI})_{y-x}(\text{KI})_{1-y}$  was shown in Figure 1. If  $n > 1.6$ , the materials belong to soft category, if  $n < 1.6$ , the materials belong to hard category. In the present work it is found that the ‘n’ values of all the grown crystals are greater than 2 So, if that should they belong to soft materials category [Onitch]. The nonlinear variation is due to the crystal imperfections and size effect. The imperfections may be dislocations, vacancies, low angle grain boundaries etc. It reveals that the mixed alkali halides are harder than the pure alkali halides. Also the hardness number of ZnO added mixed are greater than the pure mixed crystals. This difference is ionic size developed lattice strains in mixed crystals. This lattice strain may be responsible for the formation of various types of imperfections. Vicker’s hardness number for different loads in  $\text{kgmm}^{-2}$ , in GPa and in Mhos scale along with work hardening coefficient of all the grown crystals are provided in table 1

**Table 1. Vicker’s microhardness number  $H_v$  ( $\text{kgmm}^{-2}$ ) ,log d for pure and oped crystals.**

System	log d			$H_v(\text{kg/mm}^2)$		
	25	50	100	25	50	100
Pure						
NaCl	1.547	1.643	1.726	37.39	47.78	65.42
NaI	1.567	1.653	1.748	34.08	45.77	59.22
KI	1.580	1.664	1.794	32.07	43.53	47.95
$(\text{NaCl})_{0.5}(\text{NaI})_{0.1}(\text{KI})_{0.4}$	1.592	1.673	1.807	30.29	41.71	45.05
$(\text{NaCl})_{0.6}(\text{NaI})_{0.1}(\text{KI})_{0.3}$	1.652	1.742	1.840	23.03	30.46	38.75
$(\text{NaCl})_{0.7}(\text{NaI})_{0.1}(\text{KI})_{0.2}$	1.605	1.687	1.816	28.62	39.19	43.27
$(\text{NaCl})_{0.8}(\text{NaI})_{0.1}(\text{KI})_{0.1}$	1.628	1.720	1.832	25.69	33.70	40.26
$(\text{NaCl})_{0.5}(\text{NaI})_{0.2}(\text{KI})_{0.3}$	1.663	1.760	1.853	21.81	27.94	36.54
$(\text{NaCl})_{0.6}(\text{NaI})_{0.2}(\text{KI})_{0.2}$	1.703	1.787	1.883	18.20	24.70	31.81
$(\text{NaCl})_{0.7}(\text{NaI})_{0.2}(\text{KI})_{0.1}$	1.684	1.772	1.867	19.83	26.52	34.16
Doped	25	50	100	25	50	100
NaCl	1.550	1.626	1.743	46.28	51.79	60.46
NaI	1.527	1.657	1.766	40.87	45.02	54.45
KI	1.550	1.675	1.788	36.85	41.36	49.24
$(\text{NaCl})_{0.5}(\text{NaI})_{0.1}(\text{KI})_{0.4}$	1.730	1.773	1.876	16.20	26.35	32.77
$(\text{NaCl})_{0.6}(\text{NaI})_{0.1}(\text{KI})_{0.3}$	1.678	1.755	1.764	20.44	28.60	31.21
$(\text{NaCl})_{0.7}(\text{NaI})_{0.1}(\text{KI})_{0.2}$	1.558	1.692	1.795	35.53	38.22	47.69
$(\text{NaCl})_{0.8}(\text{NaI})_{0.1}(\text{KI})_{0.1}$	1.710	1.796	1.836	17.64	23.75	39.47
$(\text{NaCl})_{0.5}(\text{NaI})_{0.2}(\text{KI})_{0.3}$	1.636	1.744	1.822	24.81	30.14	42.12
$(\text{NaCl})_{0.6}(\text{NaI})_{0.2}(\text{KI})_{0.2}$	1.615	1.721	1.791	27.25	33.50	48.47
$(\text{NaCl})_{0.7}(\text{NaI})_{0.2}(\text{KI})_{0.1}$	1.656	1.758	1.865	22.61	28.31	34.58

The hardness number increases as the load increases. The hardness number of the mixed crystals is found to be greater than those for end member crystals. It reveals that the mixed alkali halides are harder than the pure alkali halides [11]. The variation of log P versus log d for all the grown crystals is shown in figure 2.



**Figure.3** Variation of log P vs log d for all grown Crystals

For the mixed crystals log p and log d value of the pure NaCl, NaI and KI crystals increased simultaneously. The  $(\text{NaCl})_{0.6}(\text{NaI})_{0.2}(\text{KI})_{0.2}$  very high value and also  $(\text{NaCl})_{0.5}(\text{NaI})_{0.1}(\text{KI})_{0.4}$ ,  $(\text{NaCl})_{0.6}(\text{NaI})_{0.1}(\text{KI})_{0.3}$ ,  $(\text{NaCl})_{0.8}(\text{NaI})_{0.1}(\text{KI})_{0.1}$ ,  $(\text{NaCl})_{0.5}(\text{NaI})_{0.2}(\text{KI})_{0.3}$ ,  $(\text{NaCl})_{0.7}(\text{NaI})_{0.2}(\text{KI})_{0.1}$  increased linearly. But the  $(\text{NaCl})_{0.7}(\text{NaI})_{0.1}(\text{KI})_{0.2}$  slightly increased then suddenly increase high value. We also draw the Figure 2, a graph is plotted between log p vs log d for all mixed crystals. The theoretical and experimental value of micro hardness  $H_v$  are same. The graph was plotted between the system along the X-axis and the hardness number  $H_v$  along Y-axis. To know hardness of the materials, a graph of log P versus log d is plotted.

**Table 2: Micro hardness number in kg/mm<sup>2</sup> and GPa, Mohr Scale and Work hardening coefficient for all grown crystal**

System	Hv kg/mm <sup>2</sup>			Hv (GPa)			H <sub>M</sub>			n
	25	50	100	25	50	100	25	50	100	
NaCl	37.	47.	65.	0.3	0.4	0.6	2.2	2.4	2.7	3.34
NaI	39	78	42	67	69	42	54	46	16	61
KI	34.	45.	59.	0.3	0.4	0.5	2.1	2.4	2.6	2.76
(NaCl) <sub>0.5</sub>	08	77	22	14	49	81	86	11	27	12
(NaI) <sub>0.1</sub> (KI) <sub>0.4</sub>	32.	43.	47.	0.3	0.4	0.4	2.1	2.3	2.4	3.31
(NaCl) <sub>0.6</sub> (NaI) <sub>0.1</sub> (KI) <sub>0.3</sub>	07	53	95	34	27	70	42	71	49	22
(NaCl) <sub>0.7</sub> (NaI) <sub>0.1</sub>	29	71	05	97	09	42	02	38	99	53
(KI) <sub>0.2</sub>	23.	30.	38.	0.2	0.2	0.3	1.9	2.1	2.2	3.18
(NaCl) <sub>0.8</sub> (NaI) <sub>0.1</sub>	03	46	75	26	99	80	18	06	81	96
(KI) <sub>0.1</sub>	28.	39.	43.	0.2	0.3	0.4	2.0	2.2	2.3	2.79
(NaCl) <sub>0.5</sub> (NaI) <sub>0.2</sub>	62	19	27	81	84	24	62	90	67	73
(KI) <sub>0.3</sub>	25.	33.	40.	0.2	0.3	0.3	1.9	2.1	2.3	2.93
(NaCl) <sub>0.6</sub> (NaI) <sub>0.2</sub>	69	70	26	52	30	95	90	78	11	18
2(KI) <sub>0.2</sub>	21.	27.	36.	0.2	0.2	0.3	1.8	2.0	2.2	3.17
(NaCl) <sub>0.7</sub> (NaI) <sub>0.2</sub>	81	94	54	14	74	58	84	46	37	39
2(KI) <sub>0.1</sub>	18.	24.	31.	0.1	0.2	0.3	1.7	1.9	2.1	3.32
	20	70	81	78	42	12	74	64	36	84
	19.	26.	34.	0.1	0.2	0.3	1.8	2.0	2.1	3.27
	83	52	16	94	60	35	25	11	88	71

The Mohs scale also indicates that the crystals grown in the present study. For mixed the hardness value gets increased when the load is increased. The mechanical strength of the ZnS doped crystals is weak. It is found that the hardness number increases with increase of load and it varies non-linearly with bulk composition as shown in figure2. Non-linear variation may also be due to the presence of imperfections like vacancies, impurity-vacancy pairs, dislocation, low angle boundaries etc. The Stiffness constant, Second order elastic constant, Young's modulus, Yield strength, Fracture toughness and Brittleness index along with the crack length for the load of 100g are given in the table2.

**Table 3. Average diagonal length, Stiffness constant, second order elastic constant, Young's modulus, Yield strength, Fracture toughness and Brittleness index along with the crack length for the load of 100g of all the grown crystals.**

System	d	l ( $\mu$ m)	Hv (GPa )	E (GP a)	Y (GP a)	C <sub>11</sub> (GPa)	C <sub>12</sub> ( GPa)	K <sub>c</sub> /( $\mu$ m) <sup>3/2</sup>	B <sub>i</sub> ( $\mu$ m) <sup>-1/2</sup>
NaCl	53.	28.	0.64	52.	0.2	0.460	0.73	0.094	6.83
NaI	24	54	2	62	14	0.387	3	0.086	0
KI	55.	30.	0.58	47.	0.1	0.267	0.67	0.074	6.75
(NaCl) <sub>0.5</sub>	96	12	1	62	94	0.240	8	0.078	6
(NaI) <sub>0.1</sub> (KI) <sub>0.4</sub>	62.	33.	0.47	38.	0.1	0.184	0.57	0.068	6.35
(NaCl) <sub>0.6</sub>	18	41	0	52	57	0.223	2	0.676	1
(NaI) <sub>0.1</sub> (KI) <sub>0.3</sub>	64.	32.	0.44	36.	0.1	0.197	0.54	0.071	5.66
(NaCl) <sub>0.7</sub>	16	23	2	23	47	0.166	3	0.067	7
(NaI) <sub>0.1</sub> (KI) <sub>0.2</sub>	69.	35.	0.38	31.	0.1	0.130	0.47	0.058	5.58
(NaCl) <sub>0.8</sub>	18	28	0	15	27	0.148	8	0.062	8
(NaI) <sub>0.1</sub> (KI) <sub>0.1</sub>	65.	32.	0.42	34.	0.1		0.52		5.57
(NaCl) <sub>0.5</sub>	46	95	4	75	41		5		9
(NaI) <sub>0.2</sub> (KI) <sub>0.3</sub>	67.	34.	0.39	32.	0.1		0.49		5.56
(NaCl) <sub>0.6</sub> (NaI) <sub>0.2</sub> (KI) <sub>0.2</sub>	86	18	5	38	32		4		3
(NaCl) <sub>0.7</sub> (NaI) <sub>0.2</sub> (KI) <sub>0.1</sub>	71.	35.	0.35	29.	0.1		0.45		5.34
	24	84	8	34	19		4		3
	76.	39.	0.31	25.	0.1		0.40		5.37
	35	21	2	57	04		3		9
	73.	37.	0.33	27.	0.1		0.42		5.40
	68	41	5	46	68		9		3

Among the investigated combinations, atoms are more tightly bound to their neighboring atoms in the mixed crystals (NaCl)<sub>0.5</sub>(NaI)<sub>0.1</sub>(KI)<sub>0.4</sub>. Among the investigated samples, the sample (NaCl)<sub>0.5</sub>(NaI)<sub>0.1</sub>(KI)<sub>0.4</sub> is answering for a higher value of C<sub>11</sub> for all the loads. It is observed that all the above parameters determined in the present study increase with increase of load and vary non-linearly with bulk composition of mixed crystals. They are found to be high for the average diagonal length and crack length (NaCl)<sub>0.6</sub>(NaI)<sub>0.2</sub>(KI)<sub>0.2</sub> mixed crystal when it is compared to the other mixed crystals, and it is low for the average diagonal length and crack length (NaCl)<sub>0.5</sub>(NaI)<sub>0.1</sub>(KI)<sub>0.4</sub> mixed crystal.

#### 4. CONCLUSIONS

Good optically transparent ternary mixed alkali halide crystals  $(\text{NaCl})_x(\text{NaI})_{y-x}(\text{KI})_{1-y}$  of different compositions were grown by slow evaporation from aqueous solution. Microhardness has varied non-linearly with composition. Microhardness is more in ternary mixed crystals compare the pure and binary mixed crystals. The value of the dielectric constant in mixed  $(\text{NaCl})_x(\text{NaI})_{y-x}(\text{KI})_{1-y}$  crystals is more than the pure components. Dielectric constant varies non-linearly with composition. Large value of dielectric constant at low frequencies in the present study confirms the purity of the sample. The stiffness constant value indicate that the bonding neighbouring atoms increases with increased NaCl constant and also indicate that for doped mixed crystals , the bonding is very low. Vickers microhardness study on NaClNaI KI crystal reveals that the hardness number increase with increase of load and after 50gm it decreases with increase of load. Meyer's index n is around 1.6 suggesting that grown crystal falls under the moderately soft material category. All the mechanical parameters determined in the present study are also varies non-linearly with bulk composition.

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