Comparative study of Thermo luminescence, Optical absorption and Micro hardness in $(KCl)_{0.9-x}$ $(KBr)_x$ $(NaI)_{0.1}$ doped with lithium sulphate

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ABSTRACT: In the present study $(KCl)_{0.9-x}$ $(KBr)_x$ $(NaI)_{0.1}Li_2So_{4\ (0.05)}$ crystals of different compositions are grown by two methods; Melt and Slow Evaporation Method using AR grade chemicals and composition was confirmed by EDAX report. Crystals are irradiated with ^{60}Co source and comparative study of Optical absorption, Thermo luminescence and Micro hardness properties of these two sets of crystal has been carried out. From the result, shift in TL peaks and optical absorption peaks is observed with change in composition and non-linear variation of Vicker hardness constant with composition is resulted.

Keywords: Thermolumenescence; optical absorption; Micro hardness; Melt method; Evaporation method.

1. INTRODUCTION

Alkali atoms and halogens combine to form a very interesting group of compounds called alkali halides which are the core of material physics. Because of their structure and chemical properties, they are first materials of choice for experimental and theoretical studies [1]. They are the model crystals for scaling properties like Debye's temperature, color centre parameters like optical absorption, thermo luminescence i.e., concentration of defects, and hardness [2]. Since few decades they emerged as useful materials in many devices like laser window materials, preferably poly crystalline alkali halides are used from last decades for this purpose [3, 4]. Potassium halides (KBr-KCl) are used as neutron monochromators [5]. LiF and NaCl are used as X-ray monochromator. Armington and et al.,succeed to increase the mechanical strength and stability of alkali halides by mixing the alkali halides and by doping methods. These crystals are found to have better stability and mechanical strength than their end products and are proved to be much suitable for optoelectronic device applications [1, 4].

The physical property of mixed and doped alkali halides varies linearly in some crystals and non-linearly in some other crystals [6, 7]. It is just like having a new set of crystals, if we grow the crystals with new compositions by knowing the trend in composition dependent property [8]. The defect formed due to the composition will give rise many optical properties like thermo luminescence and optical absorption [8-11]. Hence, it is necessary and useful to grow mixed crystals and characterize them.

R.W. Phol et al., worked on Color centers which is responsible for the Optical absorption in a normally transparent spectral region [12]. Mahadevan et al., studied about ternary alkali halides, they proved addition of KBr to NaCl-KCl has increased the stability of the crystal [12, 13]. The Effect of Au+ doping on TL intensity and absorption coefficient in KBr–NaI mixed crystals doped with gold was reported by A. Freund et al., [14]. R. K. Gartia and V.V.Ratnam have justified the role of ionic size on micro hardness, lesser rate of hardening in mixed crystals due to irradiation [15, 16]. R. Ananda Kumari has reported non-linear variation of colour centre peak position and half band width of F-centre with composition $(KCl)_{1-x}$ $(KBr)_{x}$ mixed alkali halide crystals doped with gold [6]. G. Maruthi and R. Chandramani have reported the increase in hardness with the composition [7]. G.S. Bangaru reports the TL study suggests the presence of low concentration of Ce^{3+} ions which reduces the TL efficiency with respect to pure KCL samples [17]. These studies raise the interest and curiosity on alkali halide crystals. Hence the comparative study of these crystals grown in different methods is done in view of the properties like thermo luminescence, optical absorption and micro hardness.

2. MATERIALS AND METHODS:

2.1. Material preparation:

Study has been made by synthesising one set of $KCl_{[0.9-x]}$ $KBr_{[x]}$ $NaI_{[0.1]}$ doped with $Li_2SO_4{}_{[0.05]}$crystals for x = 0.1 to 0.8 composition by melt method and another set by evaporation method [18]. AR grade crystals are used as startingmaterials. For this, $Li_2SO_{4[0.05]}$ doped $KCl_{[0.9-x]}$ $KBr_{[x]}$ $NaI_{[0.1]}$ solid solution is prepared as explained by Mahadevan [19] with x = 0.1 to 0.8. These three salts along with the dopant mixed together so that they would melt together uniformly. The mixture was then furnace melted in a silica crucible in order to obtain the crystals by melt method [20, 21]. Another set of same composition was kept for slow evaporation at room temperature. With proper nucleation crystals were formed by slow

evaporation method. Then both set of crystals are tested for the composition by EDAX. The samples are irradiated at room temperature using a ^{60}Co source with beam energy 1.17MeVand 1.27MeVto a dosage of 15 Mrads. Then subjected to thermo luminescence and optical absorption studies and the non- irradiated crystals were subjected to Micro hardness study.

2.2. THERMO LUMINESCENCE STUDY:

γ- Irradiated crystals under study were subjected to the TL studies. The samples were heated at a uniform rate from room temperature to the high temperature and the thermo luminescent intensity – glow peaks emitted by the sample are recorded using pc controlled experimental set up, Nucleonix Thermo Luminescence Instrument, where the radiation was converted to electric current by a 931A photomultiplier tube. [15, 19]

2.3. OPTICAL ABSORPTION:

2.3.1. Optical absorption study of crystals grown by Melt method.

Pre- irradiated crystals grown by melt method were used to study the optical absorption spectra. Hitachi U-3200 spectrophotometer ranging 200-800nm at room temperature in which the accuracy of 0.001 absorption coefficient can be expected is used for this purpose [15, 19].

2.3.2 Diffused Reflectance study of crystals grown by Evaporation method

DRSis an effective method to study the optical absorption in variety of materials. LAB INDIAUV-VIS Spectrophotometer 3092 in the range 190 nm to 900 nm at room temperature, working on dual beam optical absorption instruments is used for DR studies. The corresponding absorption spectra of the studied reflectance can be obtained using K-M equation [20, 22]

$$\frac{K}{S} = F(R_\infty) = \frac{(1-R_\infty)^2}{2} \quad \text{---- (3)}$$

where,$F(R_\infty)$ for any reflectance is termed Kubelka- Munk Function for R.S and K are thescattering and absorption cross sections constants. R_∞ is the total diffused reflectance.

2.4. MICRO HARDNESS:

Micro hardness is an important solid-state property which determines the mechanical strength of material as it measures the resistance offered by the lattice to the motion of dislocations. One can determine the stress required to produce plastic flow in the materials. Indentation method is used to test the hardness and it is done by using Micro Vicker hardness tester (Zwick UK 3212) for the crystals grown by melt method and by using Clemex Vickers hardness tester for the crystals grown by Evaporation method [22].

Vicker hardness number H_v can be calculated using the expression,

$$H_y = \frac{1.8544P}{d^2} \quad \text{kg mm2} \qquad \text{---- (4)}$$

where, P - applied load in kg, d - diagonal length of Vicker impression after unloading.Mayer's work hardening coefficient (n) is related with applied load and diagonal length by the Mayer's law,

$$P = cd^n \qquad \text{---- (5)}$$

where 'c' is the material constant.

3. RESULT AND DISCUSSIONS:

3.1: EDAX report:

The elemental composition of the crystals $KCl_{[0.9-x]}KBr_{[x]}NaI_{[0.1]}Li_2SO_{4\,[0.05]}$ crystals for x = 0.1to0.8 concentration, grown by melt method and sow evaporation method are made by EDAX. The results obtained were found to be in good inagreement with the calculated values. The report of X= 0.1 and X= 0.7, were shown in Figure 1 and Figure2, corresponding results are tabulated in Table 1andTable 2. The crystals grown by melt method were opaque and hard,whereas the crystals grown by slow evaporation method is transparent and brittle.

3.2. Thermo Luminescence Study:

TL technique is successfully used to study the dynamics of electrons and holes associated with point defects such as colour centers. Freshly cleaved, pre-excited crystals are subjected to TL study. The crystals grown by melt method answered for the characteristic glow peaks. Middle concentration crystals showed glow peaks nearly at 531K to 647K. As expected, glow peak position is shifted towards lower wavelength side by the effect Br^- ions as observed by the earlier studies [23] and the result is shown in Figure 3. But the crystals grown by slow evaporation method without showing any markable Thermo Luminescence get splitted at around 675K and are show in Figure 4.

3.3. Optical Absorption Study:

Figure 5 shows the Optical Absorption of the crystals under study. Usually, alkali halide crystals are transparent in UV and visible region, but doping and irradiation causes absorption in these regions. This fact is very well revealed in the present study. The crystals grown by melt method, shown maximum absorption in middle concentration. Example for X = 0.3 revealed peak absorption i.e., more than 4.13 cm^{-1} at 588 nm and 4.34 cm^{-1} at 766 nm, and even the concentration x = 0.4 showed absorption 3.662 cm^{-1} at 732 nm more than 4.152 cm^{-1} at 502 nm and 5 cm^{-1} at 490 nm. X = 0.5 also shown maximum absorption of 5cm^{-1} at 204 nm and 465 nm and 3.0693cm^{-1} for 746nm. In the present study, overlapped or broad band graphs are obtained, instead of usual bell shape. But the result is obeying the previous observation of Tsebin and Jacobs [23]. Absorption in UV-region is attributed to the presence of KBr, but the slight shift observed in wavelength in this region is the due to dopant Li_2So_4. Absorption in visible region is due to F-centres formed by irradiation of the crystals as expected earlier [25]. The lower and higher concentrations are not shown any marked absorption. Optical absorption in the crystals grown by slow evaporation method has been studied by using diffused reflectance. These crystals do not exhibit any F-centre absorption, optical absorption is clearly in UV region at low wavelength side which reveals effect of KBr and dopant Li_2So_4. The result is shown in the Figure 6.

3.4. Micro Hardness Study:

Freshly cleaved crystals are subjected to micro hardness test by applying the loads between 10 to 100 gms. A nonlinear variation is found with Vickers hardness number and the load, the results are shown in Figure 7 and Figure 8. The Mayer's work hardening coefficient (n) can be estimated from the slope of best linear fitted graph of Log (p) verses Log (d) which is given in Figure 9 and Figure 10. The value of 'n' has been mentioned in Table 3.

From equation (4) and equation (5);

$$H_V = kp^{\frac{n-2}{n}} \qquad \text{---- (6)}$$

where the constant k= $1.8544c^{1+n/2}$, above equation indicates that H_e should decrease with load for n < 2, which is very well revealed in most of the compositions, for the crystals grown by evaporation method but the crystals grown by melt method shown nonlinear variation of $H_v$with p [26]. According to Onitsch and Hanneman 'n' should lie between 1.0 And 1.6 for hard materials and above 1.6 for soft ones [27]. The 'n' values observed in the present study are more than 1.6 which indicates that all the crystals grown by evaporation method belong to

soft materials category. Thus, the experimental results observed in the present study follow the normal ISE trend. Indentation size effect is the phenomenon of dependence of micro hardness of a solid on the applied load at low level of testing load.

The elastic stiffness constant (C_{11}) for various compositions as well as for different loads have been estimated using Wooster's empirical formula $C_{11}= H_v^{7/4}$. The C_{11} values are given in Table 4(a) and Table 4(b). These values give an idea about the tightness of bonding between neighbouring atoms. The present study shows comparable difference of C_{11} value between the crystals grown by melt method and slow evaporation method, crystals grown by evaporation method shown higher stiffness value [28].

CONCLUSION:

The present study revealed that $(KCl)_{0.9-x}$ $(KBr)_x$ $(NaI)_{0.1}$ doped with lithium sulphate crystals (where X=0.1 to 0.8), are greatly influenced by the method of synthesis and crystal compositions are confirmed by EDAX report. The crystals grown by Melt method have answered for the characteristics TL glow peaks at 531K to 647 K. The presence of Br ions shifted the glow peak position to the lower wavelength side. Optical absorption study of the crystals is again proved the presence of KBr and Li_2So_4 attributes to absorption in UV region and irradiation forms F centers which are responsible for optical absorption in visible region. The crystals grown by evaporation method are resulted higher Vicker hardness value compare to the other set of crystals. Correspondingly they have higher elastic stiffness constant, which indicates the tight bonding between the atoms.

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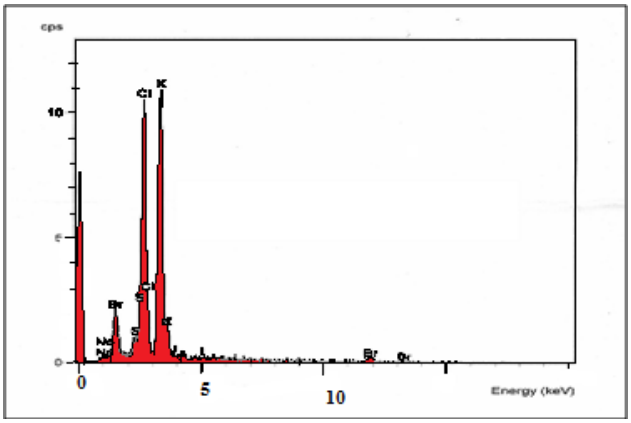

Figure 1. EDAX report of $(KCl)_{0.8}(KBr)_{0.1}(NaI)_{0.1}(Li_2SO_4)_{0.05}$ crystal grown by Evaporation method.

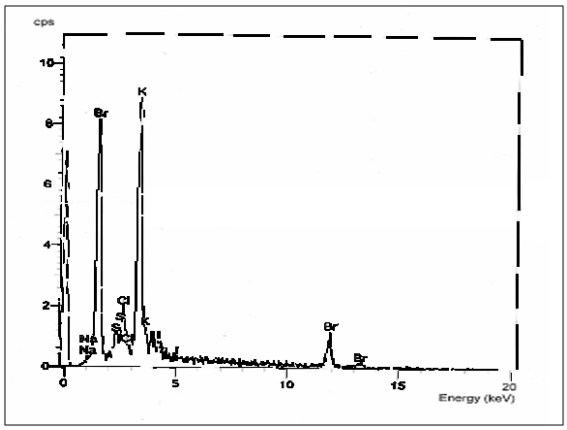

Figure 2. EDAX report of $(KCl)_{0.1}(KBr)_{0.7}(NaI)_{0.1}(Li_2SO_4)_{0.05}$crystal grown by Melt method.

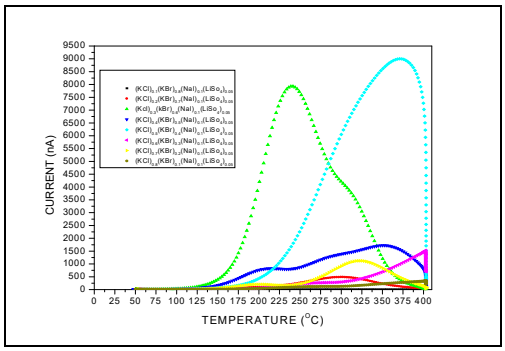

Figure3. Thermo luminescence glow curve of $(KCl)_{0.9-x}(KBr)_{x}(NaI)_{0.1\ ternary}$ crystal doped with $(LiSo_4)_{0.05}$ grown by Melt method irradiated with a dosage of 15Mrad.

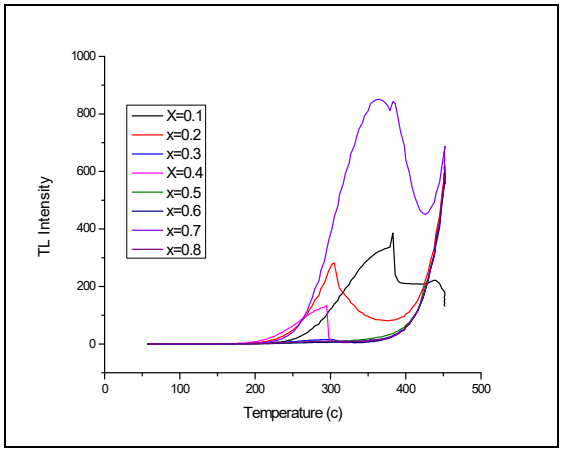

Figure4. Thermo luminescence glow curve of (KCl) $_{0.9-x}$ (KBr) $_x$ (NaI) $_{0.1\ ternary}$ crystal doped with $(LiSo_4)_{0.05}$ grown by Evaporation method irradiated with a dosage of 15Mrad.

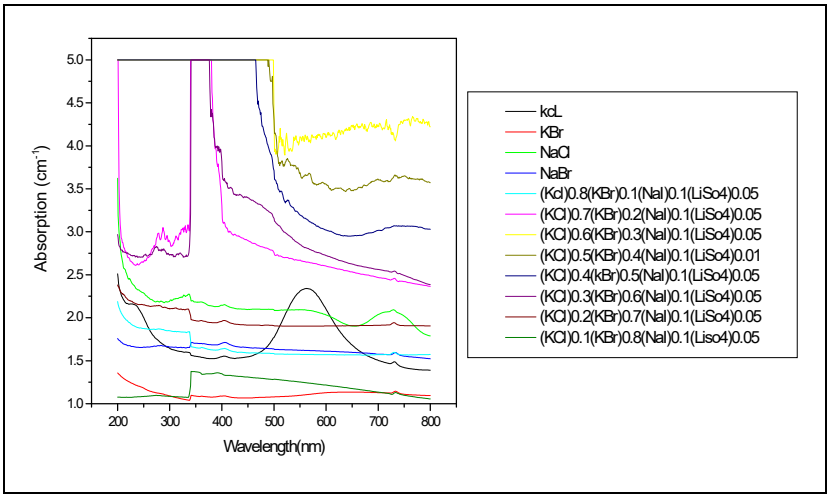

Figure.5. Optical Absorption Spectra of KCl, KBr, NaCl, NaBr, $(KCl)_{9-x}$ $(KBr)_x$ $(NaI)_{0.1}$ ternary crystal doped with $(LiSo_4)_{0.05}$ grown by melt method irradiated with a dosage of 15 Mrad.

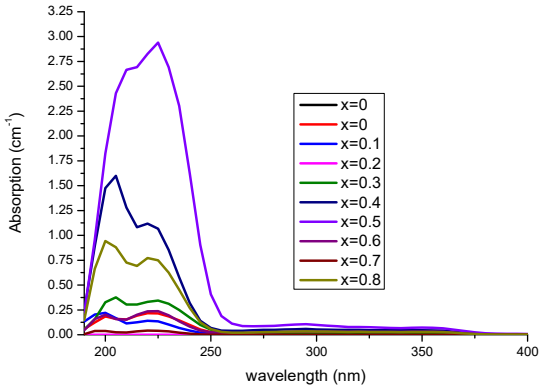

Figure 6: Optical absorption of (KCl) $_{0.9-x}$ (KBr) $_x$ (NaI) $_{0.1}$ $(Li_2So_4)_{0.05}$ Crystal Grown by Evaporation method irradiated with a dosage *of* 15Mrad.

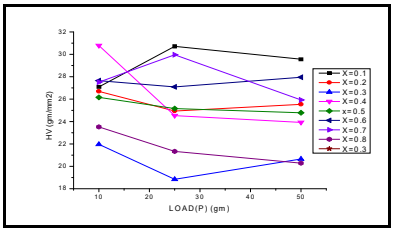

Figure 7: Micro hardness of $(KCl)_{0.9-x}$ $(KBr)_x$ $(NaI)_{0.1}(Li_2So_4)_{0.05}$ crystals grown by Evaporation method

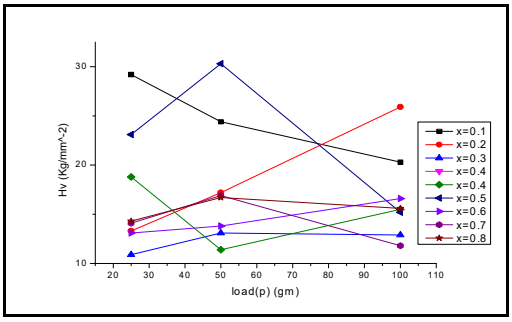

Figure 8: Micro hardness of $(KCl)_{0.9-x}$ $(KBr)_x$ $(NaI)_{0.1}(Li_2So_4)_{0.05}$ crystals grown by Melt method

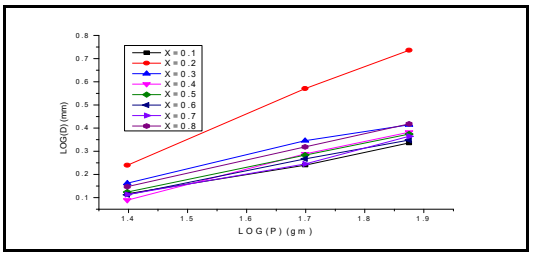

Figure 9: Log (d) verses of Log (p) graph $(KCl)_{0.9-x}$ $(KBr)_x$ $(NaI)_{0.1}(Li_2So_4)_{0.05}$ crystals grown by Evaporation method

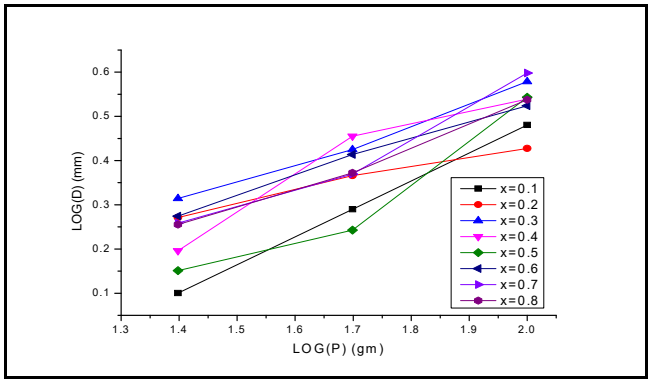

Figure 10: Log (d) verses of Log (p) graph $(KCl)_{0.9-x}$ $(KBr)_x$ $(NaI)_{0.1}(Li_2So_4)_{0.05}$ crystals grown by Melt method

Table 1: EDAX report of $(KCl)_{0.8}(KBr)_{0.1}(NaI)_{0.1}(LiSo_4)_{0.05}$ crystal grown by Evaporation method irradiated with a dosage of 15 Mrad.

Elements	Experimental Values	
	Weight %	Atomic%
Na (K)	0.93	1.66
S (K)	2.32	2.98
Cl (K)	33.02	38.39
K (K)	44.76	47.20
Br (K)	18.87	9.74
I (L)	0.10*	0.03*
Total	100.00	100.00

Table 2: EDAX report of $(KCl)_{0.2}(KBr)_{0.7}(NaI)_{0.1}(LiSo_4)_{0.05}$ crystal grown by Melt method irradiated with a dosage of 15 Mrad.

Elements	Experimental Values	
	Weight %	Atomic%
Na (K)	1.31	3.21
S (K)	2.58	4.53
Cl (K)	5.09	8.08
K (K)	29.96	43.12
Br (K)	53.62	37.76
I (L)	7.44	3.30
Total	100.00	100.00

Table3: work hardening function.

Composition	Value of 'n' for the crystals grown by	
	Melt method	Evaporation method
$(KCl)_{0.8}(KBr)_{0.1}(NaI)_{0.1}(Li_2SO_4)_{0.05}$	1.58448	2.1989
$(KCl)_{0.7}(KBr)_{0.2}(NaI)_{0.1}(Li_2SO_4)_{0.05}$	3.79525	0.9560
$(KCl)_{0.6}(KBr)_{0.3}(NaI)_{0.1}(Li_2SO_4)_{0.05}$	0.234	1.86268
$(KCl)_{0.5}(KBr)_{0.4}(NaI)_{0.1}(Li_2SO_4)_{0.05}$	-----	1.61240
$(KCl)_{0.4}(KBr)_{0.5}(NaI)_{0.1}(Li_2SO_4)_{0.05}$	-0.2085	1.90519
$(KCl)_{0.3}(KBr)_{0.6}(NaI)_{0.1}(Li_2SO_4)_{0.05}$	0.15359	2.0114
$(KCl)_{0.2}(KBr)_{0.7}(NaI)_{0.1}(Li_2SO_4)_{0.05}$	-0.06671	1.9321
$(KCl)_{0.1}(KBr)_{0.8}(NaI)_{0.1}(Li_2SO_4)_{0.05}$	0.1308	1.7617

Table 4(a): Elastic stiffness constant for the crystals grown by Melt method

Composition	Value of Elastic stiffness constant C_{11} in Pa		
	Load(p) 25g	Load(p) 50g	Load(p) 100g
$(KCl)_{0.8}(KBr)_{0.1}(NaI)_{0.1}(Li_2SO_4)_{0.05}$	5E+09	1E+09	3.6E+08
$(KCl)_{0.7}(KBr)_{0.2}(NaI)_{0.1}(Li_2SO_4)_{0.05}$	2E+07	1E+08	2E+09
$(KCl)_{0.6}(KBr)_{0.3}(NaI)_{0.1}(Li_2SO_4)_{0.05}$	5E+06	2E+07	1.5E+07
$(KCl)_{0.5}(KBr)_{0.4}(NaI)_{0.1}(Li_2SO_4)_{0.05}$	2E+08	6E+06	5.4E+07
$(KCl)_{0.4}(KBr)_{0.5}(NaI)_{0.1}(Li_2SO_4)_{0.05}$	9E+08	6E+09	4.7E+07
$(KCl)_{0.3}(KBr)_{0.6}(NaI)_{0.1}(Li_2SO_4)_{0.05}$	2E+07	2E+07	8.7E+07
$(KCl)_{0.2}(KBr)_{0.7}(NaI)_{0.1}(Li_2SO_4)_{0.05}$	3E+07	1E+08	7963685
$(KCl)_{0.1}(KBr)_{0.8}(NaI)_{0.1}(Li_2SO_4)_{0.05}$	3E+07	9E+07	5.6E+07

Table 4(b): Elastic stiffness constant for the crystals grown by Evaporation method

Composition	Value of Elastic stiffness constant C_{11} in Pa		
	Load(p) 10g	Load(p) 25g	Load(p) 50g
$(KCl)_{0.8}(KBr)_{0.1}(NaI)_{0.1}(Li_2SO_4)_{0.05}$	3E+09	6.45E+09	4.93E+09
$(KCl)_{0.7}(KBr)_{0.2}(NaI)_{0.1}(Li_2SO_4)_{0.05}$	2E+09	1.5E+09	1.77E+09
$(KCl)_{0.6}(KBr)_{0.3}(NaI)_{0.1}(Li_2SO_4)_{0.05}$	6E+08	2.11E+08	4.01E+08
$(KCl)_{0.5}(KBr)_{0.4}(NaI)_{0.1}(Li_2SO_4)_{0.05}$	7E+09	1.34E+09	1.12E+09
$(KCl)_{0.4}(KBr)_{0.5}(NaI)_{0.1}(Li_2SO_4)_{0.05}$	2E+09	1.59E+09	1.44E+09
$(KCl)_{0.3}(KBr)_{0.6}(NaI)_{0.1}(Li_2SO_4)_{0.05}$	3E+09	2.68E+09	3.34E+09
$(KCl)_{0.2}(KBr)_{0.7}(NaI)_{0.1}(Li_2SO_4)_{0.05}$	3E+09	5.42E+09	1.97E+09
$(KCl)_{0.1}(KBr)_{0.8}(NaI)_{0.1}(Li_2SO_4)_{0.05}$	1E+09	5.03E+08	3.54E+08