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X-ray Diffraction and Micro Hardness measurement on KCl_XBr_{1-X} single crystals doped with ZnO grown from aqueous solution

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ABSTRACT

Single crystals of KCl_xBr_{1-x} mixed crystals were grown from aqueous solution. All the grown crystals were characterized by XRD and Vicker's microhardness measurement. All mixed crystals were able to index with single lattice parameter and all the grown crystals belong to hard category materials.

Keywords: Alkali halides, mixed crystals, lattice parameter, micro-hardness.

INTRODUCTION

The alkali halide crystals have importance in past six decades. They have been "model crystals" for testing many solid-state theories. In recent decades, they have also proved useful in several applications ranging from X-ray mono chromators to tunable lasers.

The use of pure simple alkali halides is limited by the mechanical systems and hence there exist the need to strengthen them. Armington et al [1] discussed two methods of improving the hardness of alkali halides (i) Solid solution hardening and (ii) impurity hardening. In the present study mixed crystals of KCl and KBr and impurity (ZnO) added KCl and KBr mixed crystals well grown by slow evaporation method and characterize them by XRD and Vicker's microhardness test.

MATERIALS AND METHODS

2.1 Growth of sample crystals

Analytical Reagent (AR) grade KCl and KBr and doubly distilled water were taken for the growth. Supersaturated solutions of KCl_xBr_{1-x} were prepared for various values of x (0.2, 0.4, 0.5, 0.6 and 0.8). Doped mixed crystals were grown by adding 2.5ml of ZnO solution to 25ml of aqueous solution totally 14 (2 pure end members, five pure mixed, 2 doped end members as five doped mixed) crystals were grown in identical conditions. The end member crystals were grown for comparison purposes.

2.2 Lattice parameter

X-ray diffraction data were collected from powdered samples using an automated X-ray powder diffractometer with monochromated CuK_α (λ=1.5406) radiation. The reflections were indexed following the procedure of Lipson and Steeple [2].

The lattice parameters were also calculated from the Vegard's Law

$$a = x\ a_1 + (1-x)\ a_2$$

Where a_1 and a_2 are lattice parameters of end members and *x* is composition.

2.3 Microhardness measurement

The hardness of a material is defined as [3] the resistance it offers to the motion of dislocations, deformation or damage under an applied stress.

Hardness testing provides useful information on the strength and deformation characteristics of materials [4]. It is correlated with other mechanical properties like elastic constant [5] and yield stress [6]. Meyer [7] established a relationship between indentation hardness and plastic and work-hardening capacity of a material.

Vicker's microhardness measurements were done on all the fourteen crystals grown using Zeitz Wetzler hardness test fitted with a diamond pyramidal indentor and attached with Leitz incident light microscope.

Indentation test was done in air at room temperature. Different loads (25, 50 and 100g) were used for indentation. Diagonal lengths'd' of indented impressions obtained for various loads were measured. The average value of the diagonal lengths of the indentation marks in each trial was calculated. Hardness of the crystal was calculated using the relation.

$$H_v = 1.8544\ (P / d^2)\ kg/mm^2$$

Where P is the applied load in kg and 'd' the average diagonal length.

To know hardness of the materials, a graph of log P versus log d is plotted. The slope of the best linear fit gives Mayer's work hardening co-efficient 'n'.

RESULTS AND DISCUSSIONS

The mixed crystals grown in the present study are shown in the Fig. 1. They are found to be more hard, stable and transparent when compared to the end member crystals.

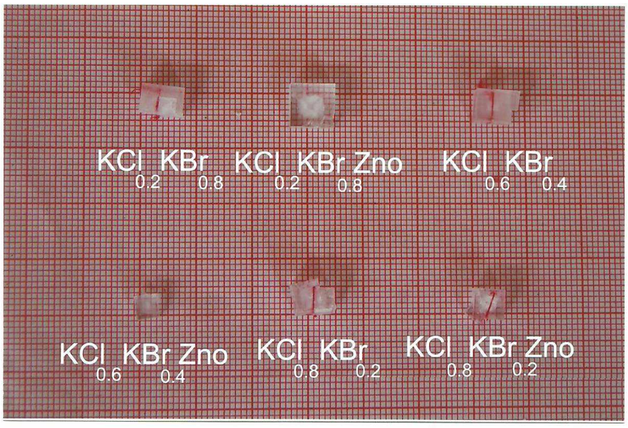

Figure 1

The lattice parameters obtained in the present study along with those estimated from Vegard's law are provided in Table 2. According to Tobolsky [8] two alkali halides AB and AC will form continuous solid solutions AB_xC_{1-x} at 20°C, provide the difference δ between their lattice parameters is less than 6%. Two compounds or elements are said to form a continuous solid solution if a single lattice parameter as measured by X-ray powder photographs, can be assigned to the solid solution at all compositions. In the present work, from the Table 1 it is found that all the compositions are assigned single lattice parameter and it indicates that they form continuous solid solutions.

Table 1 : Lattice parameters for pure and doped crystals

System	Lattice constant Pure	Doped	From Vegard's law	Deviation Pure	Doped
KCl	6.353	6.3316	---	---	---
KBr	6.679	6.6050	---	---	---
$KCl_{0.2}KBr_{0.8}$	6.557	6.556	6.6138	0.0568	0.0578
$KCl_{0.4}KBr_{0.6}$	6.464	6.441	6.5486	0.0846	0.1076
$KCl_{0.5}KBr_{0.5}$	6.3966	6.4188	6.516	0.0972	0.1194
$KCl_{0.6}KBr_{0.4}$	6.3748	6.3748	6.4834	0.1086	0.1086
$KCl_{0.8}KBr_{0.2}$	6.3529	6.3315	6.4182	0.0653	0.0867

The microhardness value and the work-hardening coefficient for both pure and ZnO added crystals are provided in Table 2. The variation of microhardness value with composition is shown in Fig. 2.

Table 2 : Microhardness value and work-hardening coefficient

System	Hardness Pure			Value Doped			n value pure	Doped
	25	50	100	25	50	100		
KCl	8.265	10.70	12.05	---	---	---	0.363	----
KBr	31.1	50.00	66.50	---	---	---	0.226	----
$KCl_{0.4}KBr_{0.6}$	11.45	13.35	17.35	15.25	20.2	29.15	0.347	0.280
$KCl_{0.5}KBr_{0.5}$	9.055	12.25	14.70	11.25	14.55	18.3	0.337	0.323
$KCl_{0.6}KBr_{0.4}$	7.65	9.70	14.05	13.35	22.35	32.5	0.311	0.180

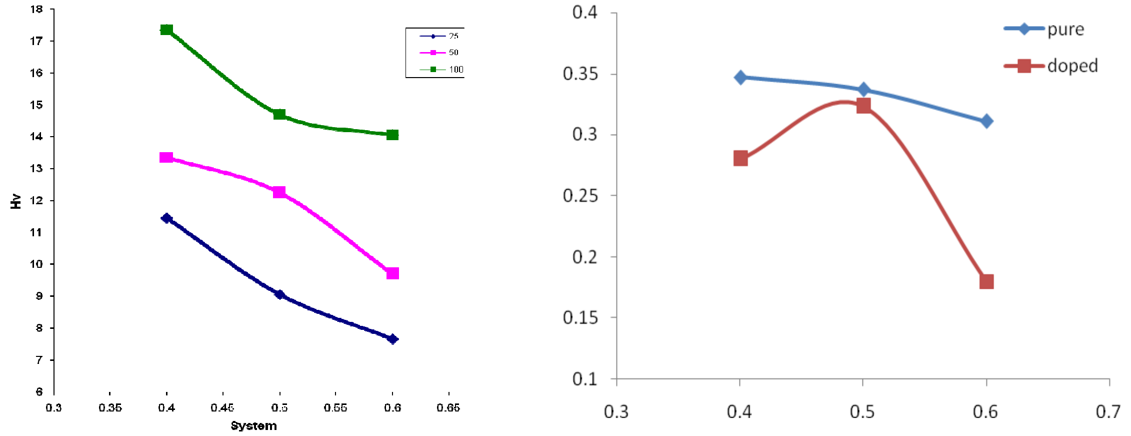

Figure 2

It is observed that the hardness values are greater for mixed crystals than the end member crystal KCl . The non-linear 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.

The ionic size of chloride (Cl) and Bromine (Br) atoms are 1.81 and 1.95 respectively. 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.

The 'n' values for different composition are shown in Table 2. The 'n' values for all the samples are found to be less than 1.6. According to Onitch [9] the 'n' values below 1.6 for hard materials and more than 1.6 for soft materials. The values obtained imply that the mixed crystals grown in the present study belong to hard category.

CONCLUSION

The mixed crystals grown in the present study are harder than the end member crystals. The XRD pattern reveals that all the mixed crystals can be an assigned a single lattice parameter. The work-hardening co-efficient values imply that all the grown crystals belong to hard category materials.

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