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Vickers microhardness studies on solution-grown single crystals of potassium boro-succinate

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ABSTRACT

The semiorganic crystals of potassium boro-succinate (KBS) were grown by slow evaporation method. KBS crystallizes in monoclinic system which was confirmed by powder XRD analysis. Vickers microhardness study has been carried out over a load range of 25–100 g. The Vickers hardness numbers (H_v) of the material increases as the load increases so the material is suitable for device fabrication. The Meyer index '*n*' is estimated to be greater than 1.6, the crystal system belongs to the soft material category. The elastic stiffness coefficient, c_{11}, has also been calculated using Wooster's empirical relation from the hardness data. The fracture toughness values 'K_c', determined from measurements of crack lengths, were estimated to be 0.15166 $MN/m^{3/2}$. The brittleness indices 'B_i' were estimated as 276 $m^{-1/2}$.

Key words:hardness, mayer index, fracture toughness

1. INTRODUCTION

Potassium boro-succinate crystals were grown by slow evaporation method. The crystal structure is determined by powdered XRD, and it is found that the crystal belongs to monoclinic system. FTIR spectrum was recorded to confirm the presence of functional groups and it is found that the presence of potassium and borate ions in the crystal lattice of succinic acid. UV-Vis-NIR was carried out and the spectrum shows the maximum absorption at UV region. The melting point of the material is found to be 195.1°C by thermal analysis. In dielectric studies the dielectric constant decreases with increase in frequency, low value of dielectric loss reveals the high purity of crystal [1].

Mechanical properties, hardness testing provides useful information on the strength and deformation characteristics of the material [2]. The chemical forces in a crystal resist the motion of dislocations as it involves the displacement of atoms. This resistance is the intrinsic hardness of a crystal. As hardness properties are basically related to the crystal structure of the material and hardness studies are carried out to understand the plasticity of the crystal [3]. So far the mechanical properties of KBS were not studied. So, in this investigation, we have measured the hardness and the related physical constant of the solution grown Potassium boro-succinate using Vickers microhardness tester.

2. EXPERIMENT

Potassium boro-succinate (KBS) was grown by slow evaporation method. Potassium hydroxide, boric acid, succinic acid, was dissolved in millipore water in equimolar ratio and the

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solution was stirred well by using a magnetic stirrer for 4 hours to get saturated solution. The pH of the solution is found to be 3.The solution is filtered using Whatman filter paper .It is poured in a petridish, covered with a perforated cover and kept in dust free atmosphere. Small seeds were observed after 5 days and transparent crystals are harvested after 45 days. The grown crystal is powdered well and powder XRD was taken and the analysis of the peak using POWDERX software, it is confirmed that the crystal belongs to monoclinic system which agrees with the reported values [1].

The microhardness of KBS crystals was determined using Vickers tester. For the static indentation test, loads varying from 25 to 100 g were applied on the grown crystal using Vickers diamond pyramid indenter connected to an incident ray research microscope. For each load *P*, an average of three impressions were recorded and the average of diagonal lengths (*d*) of the indentation mark after unloading was measured using a calibrated micrometer attached to the eyepiece of the microscope.

The Vickers hardness numbers (*H*v) were calculated using the formula

$$_v = 1.8544\ X\ P/_{d^2}\ \text{kg/mm}^2 \quad (1)$$

P − applied load in kg
d − diagonal length of the indentation in mm

3. RESULTS AND DISCUSSION

The hardness value calculated by using equation (1) and plotted against the applied load and the graph is shown in figure 1. The hardness test could not be carried out above 100 g because crack initiation and materials chipping become significant beyond this load. The hardness of the material is found to be increases with increase in the applied indentation load, due to the softer superficial layers on the specimen surface. The observed increase in hardness with increase in load is usually termed as reverse indentation size effect. Because of this KBS can be used for device fabrication.

The Meyer's law, provides an expression regarding load and size of indentation [4]:

$$P = k_1 d^n \quad (2)$$

Where k1 is the material constant and n is the Meyer index

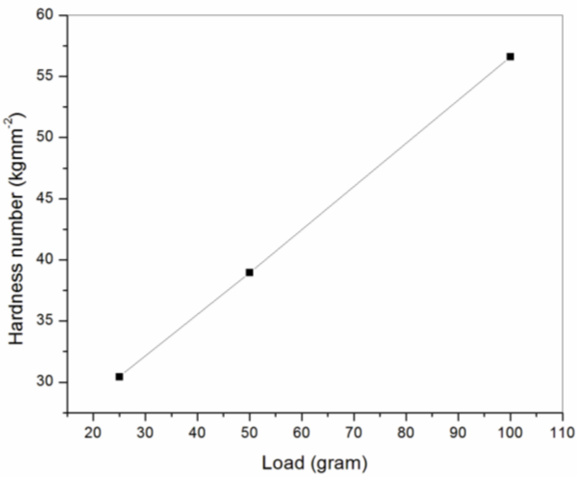

Figure.1 Load vs. Hardness

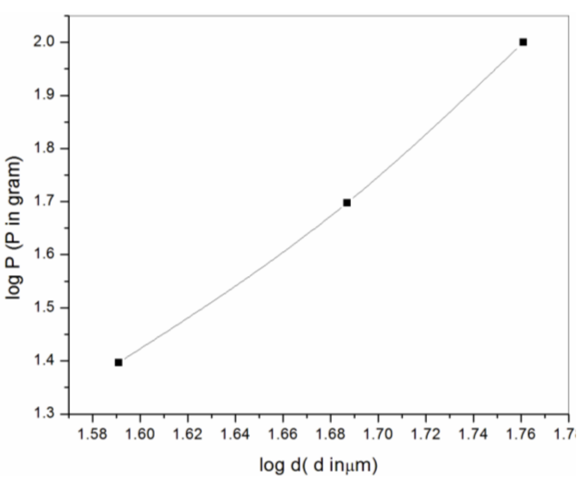

Figure.2 log d vs. log p

The plot of log *P* against log d results in straight-line as shown in figure 2 and from the graph the value of n is found to be 3 which belong to soft material [5]. Material constant was calculated from the plot figure.3 and k_1 was found out to be 4.545 kg/mm.

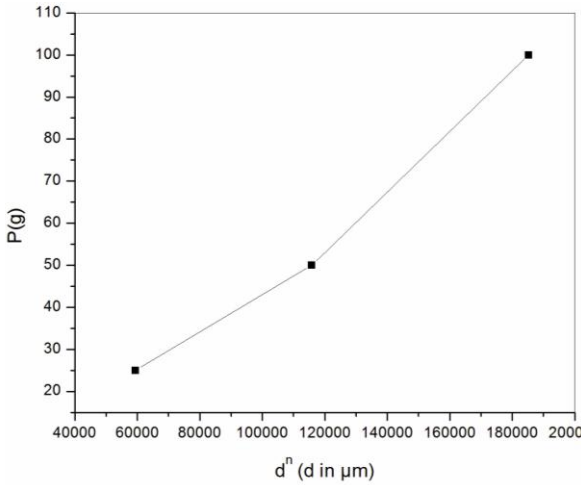

Figure.3 d^n vs P

3.1 ELASTIC STIFFNESS CONSTANT (C_{11})

The elastic stiffness constant (C_{11}) was computed by Wooster's empirical relation [6].

$C_{11}=Hv^{7/4}$ (4)

Table.1 Elastic stiffness constant of KBS

S.No	Load (g)	C_{11} X 10^{14} (Pa)
1	25	3.94
2	50	6.07
3	100	7.03

The elastic stiffness increase with increase in load (Table.1), which authenticates the tightness of bonding between neighbour atoms.

3.2 FRACTURE MECHANICS

The resistance to fracture indicates the toughness of a material and the fracture toughness *K*c determines how much fracture stress is applied under uniform loading and is given by a relation [7]

$K_c = P/\beta_0 c^{3/2}$ for $c \geq d/2$. (5)

where β_0 is a constant that depends upon the indentation geometry. For Vickers indenter β_0 is equal to 7. For the KBS crystal the value of c/a was 2.034 and the calculated K_c was 0.15166 $MNm^{-3/2}$.

3.3 BRITTLENESS INDEX

Brittleness is an important property that affects the mechanical behavior of a material and gives an idea about the fracture induced in a material without any appreciable deformation. The value of brittleness index B_i is computed using relation

$B_i = H_v / K_c$. (6)

The calculated value of B_i was 276 $m^{-1/2}$.

4. CONCLUSION

The KBS crystals were grown by slow evaporation method. The Vickers microhardness, *H*v was carried out different load. It was observed that the hardness increases with increasing load, termed as reverse ISE. As Vickers hardness number is calculated as 3, so the material belongs to soft material category. The fracture toughness of the material is found to be 0.15166 $MNm^{-3/2}$. The B_i value is computed as 276 $m^{1/2}$. The value of C_{11} gives an idea of tightness of bonding between neighboring ions. The hardness measurements may be useful in indicating the order of magnitude to be expected for the elastic constant in a new material.

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