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Study of Micro hardness and its related physical constants of L-Cysteine doped BTCA single crystals

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Abstract **The nonlinear optical single crystal of L-cysteine doped Bis thiourea Cadimum acetate(L-BTCA) was grown successfully by slow evaporation technique using water as solvent at room temperature. Vickers micro hardness study has been carried out over a load range of 25-100g.The hardness number (Hv) of the material increases with the load, indicates that the material is suitable for device fabrication. The Meyer index 'n'is estimated and found to be 5.76 shows that the material belongs to soft material category. The elastic stiffness coefficient ($C_{11)}$ has calculated for different loads. The result shows that it increases with the load, which is an indication of the tightness of bonding with neighboring atoms of the crystal. The hardness related physical constants like the yield strength (σv), fracture toughness value (Kc) and Brittleness index (Bi) were also estimated in this paper. The hardness result and the indentation induced cracking yield the values of the yield strength,fracture toughness and brittleness index for L-BTCA.**

Keywords —Micro hardness, Hardness number, Elastic stiffness, Yield strength, Fracture toughness, Brittleness index

I. INTRODUCTION

Hardness is a measure of the resistance to localized plastic deformation induced by mechanical indentation. Macroscopic hardness is generally characterized by strong intermolecular bonds, but the behavior of solid materials under force is complex[1] .Hardness dependent on ductility, elastic stiffness, plasticity, strain, strength, toughness etc. Micro hardness studies have been applied to understand the strength and deformation characteristics of a material, since the hardness properties are basically related to the crystal structure of the material [2].

The Vickers test is often easier to use than other hardness tests since the required calculations are independent of the size of the indenter, and the indenter can be used for all materials irrespective of hardness [3]. The basic principle, as with all common measures of hardness, is to observe the questioned material's ability to resist plastic deformation from a standard source. The Vickers test can be used for all metals and has one of the widest scales among hardness tests. The unit of hardness given by the test is known as the Vickers Pyramid Number (Hv) or Diamond Pyramid Hardness (DPH).

II. EXPERIMENTAL

Saturated solution of pure BTCA crystal was grown by slow evaporation technique by mixing aqueous solution of zinc acetate and thiourea in the ratio 1:2.The saturated solution of pure BTCA is obtained by dissolving BTCA salt in double distilled water. The amino acid L cysteine 2 wt % is added to the supersaturated solution and stirred well for six hours at constant speed to achieve homogeneity throughout the volume. The saturated solution is filtered and kept for evaporation at room temperature. Good quality optical transparent crystals were obtained within a period of 20-25 days.

The XRD analysis was taken and it is confirmed that the crystal belongs to the orthorhombic system.FTIR spectrum confirms the presence of functional group and it is found that cadmium is bonded to sulphur.UV-Vis spectrum of the crystal shows maximum absorption in the UV region and optical band gap is found to be 2.5eV.The melting point of the material is found to be 189.30 by thermal analysis. The surface properties of the crystals were investigated by AFM and it is found that the surface is perfectly smooth and flat[4][5].

The mechanical property of the L-BTCA crystals was studied using Vickers hardness tester, fitted with a Vickers's diamond pyramidal indenter. Indentation can be done with different loads from 25g to 100g. The length of the two diagonals is measured by a calibrated micrometer attached to the eyepiece of the microscope after unloading and the average (d) is found out. For a particular load at least two well-defined impressions have considered

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and the Vickers micro hardness profile of the crystal is calculated using the formula [6]

$$H_v = \frac{1.8544 \, xP}{d2} \, Kg/mm^2$$

where P is the applied load and *d* is the mean diagonal length of the indentation.

III. Result and Discussion

A. Load variation

The dependence of micro hardness number with applied load was studied and is shown in the figure 1. It shows that the hardness increases with the increasing of load. This can be considered as the reflection of Indentation Size Effect (ISE) [7]. The ISE can be related to energy loss owing to the cracking of the specimen during indentation [4]. An increase in the mechanical strength will have a significant effect on NLO device fabrication and processing such as ease in polishing and less wastage due to cracking or breakage while polishing [8].

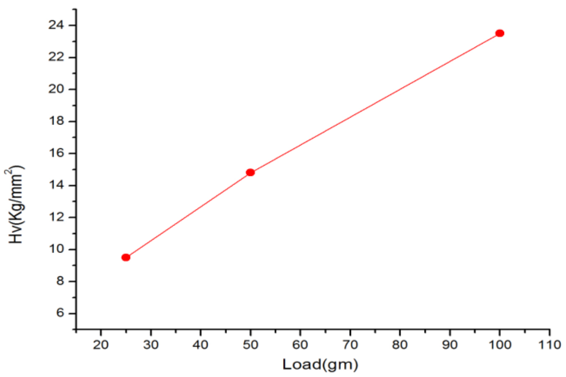

Fig.1 graph of load vs hardness number[4]

B. Determination of Meyer index

Meyer's law relates to the load and size of the indentation as [9]

$$P = k_1 \, d^n$$

Here, 'k_1' is the constant for a given material and ' n' is the Meyer index. Using the above Equation the work hardening coefficient 'n' was determined from the slope of a plot of log P versus log d.

Figure 2 shows the plot of log P versus log d fitting data using least-squares fit method and the value of n was found to be 5.76. The working hardening coefficient (n) should be greater than 1.6 for soft materials [10]. The 'n' value of L-BTCA crystal is 5.76 and hence it belongs to soft material category.

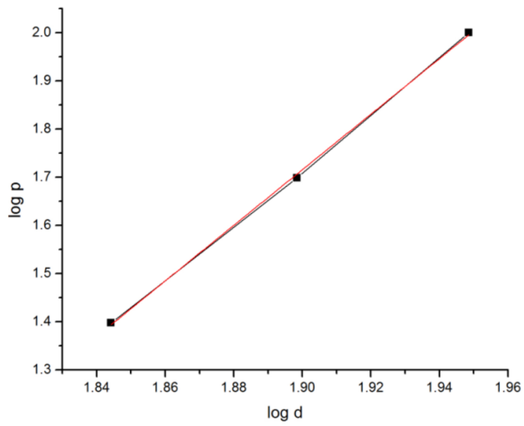

Fig.2 graph of log d vs log P

C. Elastic stiffness constant (C_{11})

The elastic stiffness constant (C_{11}) for different loads is calculated using Wooster's empirical formula [11]

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

The Elastic stiffness constant (C11) indicates the binding forces between the ions. The calculated C11 values of L-BTCA crystal for different loads are given in the Table .

Sl No	Load (gm)	C_{11}($x10^6$ Pa)
1	25	17.45
2	50	38.88
3	100	989.49

Elastic stiffness increases with increase in load which indicates the tightness of bonding with neighboring atoms [12]

D. The yield strength (σv)

The yield strength (σv) can be calculated from the micro hardness value for n>2 using the equation. [13]

$$\sigma v = \frac{Hv}{2.9}[1-(n-2)] \left[\frac{12.5(n-2)}{1-(n-2)}\right]^{(n-2)}$$

The yield strength was calculated for various loads as shown in Table.

Sl. No	Load (gm)	σv ($x10^6$ Pa)
1	25	4.03767
2	50	6.29027
3	100	9.98793

E. Fracture mechanics

The fracture mechanics in the indentation process have provided an equilibrium relation for a well developed crack extending under center loading conditions. For each measurement, only well-defined cracks developed by the indentation were considered and the average crack length of

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all such cracks was taken for a particular indentation impression [3]. The crack length was measured from the center of indentation mark up to the tip of crack in µm. The resistance to fracture indicates the toughness of a material and the fracture toughness Kc determines how much fracture stress is applied under uniform loading and is given by a relation [14]

$$Kc = \frac{P}{\beta_0 c^{3/2}} \quad \text{for } c > \frac{d}{2}$$

where β_0 is a constant that depends upon the indentation geometry. For Vickers indenter β_0 is equal to 7.

Sl. No	Load (gm)	Kc (gm/$\mu m^{3/2}$)
1	25	0.030376
2	50	0.060751
3	100	0.121502

F. 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 [2]. The value of brittleness index Bi is computed using relation, [15]

$$B_i = \frac{Hv}{Kc}$$

Sl. No.	Hv (Kg/mm^2)	Bi ($m^{-\frac{1}{2}}$)
1	9.5	312.7509
2	14.8	243.6165
3	23.5	193.4117

IV. Conclusion

The L-BTCA crystals were grown by slow evaporation method. Vickers microhardness study reveals that hardness value increases up to a load of 100 g. Cracks developed around the indentation mark for loads above 100 g. The Meyer's index, 'n'of L-BTCA shows that it belongs to soft material category. The high value of C_{11} reveals the tightness of bonding between neighboring ions. Fracture toughness values Kc determined from the measurements of crack length. The brittle index and yield strength were also calculated. The hardness measurements reveals that the material is a leading candidate for device fabrication.

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