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INVESTIGATIONS ON VICKERS MICROHARDNESS AND ITS RELATED CONSTANTS OF SINGLE CRYSTAL: L-HISTIDINIUM SEMISUCCINATE (LHS)

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ABSTRACT

Single crystals of L-Histidinium semisuccinate were grown by slow cooling technique. Single crystal X-ray diffraction study confirms the formation of the crystal structure. Vickers microhardness studies were carried out on the grown crystals of L-Histidinium semisuccinate over a load range of 10-500 g. It was found that the grown crystal exhibits normal indentation size effect as the hardness number decreases with increase in load. The Meyer's index number 'n' was found to be greater than 1.6, showing the grown crystal belongs to a soft material category. The resistance pressure 'W' was calculated using Hays-Kendall approach. The values of fracture toughness (K_c) were determined from the measurements of crack lengths and the types of cracks were identified. Brittleness indexes (B_i) and yield strength (σ_y) were also assessed from the values of Vickers hardness number (H_v). The elastic stiffness coefficient (C_{11}) values have been calculated using Wooster's empirical formula. The results were discussed in detail.

Keywords: Solution growth, Vickers hardness, Work hardening coefficient, Elastic stiffness constant, Brittleness index

INTRODUCTION

Nonlinear optics is continuing to develop and refinement in the evolution of lasers and higher nonlinear optical materials have resulted in a variety of commercially available nonlinear optical devices[1]. Strength and deformation characteristics of the material will be determined using Vickers microhardness analysis. 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[2,3]. The interest in hardness measurement does not depend on the technical point of view but it also has an opportunity to analyze the degree of lattice order of single crystalline materials[4,5] So far the mechanical properties of LHS was not studied. In the current investigation, we have measured the hardness and the related physical constant of the solution grown L-Histidinium semisuccinate using Vickers microhardness tester.

EXPERIMENTAL

Growth solution was prepared by dissolving 1:3 ratio of L-Histidine and Succinic acid in triple distilled water. Growth was progressed by following the evaporation of saturated solution for a period of 16 days. Single crystals of L-Histidinium semisuccinate were grown by slow cooling method after optimizing the growth conditions (Figure 1). Powder X-Ray diffraction confirmed the formation of title and the high intensity peaks shows the crystalline nature of the sample. In conventional testing, Vickers hardness of LHS is determined as the ratio between the applied load P and the corresponding contact area between the material and indenter. The mechanical characterization of the grown LHS was carried out using

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Economet VH 1 MD hardness tester for loads varying from 10 g to 500 g at room temperature with a dwell time of 10 S.

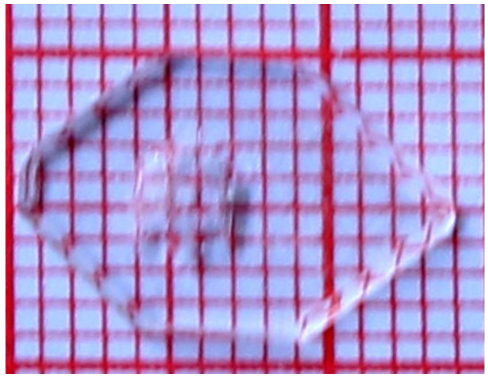

Fig.-1: As grown crystal of LHS

RESULTS AND DISCUSSION

Structural Analysis

Powder XRD was carried out using Bruker D8 advanced, the intense peaks were indexed respectively using Powder X ver 3.2. Lattice parameters were calculated as $= 7.030$ Å, $b = 8.762$ Å, $c = 24.322$Å $and\ volume\ V = 1510$ Å^3 . The grown crystal belongs to orthorhombic system with non centrosymmetric space group of $P2_12_12_1$. The obtained values are in good agreement with the reported values [6].

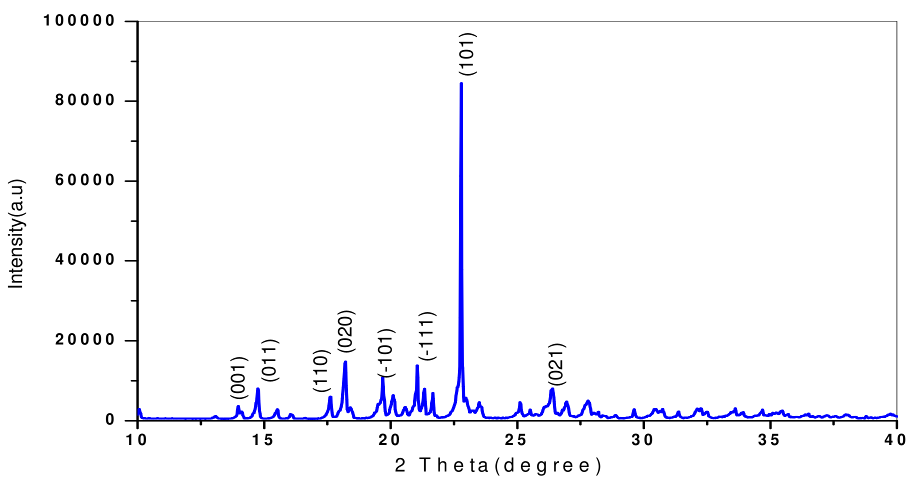

Fig.-2: Powder XRD pattern of LHS

Vickers Micro Hardness Analysis

The microhardness of LHS crystals was determined using Vicker tester. For the static indentation test, loads varying between 10 to 500 g were applied on the grown crystal using diamond pyramid indenter connected to an incident ray research microscope. An average of at least three impressions was recorded for each load with a dwell time of 10 s. The calibrated microscope attached to the system has measured the diagonal length (d) of the indentation mark after unloading. It was calculated using the formula:

$$H_v = 1.8544\, P/d^2\ kg/mm^2 \quad (1)$$

It is observed from the hardness profile (Fig.-3), that the hardness value decreases with the increase in load which indicates LHS is exhibiting Normal Indentation Size effect (ISE). When the applied load is small, the indenter penetrates only on an upper surface layer of the crystal and depending on the strain distribution of the upper layer, there is a fall of hardness value in low load region. As the load increases, the depth of indenter increases and both the effects of the inner layer and surface layer contribute to less hardness value[7].

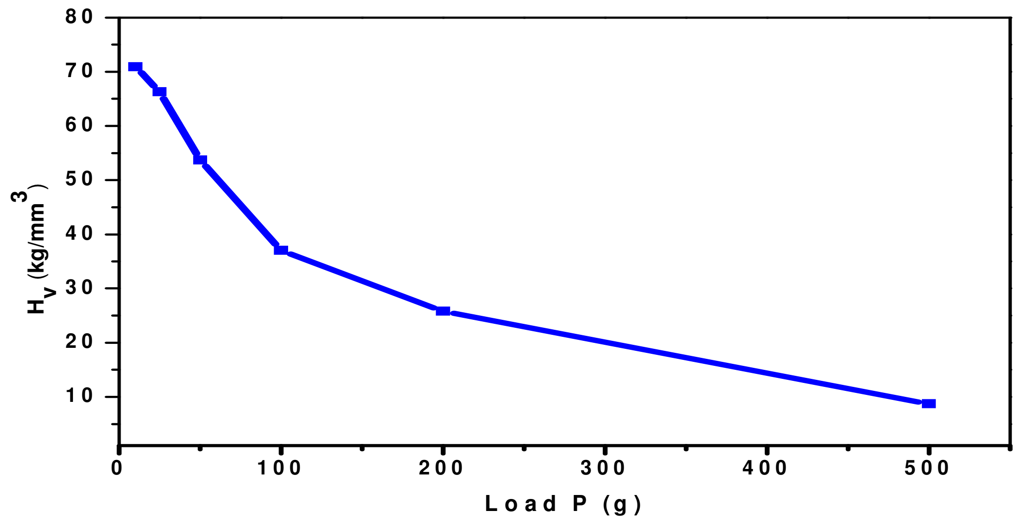

Fig.-3: Vickers Hardness number

The Meyer's law provides an expression corresponding to load and size of indentation[8]. The hardness of the material depends on the strength of the chemical bonding between the molecules which is responsible for its distinct hardness. From the result, we concluded that the hardness of the material got decreased as the strength of the chemical bonding consequently increased.

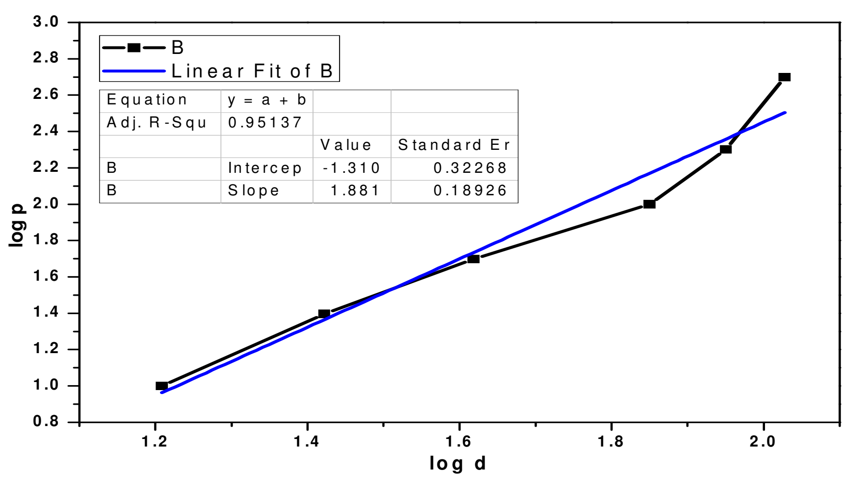

Fig.-4: Plot of Log p vs. log d

The Meyer's law, provides an expression regarding load and size of an indentation [8].
$P = K_1 d^n$ K_1 is the material constant and n is the meyer index.

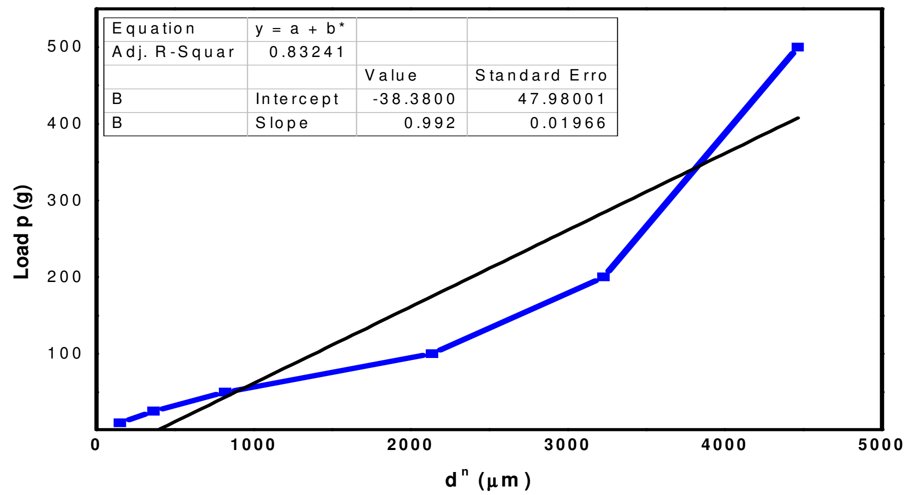

Fig.-5: Plot of Load vs d^n

The plot of load vs. d^n shows that the materials constant value of 0.922 kg/mm.

Elastic Stiffness Constant

The elastic stiffness constant (C_{11}) was calculated using Wooster's empirical relationship [9] $C_{11} = (H_v)^{7/4}$.

Table-1: Elastic Stiffness Constant Values of LHS

Load (g)	H_v (kg/mm^2)	C_{11} x10^{14}(Pa)
10	70.9	17.32
25	66.3	15.40
50	53.7	10.65
100	37.2	5.55
200	25.8	4.42
500	8.7	2.92

Table-2: Calculated Vickers Hardness Parameters

Parameter	Value
Work hardening co-efficient (n)	1.829
Material constant (k_1)	0.92 kg / mm
Resistance Pressure (W)	12.67 g
Load dependent constant (A_1)	39.27 kg / mm^2
Fracture mechanics (K_c)	2.504 MNm$^{-3/2}$
Type of Crack	Palmquist
Yield Strength	2.9 kg/ mm^2
Brittleness index (B_i)	3.474 m$^{-1/2}$

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:

$$K_c = \frac{P}{\beta_0 C^{3/2}} \text{ MNm}^{-3/2} \qquad (2)$$

For c ≥ d/2 where β_0 is a constant that depends upon the indentation geometry. For Vickers indenter $\beta 0$ is equal to 7. For the LHS crystal the value of c/a was 2.034 and the calculated Kc was 2.504 $MNm^{-3/2}$. The nucleation of the cracks is due to the stress field formed during the course of indentation. Based on the analysis, the reliable value of the fracture toughness only if c/a ≥ 2.5 (Median crack) whereas c/a ≤ 2.5 (Palmquist crack). The fracture toughness is calculated for higher load where c/a ≤ 2.5 so it is seen that crack is Palmquist.

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 [10]

$$B_i = H_v / K_c \qquad (3)$$

CONCLUSION

The LHS crystals were grown by a 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 normal ISE. As Vickers hardness number is calculated as 1.88, so the material belongs to a soft material category. The fracture toughness of the material is found to be 2.504 $MNm^{-3/2}$. The Bi value is computed as 3.474 $m^{-1/2}$. The value of C_{11} gives an idea of the tightness of bonding between neighboring ions and the nature of crack is Palmquist. 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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