Mechanical properties of L-Valine single crystals

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The mechanical properties of crystals were evaluated by mechanical testing. The fastest and simplest type of mechanical testing is hardness measurement. The Vickers microhardness studies have been carried out for L-Valine crystals grown by a slow evaporation technique over a load range of 10–100 g. The Vickers hardness number (H_v) was found to increase with the increase in load. The Meyer's index number '*n*' was calculated from H_v and yield strength (σ_v). The Young's modulus was calculated using the Knoop hardness values. Hardness anisotropy has been observed in accordance with the orientation of the crystal.

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1. Introduction

Recently, there is an increasing demand for exploring new electro-optic materials having simple structure. The measurement of hardness is very important as far as the fabrication of devices is concerned. Measurement of hardness is a useful non-destructive testing method to determine the bond strength [1]. The microhardness correlates with other mechanical properties such as elastic constant and yield strength [2]. According to Jianghong Gong [3], during an indentation process, the external work applied by the indenter is converted into a strain energy component proportional to the volume of the resultant impression and a surface energy component proportional to the area of the resultant impression. Microhardness is a general microprobe for assessing the bond strength, apart being a measure of bulk strength. The crystal slices are well polished to avoid surface defects which influence the hardness value strongly. In the present investigation hardness parameters and hardness anisotropy have been analysed.

2. Preparation and mechanical properties

L-Valine single crystals were grown from aqueous solution by slow evaporation technique using water as a solvent. The starting material was synthesized by dissolving high purity L-Valine (AR grade) in triple distilled water. After a period of three weeks, optically transparent defect free crystals were obtained from the mother solution. The mechanical characterization of L-Valine crystals was made by Vickers microhardness tests at room temperature. The grown crystal with flat and smooth faces and free from any defects was chosen for the static indentation tests. The surface (010) was polished gently with methanol and mounted properly on the base of the microscope. Now the selected face was indented gently by varying the loads for a dwell period of 10 s using Vickers indenter attached to an incident ray research microscope (Mututoyo MH112, Japan). The Vickers indented impressions were approximately square in shape. The length of the two diagonals was measured by a calibrated micrometer attached to the eyepiece of the microscope after unloading. For each load, at least five well-defined impressions were taken and the average of all the diagonals (d) was considered. The Vickers hardness number (H_v) was calculated using the standard formula,

$$H_{\rm v} = 1.8544 P / d^2 \tag{1}$$

where P is the applied load and d is the mean diagonal length of the indentation. The Knoop indented impressions were approximately rhombohedral in shape. The long diagonal length (d) was considered for the calculation of the Knoop hardness number (H_k) using the relation

$$H_{K} = 14.229P/d^{2}$$
(2)

where P is the applied load in kg, d in mm and H_k is in kg/mm². Crack initiation and fragmentation become significant beyond 100 g of the applied load. So hardness test could not be carried out above this load. From Wooster's empirical relation [4],

$$C_{11} = H_V^{-7/4} \tag{3}$$

elastic stiffness constant (C₁₁) of L-Valine was calculated. As indentation initiates plastic deformation in a crystal, which is highly directional in nature, the hardness measurement may be a function of the orientation of the indented crystal. Thus, any anisotropic effect shown by the size of the indentation mark will be reflected in the hardness number. To study this hardness anisotropy present in the L-Valine crystal, the crystal was initially mounted on the stage of the microscope properly and indented using Vickers indenter. The hardness was measured taking the initial position as 0°. The stage of the microscope was then rotated keeping the indenter fixed, and H_v was measured at every 30° interval till the original position (360° or 0°) comes back.

3. Results and discussion

3.1. Vickers microhardness test

Fig. 1 shows the variation of H_v as a function of applied load ranging from 10 g to 100 g for the L-Valine crystal. It is very clear from the figure that H_v increases with the increase of load. The Meyer's index number was calculated from the Meyer's law, which relates the load and indentation diagonal length as

$$P = kd^n \tag{4}$$

$$\log P = \log k + n \log d \tag{5}$$

where k is the material constant and 'n' is the Meyer's index. In order to find the value of 'n', a graph is plotted for log P against log d (Fig. 2) which gives a straight line. From the slope of the line the Meyer's index number 'n' was calculated to be 2.66.

According to Onitsch [5] 'n' lies between 1 and 1.6 for hard materials and is greater than 1.6 for soft materials [4]. The 'n' value observed in the present studies is around 2.66 suggesting that the grown L-Valine crystal is a relatively softer material. Yield strength can also be calculated using the relation [6]

$$\sigma_{v} = (H_{v}/3)(0.1)^{n-2} \tag{6}$$

where σ_y is the yield strength, H_v is the hardness of the material and n is the logarithmic exponent. According to the relation, the yield strength is found to be 3.09 MPa and hence the grown L-Valine crystal has relatively low mechanical strength.



Fig. 1. Variation of the microhardness number H_v with load.



Fig. 2. log P vs log d.

The elastic stiffness constant (C_{11}) was calculated using Wooster's empirical formula [4], which gives an idea about the tightness of bonding between neighboring atoms. The calculated elastic stiffness constant for different loads is shown in Table 2.

$$C_{11} = H_V^{-7/4} \tag{7}$$

Table 1. Elastic stiffness constant of L-Valine.

$C_{11 x 10}^{14} Pa$
2.48
4.67
7.33
14.00

3.2 Anisotropic behaviour

For studying the crystal anisotropy, the microhardness was measured by varying the crystal orientation over the range of 0° -360° in steps of 30°. From Fig. 3, it is seen that the hardness number is changed when the crystal is rotated through 360° in step of 30°. It is also observed that the hardness number reaches maximum value at 60°, 210°, 270° and 330° orientations respectively. The variation in the hardness number due to rotational orientation of the crystal indicates the anisotropic nature of L-Valine crystal in respect of mechanical properties.



Fig. 3. Anisotropy nature of the L-Valine crystal.

3.3 Knoop microhardness test

The graph was plotted against Knoop hardness (H_k) and load (P) and it is shown in Fig. 4. From this measurement, it was found that as the load increases up to 50 g, the Knoop microhardness number also increases as in the case of Vickers microhardness number. The long diagonal length (d) was considered for the calculation of the Knoop hardness number (H_k) using the relation,

$$H_{\kappa} = 14.229 P / d^2 \tag{8}$$

where P is the applied load in kg, d in mm and H_k in kg/mm². From the Knoop microhardness measurements the Young's modulus (E) of the crystal was calculated using the relation [7],

$$E = 0.45H_{\kappa} / (0.1406 - b/a) \tag{9}$$

where H_k is the Knoop microhardness values at a particular load and b and a are the shorter and longer Knoop indentation diagonals respectively. The calculated Young's Modulus is $1.028 \times 10^{10} \text{ Nm}^{-2}$.



Fig. 4. Variation of the knoop microhardness with load.

4. Conclusion

Vickers and Knoop hardness numbers were calculated for L-Valine crystal by the application of load and the hardness numbers was found to increase with increase in load. The anisotropic nature of the crystal was revealed due to the change in the hardness number for different rotational orientations of the crystal. The value of the Meyer's index, n turned to be greater than 1.6 and thus L-Valine falls in the soft material category. The calculation of the stiffness constant reveals that the binding force between the ions is quite strong. Also the Young's modulus was calculated from the diagonal lengths of Knoop indentation.

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