Journal of Advances in Applied Sciences and Technology
ISSN : 2393-8188 (print)
2393-8196 (online)
www.milliyasrcollege.org.journal.php

Microhardness andAntimicrobialproperties of L-valine doped ADP crystal

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Abstract :
The single crystal oftwo mole% L-valine doped ammonium dihydrogen phosphate(ADP) crystal has been grown by slow evaporation technique at room temperature. The fastest and simplest type of mechanical testing is hardness measurement. The Vickers microhardness study has been carried out over a load range of 10–100 g. The Vickers hardness numbers (Hv) 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(c11) and Yield strengthwere also been calculated using Wooster's empirical relation from the hardness data.

1. Introduction

Ammonium dihydrogen phosphate (ADP) crystal is an interesting inorganic material with allied non linear optical (NLO), dielectric and antiferroelectric properties with a wide scope of distinct applications such as electro optic modulator, parametric generators, optical switches in inertial confinement fusion and acoustic-optical devices[1]. The amino acids contains a deprotonated carboxylic group (COOH) and protonated amino group (NH_2), which makes them ideal candidates for NLO applications. Theeffect of amino acids on the growth and properties of different material crystals have been reported for NLO applications [2-7]. R.N Shaikh et.al. reported studies on L-valine doped ADP crystal which has been demonstrated to exhibit large second harmonic generation,1.92 times greater than that of potassium dihydrogen phosphate (KDP).The measurement of hardness is very important as far as the fabrication of devices is concerned. So far no report is found on the mechanical properties of the grown crystal in the literature to our knowledge.

2. Experimental procedure

2.1 Crystal growth

The AR grade ADP salt was gradually dissolved in deionized water until a saturated solution was obtained. In the saturated solution of ADP prepared in separate beakers, the calculated amount of 1 and 2 mole% L-valine was added with constant stirring. The prepared solutions were allowed to evaporate at room temperature to procure the respective salts. The purity of the doped compounds was gained by successive recrystallization using deionized waterThe Kurtz-Perry powder test revealed that the SHG efficiency was found to be enhanced with 2 mole% L-valine in ADP crystal. Hence, the saturated solution of 2 mole% L-valine doped ADP was prepared and allowed to evaporate at room temperature. The well phased, good quality transparent seeds were harvested within 8-10 days.

3. Mechanical studies

The mechanical characterization of grown crystal was made by Vickers Microhardness test at room temperature. The grown crystal with flat and smooth faces and free from defects was chosen for static indentation test. The surface was polished gently with water and mounted properly on the base of the microscope. Now the face was intended gently by the loads varying from 10 to 200 g for a dwell period of 10 sec using Vickers diamond pyramid indenter attached to an incident ray research microscope (Mututoyo MH 112, 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:$H_v = 1.8544\ P/\ d^2$ where, P is the applied load in kg, d is in mm and H_v is in $kgmm^{-2}$. A plot of variation of hardness value and corresponding loads is shown in Fig.1. The hardness number was found to be linearly increased over the entire range of applied load.Similar behaviour was reported in the L-lysine doped ADP crystal [3]. The Meyer's index number was calculated from the Meyer's law[8], which relates the load and indentation diagonal length: $P= k\ d^n$, Where k is the material constant and n is Meyer's index.

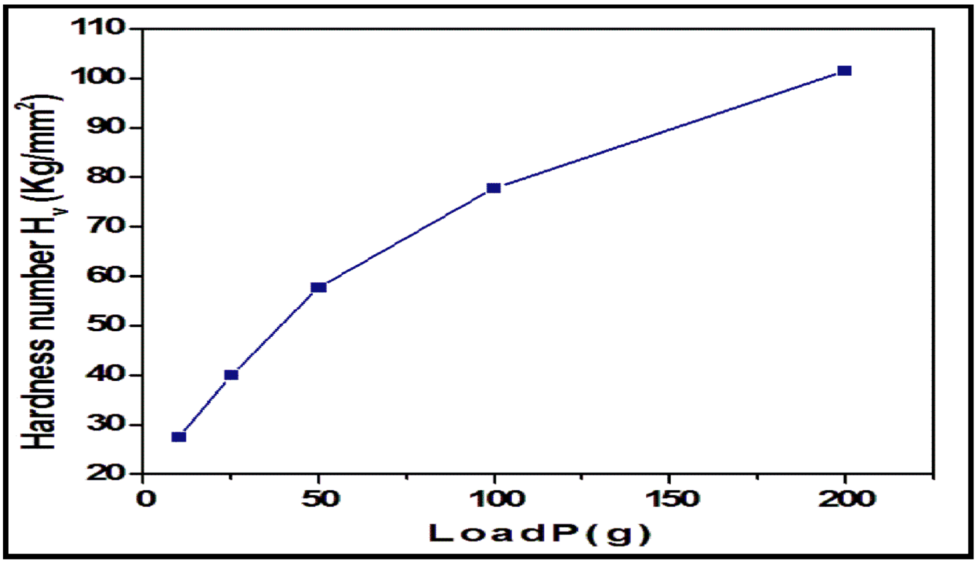

Fig.1 Variation of Vickers Hardness Number with load

Thus a plot of log P versus log d gives a straight line and the slope of which gives the value of 'n', the work hardening coefficient. The plot is given in Fig. 2 and the calculated value of 'n' is 3.51. Hv shouldincrease with the increase in P if n > 2 and decrease if n < 2. The 'n' obtained from the plot agrees well with the experiment. According to Onitsch 'n'should lie between 1 and 1.6 for harder materials and is greater than 1.6 for soft materials [9].Thus the grown crystal belongs to the soft material.

Yield strength was calculated using the relation [10]

$$\sigma_y = (0.1)^{n-2} H_v/3$$

Where σ_y is the yield strength H_v is the hardness of the material and n is logarithmic exponent. According to the relation the yield strength was found to be 6.83 M Pa.

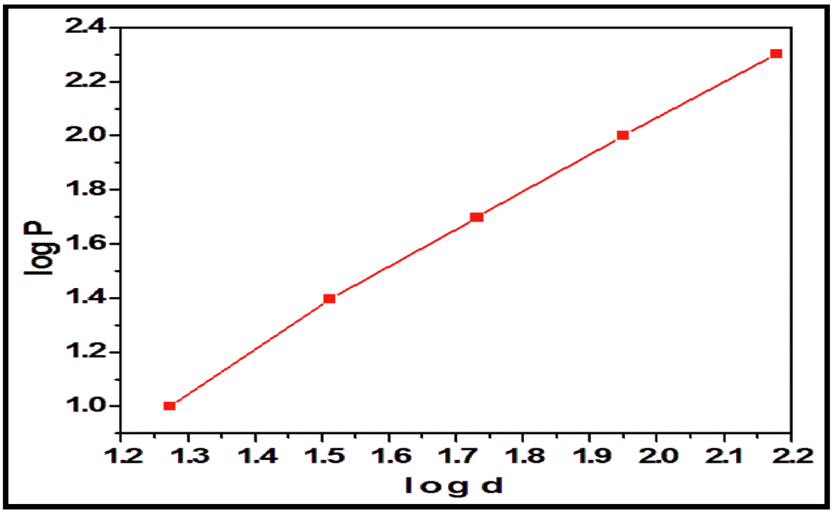

Fig. 2 Plot of logp vs. logd

The Elastic stiffness constant (C_{11}) was calculated using Wooster'sempirical formula [11],$C_{11} = H_v^{7/4}$

From the elastic stiffness constant we can get some idea about the tightness of bonding between the neighboring atoms. The calculated elastic stiffness constant for different loads is given in Table 1.The stiffness constant C_{11} is quite high, revealing that the binding forces between the ions are strong.

Table 1

Elastic stiffness constant for different load.

Sr.No.	Load (g)	H_v (Kg/mm^2)	Elastic stiffness constant C_{11}(x10^{14}Pa)
1	10	27.43	5.64
2	25	39.96	10.9
3	50	57.71	20.74
4	100	77.78	34.97
5	200	101.52	55.73

4. Antimicrobial Studies

The slat of doped crystal is subjected to antibacterial studies using Agar Diffusion Method. it was observed that the salt has significant antibacterial action against the test cultures *viz. Bacillus subtilis* and *Escherichia coli*. The compound has shown zone of inhibition if growth against both Gram's negative as well as Gram's positive cultures.

The findings are recorded on Tabe 2. The compound showed greater activity against Gram's Negative Bacteria as compared to Gram's Positive bacteria.

Table 2.: Antibacterial activity of L-valine doped ADP crystal

Sample	**Zone of Inhibition of growth (mm)**							
	Bacillus subtilis				***Escherichia coli***			
	Plate 1	**Plate 1**	**Plate 3**	**Average**	**Plate 1**	**Plate 1**	**Plate 3**	**Average**
L-valine doped ADP crystal	5	7	6	**6**	8	9	8	**8.5**

5 Conclusion

Vickers hardness numbers was calculated for two mole% L-valine doped ADP crystal by the application of load and the hardness numbers was found to increase with increase in loadso the material is suitable for device fabrication. The value of the Meyer's index, n turned to be greater than 1.6 and thus two mole% L-valine doped ADP crystal falls in the softmaterial category. Thecalculation of the stiffnessconstant reveals that the binding force between the ions is quitestrong.

The L-valine doped ADP crystal, has shown significant antimicrobial activity against both the test cultures. This clearly indicates that the synthesized compound will have stability against microbial action.

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