HARDNESS STUDIES ON SOLUTION GROWN LITHIUM POTASSIUM SULPHATE SINGLE CRYSTALS – AN INORGANIC NONLINEAR OPTICAL MATERIAL

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ABSTRACT

Single crystal of Lithium Potassium sulphate is grown by slow evaporation technique at room temperature. The grown crystal is characterized by Vickers micro hardness test. It is carried out by over a load with a range of 10 – 100 gm on the selected smooth plane surface of the crystal. Vickers’s hardness values (HV) are calculated for selected applied load and it is found to be increasing with the increasing applied load. The Meyer’s index number (n) is calculated from the Vickers hardness number and it confirms that the grown material belongs to soft material category. The Elastic stiffness constant (C11) is calculated using Wooster’s empirical relation. Further the Yield strength (σy), Brittleness index (B), and the Fracture toughness (Kc) are calculated to the grown LKS crystals.

Keywords: Crystal growth, Inorganic compounds, NLO property, X-ray diffraction, Hardness.

1. INTRODUCTION

Nonlinear optical materials are dominating in the field of telecommunication, photonics, optoelectronics etc. [1–4]. Lithium Potassium sulphate (LKS) single crystals are the promising NLO material. The grown crystal belong to hexagonal system with the space group P63 and the cell parameters a=b=5.1453Å and c = 8.6342 Å [5, 6]. The melting point of the title compound is measured and it was found to be 114 °C.

The second harmonic generation efficiency is 1.6 times higher than that of the Potassium dihydrogen orthophosphate (KDP) single crystal. With the best of our knowledge, there is no report available in the detailed discussions of its hardness studies except to calculate the hardness coefficient. Hence in this paper we report the elastic stiffness constant(C11), brittleness index(B), Yield strength(σy), etc., of LKS single crystals.
2. EXPERIMENTAL PROCEDURE

2.1. Crystal Growth

Single crystals of Lithium Potassium sulphate were synthesized by dissolving Lithium sulphate and Potassium sulphate in the molar ratio 1:1 in double distilled water \[6\]. The prepared solution was stirred continuously using magnetic stirrer for obtaining the homogenization mixture of the synthesized compounds. The solution was filtered by Whatman filter paper to remove the suspended impurities and it was kept in the vibrational and dust free atmosphere. After the growth period of 25 days, a well defined single crystals of LKS were harvested.

3. MICRO HARDNESS

Hardness is one of the properties of a material and it is related to bond strength and the structure of the crystal. Hardness study is useful to understand the plasticity of the material \[6-8\]. In a crystal, chemical forces resist the motion of dislocation which involves the displacement of an atom. Generally, this type of resistance is intrinsic hardness of the crystal. The Vickers’s micro hardness test was carried out using Shimadzu HMV-2000 micro hardness tester. Crystal with the flat and smooth plane was chosen for the static indentation test. For static indentation test the clamping devices were mounted on microscope’s base under the control of XY stage of dimensions of 50X50 mm (2’X2’). The selected face is used for Vickers’s pyramidal indenter with the loads varying from 10 to 100 gm in steps of 10g with a constant indentation period of 10s for each load. The Vickers’s pyramidal test for static indentation test reveals that the impressions of crystal were approximately square. The length was measured by calibrated micrometer in two diagonals placed inside of the eyepiece \[9\]. For the particular load the average of the diagonals (d) was also considered.

3.1. Vickers’s Hardness Studies

The Vicker’s micro hardness number was evaluated using the relation

\[
H_v = \frac{1.8544 \times P}{d^2} \text{kg/mm}^2
\]  

Where, Hv is Vickers micro hardness in kg/mm², P is applied load in gm, d is the diagonal length of indentation in mm². 1.854 is a constant of geometrical factor for the dimensional pyramid. The plot between the applied loads versus Vickers’s hardness numbers is shown in the Fig.1. From the figure, it is observed that the hardness of the crystal increases with increasing the applied load.

The variation of load (P) versus stiffness constants (C₁₁) in accordance with the respective applied loads was calculated using the equation (2). The plot of P Vs C₁₁ is shown in Fig. 2. From the figure, it is confirmed that the stiffness constant increases with increasing applied load.

\[
C_{11} = (H_v)^{7/4}
\]  

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The work hardening co-efficient ‘n’ was calculated using the following Meyer’s law [10] (Fig. 3) and it was found to be 3 - 3.6, which indicated that the grown crystal belonged to soft material category [11]. This value is in good agreement with the reported values [12].

\[ P = k_1 d^n \]  
\[ \text{Where, } P = \text{applied load in gm,} \]
\[ k_1 = \text{standard hardness value.} \]

The standard hardness value \((k_1)\) is found out from the Fig. 4 (P Vs \(d^n\)). If the material takes some time to revert to the elastic mode after every indentation of the material, a correction ‘\(\chi\)’ is applied to the \(d^n\) value and Kick’s law is related as

\[ P = K_2 (d + x)^2 \]  

From the equation (2) and (3), we get

\[ d^{n/2} = (\frac{k_2}{k_1})^{1/2} d + (\frac{k_2}{k_1}) x \]

The slope of \(d^{n/2}\) versus \(d\) yields \((\frac{k_2}{k_1})^{1/2}\) and the intercept is a measure of \(\chi\) and is shown in Fig. 5.

The fracture toughness \((K_c)\) is given by

\[ K_c = \frac{P}{\beta C^{3/2}} \]  

where \(C\) is the crack length measured from the centre of the indentation mark to the crack tip, \(P\) is the applied load and geometric constant \(\beta = 7\) for Vickers’s indenter. The brittleness index \((B)\) is given by

\[ B = \frac{H_v}{K_c} \]  

From the values of \((H_v)\) and \((K_c)\), the brittleness index is found to be 0.015331 MNm\(^{-3}\).

The Yield strength \((\sigma_v)\) of the of the material can be calculated using the relation

\[ \sigma_v = \frac{H_v}{2.9} \{1 - (2 - n)\} \left[ \frac{12.5(2 - n)}{1 - (2 - n)} \right]^{2-n} \]  

All the calculated mechanical parameters are listed in the Table 2.

4. CONCLUSION
The Single crystals of Lithium Potassium sulphate were grown from aqueous solution by slow evaporation technique. The mechanical strength of grown LKS crystal was found by Vickers micro hardness measurements. The micro hardness study reveals that the hardness number increases with increasing applied load. The work hardening coefficient showed that the grown crystal belonged to the soft material category. The stiffness constant \((C_{11})\), Brittles index \((B)\), and
the yield strength (Kc) are calculated for the grown crystal. The stiffness constant of LKS increases with increasing the applied load.

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Fig-2. Plot of Load P Vs Stiffness constant

Fig-3. Plot of log d Vs log P

Fig-4. d^n Vs P Fig. 5 Plot of d Vs d^{n/2}
Fig-1. Plot of P Vs Hv

Fig-2. Plot of Load P Vs Stiffness constant

Fig-3. Plot of log d Vs log P
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Table-1. The mechanical parameters of LKS single crystals.

| Parameters       | Values       |
|------------------|--------------|
| n                | 3 - 3.36     |
| k1 (kg/mm)       | 0.01193      |
| k2 (kg/mm)       | 5.4*10^-3    |
| $H_p$            | 50.6         |
| Pm               | 100          |
| Ps               | 10           |
| Kc (MNm^-3/2)    | 0.015331     |
| B (m^-1/2)       | 3300.71      |
| $H_B$ (MPa)      | 872.41       |

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