



GROWTH, STRUCTURAL AND MECHANICAL STUDIES OF SOLUTION GROWN STRONTIUM PHTHALATE SINGLE CRYSTALS

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Abstract:

High quality Strontium Phthalate single crystals were grown by slow evaporation solution growth method at room temperature. Powder X-Ray diffraction and Mechanical characterizations of slow evaporation grown single crystals of Strontium Phthalate are analyzed in this article. Mechanical properties such as Vicker's microhardness number, work hardening index, standard hardness value, Yield strength, fracture toughness, brittleness index and elastic Stiffness constant values are determined using Vicker's microhardness tester.

Key Words: Powder X-Ray Diffraction, Work Hardening Coefficient, yield Strength & Stiffness Constant

1. Introduction:

Organic materials can exhibit higher nonlinear optical efficiencies due to their higher optical susceptibilities and large electro optic coefficient [1]. Crystals of those materials are used in Optoelectronic applications [2-4]. The study of growth, XRD and Microhardness characterizations of Strontium Phthalate single crystals were analysed and reported in this article. The sample crystals were grown by slow evaporation method. The sample crystals of Strontium Phthalate were undergone structural and mechanical studies by using powder X-Ray diffractometer and Vicker's Microhardness Tester. The hardness of a crystal is generally defined as its resistance to structural breakdown under applied force or stress. Mechanical properties such as Vicker's microhardness number, work hardening index, standard hardness value, Yield strength, fracture toughness, brittleness index, and elastic Stiffness constant values give valuable information on the physical strength and deformation characteristics of a material [5]. The chemical forces in a crystal resist the motion of dislocations as it involves the displacements of atoms. This hardness is the intrinsic hardness of a crystal. The hardness properties are associated to the structure of the crystal material and hardness studies are carried out to understand about the plasticity of the crystal [6]. Microhardness studies on various crystals using Vicker's indenter have been reported by many researchers [7-8]. So in this study, the various hardness parameters were determined for Strontium Phthalate crystal using Vicker's microhardness tester.

2. Experimental Procedure:

Crystal Growth: Slow evaporation solution growth method was used to grow Strontium Phthalate single crystals. The saturated solution of a mixture of Phthalic acid and Strontium Chloride in the stoichiometric ratio of 1:1 was prepared with doubly distilled water. The solution was stirred constantly for about 9 hours using a magnetic stirrer. Then the solution was filtered and kept at room temperature (about 30 °C). The solution was permitted to evaporate the water slowly into the atmosphere. After 3-4 weeks, Strontium Phthalate crystals were obtained from the mother solution. The harvested crystals were recrystallised to get high quality Strontium Phthalate single crystals.

3. Results and Discussion:

3.1 Powder X-Ray Diffraction Analysis:

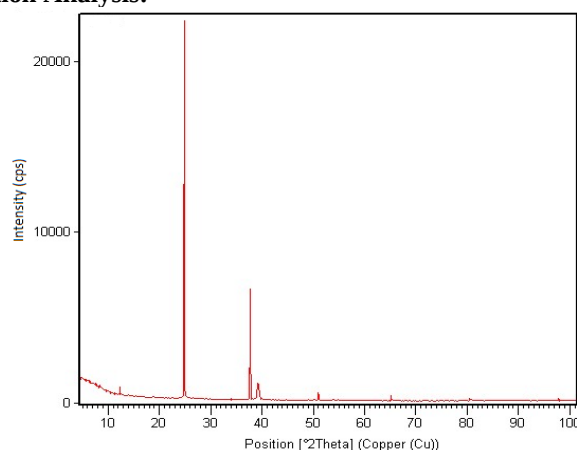


Figure 1: X-Ray diffraction pattern of TPA

The X-Ray diffraction pattern of Strontium Phthalate crystal is recorded using a RICH SEIFERT X-Ray powder diffractometer by $\text{CuK}\alpha$ ($\lambda=1.5406 \text{ \AA}$) radiation. The sample is scanned for a 2θ range of $5^\circ - 100^\circ$ at a scanning rate of $1^\circ/\text{minute}$. All the observed reflections were incorporated and which are shown in figure 1. The sharp and well defined Bragg's peaks at specific 2θ angle confirm the high crystallinity and purity of Strontium Phthalate crystals[9]. The highest peak intensity of 22500 cps was recorded at 25° of 2θ .

3.2 Mechanical Studies:

Mechanical properties are essential factors for the fabrication of optoelectronic devices. Microhardness Studies were conducted using Lietz Wetzler microhardness tester with Vicker's diamond pyramidal indenter on Strontium Phthalate crystal specimen grown by slow evaporation method. The indentations were made for various loads of 25g, 50g and 100g with a constant indentation time interval of 25 seconds.

The values of Vicker's hardness number (H_v) was calculated for different loads by the relation,

$$H_v = \frac{1.8544P}{d^2} \text{ kg/mm}^2$$

Where P is the applied load in kg and d is the mean diagonal length of the indenter impression in mm. Figure 2 shows the variation of Vicker's hardness number H_v with the applied load P. It is observed that the hardness number increases with the increasing load which is termed as reverse indentation size effect (ISE). So the material is suitable for device fabrication [10].

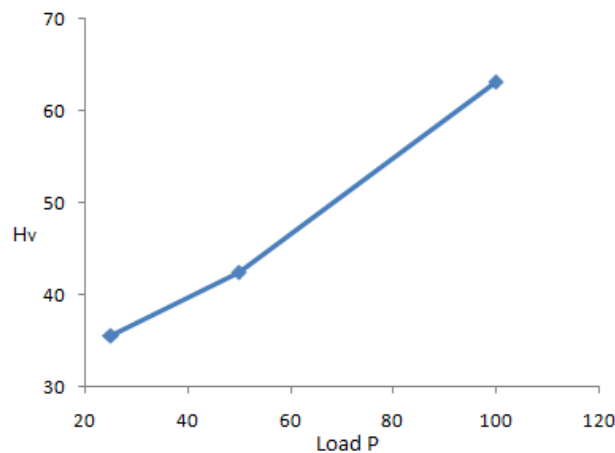


Figure 2: Plot between load P and Hv

According to Meyer's hardness analysis, the relation between load P and indentation length d is given by [11],

$$P = k_1 d^n$$

Or $\log P = \log k_1 + \log d^n$
 $\log P = \log k_1 + n \log d$

Where P is the applied load, d is the observed diagonal length of indentation and n is the Meyer's microhardening index or work hardening coefficient. Meyer's microhardening index n was determined from the slope of the curve drawn between $\log d$ and $\log P$ (Figure 3). The standard hardness k_1 was determined from the intercept of $\log k_1$.

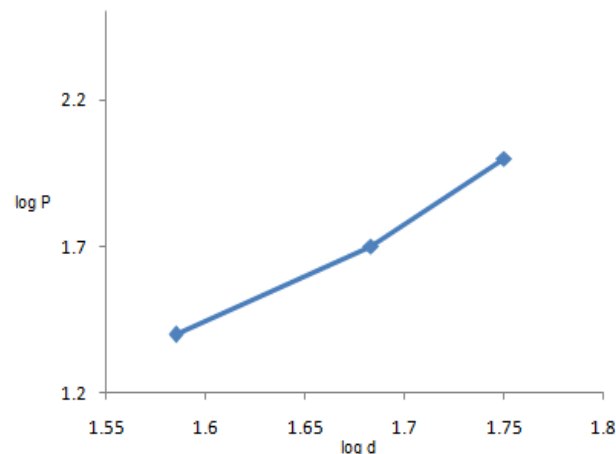


Figure 3: Plot between log d and log P

According to Onitsch, for hard materials 'n' lies between 1 and 1.6 and it is more than 1.6 for soft materials[12]. The value of work hardening coefficient(n) of Strontium Phthalate crystal was found to be 3.78, which shows that the material is a soft material.

After the removal of applied load, the material takes some interval of time to revert to the elastic mode. So a correction of x is included to the observed value d . The Kick's law is given by

$$P = k_2(d+x)^2$$

Substituting value of P, we get

$$k_1 d^n = k_2(d+x)^2$$

$$d^n = \left(\frac{k_2}{k_1}\right)(d+x)^2$$

$$d^{n/2} = \left(\frac{k_2}{k_1}\right)^{1/2}(d+x)$$

Or

$$d^{n/2} = \left(\frac{k_2}{k_1}\right)^{1/2}d + \left(\frac{k_2}{k_1}\right)^{1/2}x$$

The above equation is the equation of a straight line. Figure 4 shows the curve drawn between d and $d^{n/2}$. The slope $\left(\frac{k_2}{k_1}\right)^{1/2}$ and the intercept $\left\{\left(\frac{k_2}{k_1}\right)^{1/2}x\right\}$ were calculated from the graph. By substituting the value of k_1 , k_2 was determined. From the intercept, the correction value x has been calculated.

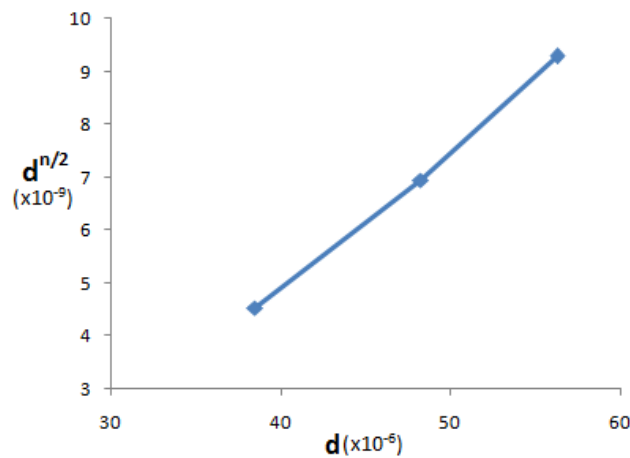


Figure 4: Plot between d and $d^{n/2}$

The fracture toughness (K_c) is given the relation [13],

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

Where C is the cracklength measured from the centre of the indentation mark to the crack tip, P is the applied load and β is the geometrical constant which depends upon the indentation geometry. For Vicker's indenter, $\beta = 7$. The fracture toughness (K_c) of Strontium Phthalate crystal was obtained from the above formula.

The Brittleness index (B_i) of Strontium Phthalate crystal was calculated for various loads by the following relation,

$$B_i = \frac{H_v}{K_c}$$

Figure 5 is the plot drawn between load P and Brittleness index B_i , which shows the decrease in Brittleness index (B_i) with the increase in load P.

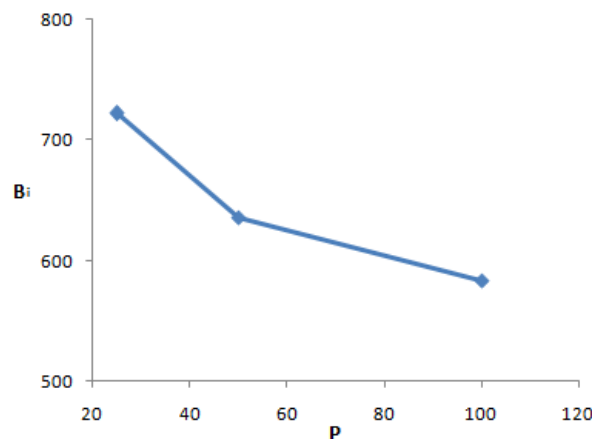


Figure 5: Plot between load P and B_i

The microhardness value correlates with other mechanical properties such as Yield strength (σ_v) and elastic stiffness constant (C_{II}). The Yield strength is an important property for device fabrication which was calculated by the relation,

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

Where H_v is the hardness number and n is the microhardening index. The Yield strength of Strontium Phthalate crystal is 1355.86 MPa. Figure 6 is the graph plotted between load P and Yield strength (σ_v), which shows the variation of σ_v with the varying load P .

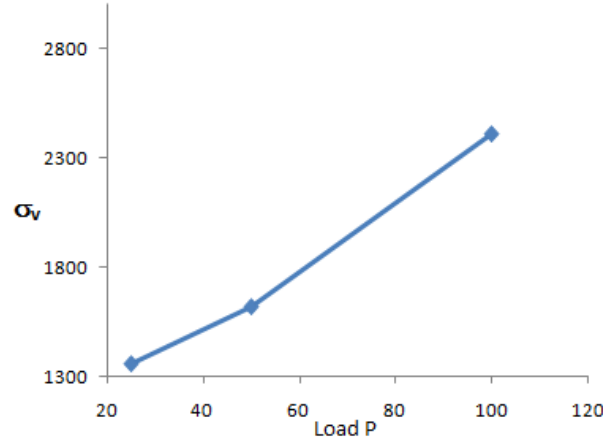


Figure 6: Plot between load P and σ_v

The stiffness constant (C_{II}) of a material determines nature of tightness of the bonding between adjacent atoms. The C_{II} for different loads has been determined using Wooster's empirical formula [14],

$$C_{II} = H_v^{7/4}$$

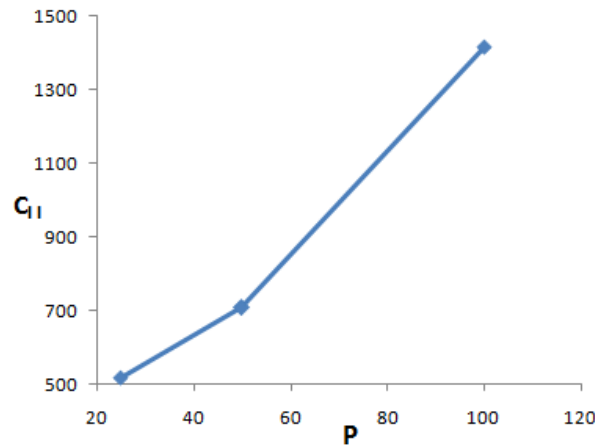


Figure 7: Plot between load P and C_{II}

Figure 7 is the curve drawn between load P and C_{II} . It shows the increase in stiffness constant with the increase in load. High value of C_{II} indicates that the binding forces between the atoms and ions are quite strong [15].

Table 1: Values of microhardness parameters of Strontium Phthalate crystal.

Hardness Parameters	Calculated Values		
n	3.78		
k_1 in kg/m	12.023×10^{-3}		
k_2 in kg/m	0.873×10^{-9}		
α in m	7.2356×10^{-3}		
	P=25g	P=50g	P=100g
K_c in $\text{MNm}^{-3/2}$	0.0493	0.0688	0.1078
B_i in $\text{m}^{-1/2}$	722.11	636.23	586.16
σ_v in MPa	1355.86	1618.61	2406.91
C_{II} in Pa	518.85	707.43	1416.63

4. Conclusion:

The X-Ray diffraction pattern of Strontium Phthalate crystal confirms the grown crystals are pure and high quality crystallinity. Mechanical properties such as Vicker's microhardness number, work hardening

coefficient, Yield strength, elastic stiffness constant, brittleness index, standard hardness and fracture toughness values were determined using Vicker's microhardness tester and tabulated. It was observed that the hardness number increases with increasing load, termed as reverse ISE. The value of work hardening coefficient(n) is 3.78 which shows that the title crystal is a softer material. The high value of stiffness constant indicated that the binding forces between atoms are quite strong. Hence the material is suitable for Optoelectronic device fabrications.

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