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# GROWTH, STRUCTURAL AND MECHANICAL STUDIES OF THIOUREA PHTHALIC ACID (TPA) SINGLE CRYSTALS BY X-RAY POWDER DIFFRACTION AND VICKER'S MICROHARDNESS TESTING

**M. Suresh Kumar*[1], S. Krishnan[2], G.V. Vijayaraghavan[3]**
[1] *Department of Physics, AMET University, Kanathur 603112, Chennai, India*
[2] *Department of Physics, RKM Vivekananda College, Chennai600004 , India*
[3] *Department of Physics, B. S. Abdur Rahman Crescent University, Chennai 600048, India*

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## ABSTRACT
Good quality Thiourea Phthalic acid (TPA) single crystals were grown by slow evaporation solution growth technique at room temperature. X-Ray powder diffraction and Mechanical characterizations of slow evaporation grown single crystals of Thiourea-Phthalic acid (TPA) 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.

**KEYWORDS**: X-Ray Powder diffraction, Yield strength and Stiffness constant.

## I. INTRODUCTION
Organic materials can exhibit higher nonlinear optical efficiencies due to their large electro optic coefficient and higher optical susceptibilities [1]. Crystals of such materials are used in Optoelectronic applications[2-4]. The study of growth, XRD and Microhardness characterizations of Thiourea Phthalic acid single crystals were analysed and reported in this article. The sample crystals were grown by slow evaporation method. The sample crystals of TPA were undergone structural and mechanical studies by using X-Ray powder 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 indentor have been reported by many researchers[7-8]. So in this study, the various hardness parameters were determined for TPA crystal using Vicker's microhardness tester.

## II. EXPERIMENTAL PROCEDURE
### Crystal Growth
Slow evaporation solution growth technique was used to grow TPA crystals. The saturated solution of a mixture of Thiourea and Phthalic acid in the stoichiometric ratio of 1:1 was prepared with doubly distilled water. The solution was stirred constantly for about 5 hours using a magnetic stirrer. After that the solution was filtered and kept at room temperature (about 30 $^{o}$ C). The solution was permitted to evaporate the water slowly into the atmosphere. After 3 weeks, TPA crystals were obtained from the mother solution. The collected crystals were recrystallaised to get good quality TPA single crystals.

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## III. RESULTS AND DISCUSSION

**X-Ray Powder diffraction Analysis**

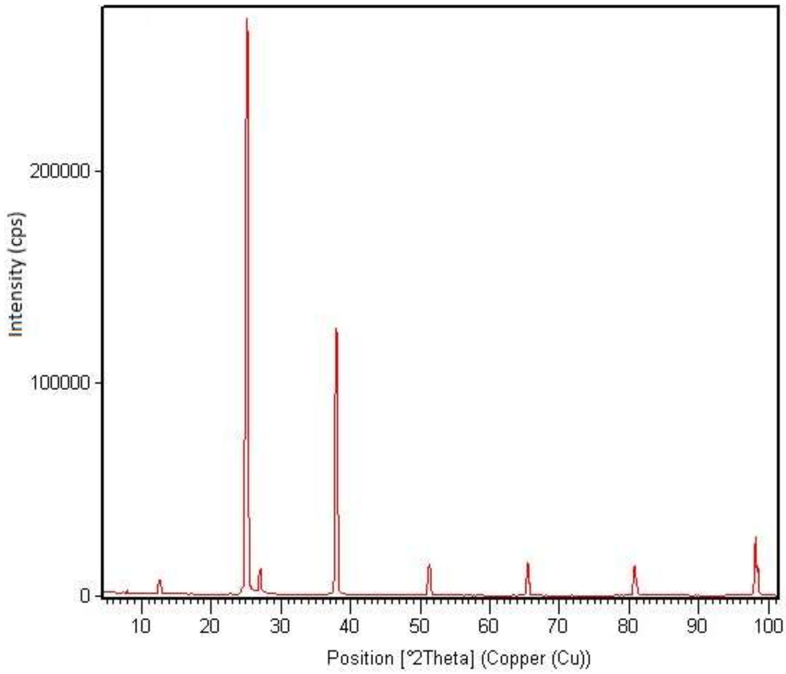


**Figure 1:** ***X-Ray diffraction pattern of TPA***

The X-Ray diffraction pattern of TPA crystal is recorded using a RICH SEIFERT X-Ray powder diffractometer by CuKα (λ=1.5406 Å) radiation. The sample is scanned for a 2θ range of 5° - 100° at a scanning rate of 1°/minute. All the observed reflections were included and which is shown in figure 1. The sharp and well defined Bragg's peaks at specific 2θ angle confirm the high crystallanity and purity of TPA crystals[9]. The highest peak intensity of 295000 cps was recorded at 25.5° of 2θ.

**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 indentor on TPA 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,

$$H_v = \frac{1.8544P}{d^2} \ \ kg/mm^2 \qquad (1)$$

Where P is the applied load in kg and d is the mean diagonal length of the indentor 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].

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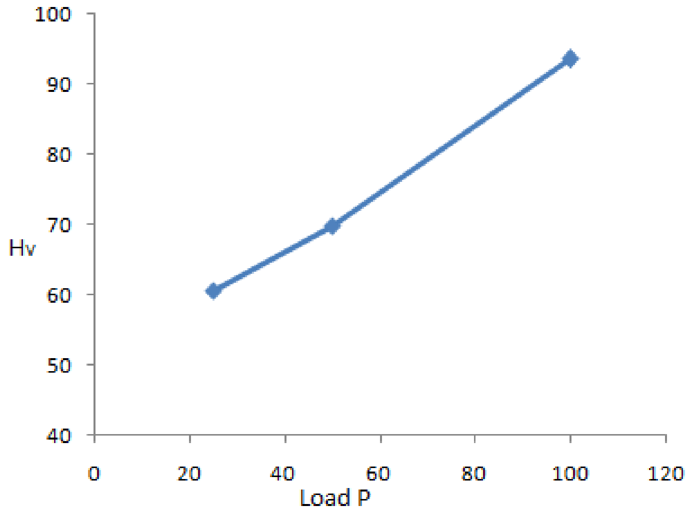


**Figure 2:*Plot between load P and Hv***

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

$$P = k_1\, d^n \qquad (2)$$

Or
$$\log P = \log k_1 + \log d^n$$
$$\log P = \log k_1 + n \log d \qquad (3)$$

Where P is the applied load, d is the observed diagonal length of indentation and n is the Meyer's microhardenning 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 $\log k_1$.

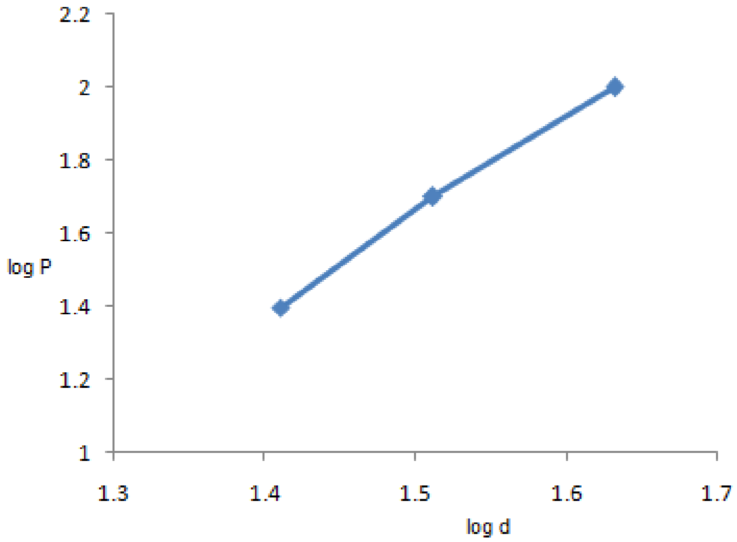


**Figure 3:*Plot between log d and log P***

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 TPA crystal was found to be 2.76, 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

$$P = k_2(d + x)^2 \qquad (4)$$

Substituting value of P, we get
$$k_1 d^n = k_2(d + x)^2$$
$$d^n = \left(\frac{k_2}{k_1}\right)(d + x)^2$$

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

Or
$$d^{n/2} = \left(\frac{k_2}{k_1}\right)^{1/2} d + \left(\frac{k_2}{k_1}\right)^{1/2} x \qquad (5)$$

The equation (5) 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.

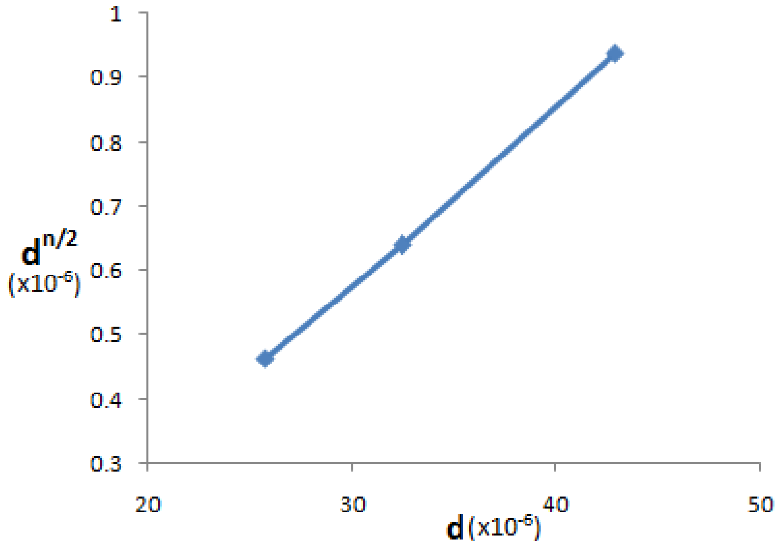


**Figure 4:*Plot between d and $d^{n/2}$***

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

$$K_c = \frac{P}{\beta C^{3/2}} \qquad (6)$$

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

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

$$B_i = \frac{H_v}{K_c} \qquad (7)$$

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.

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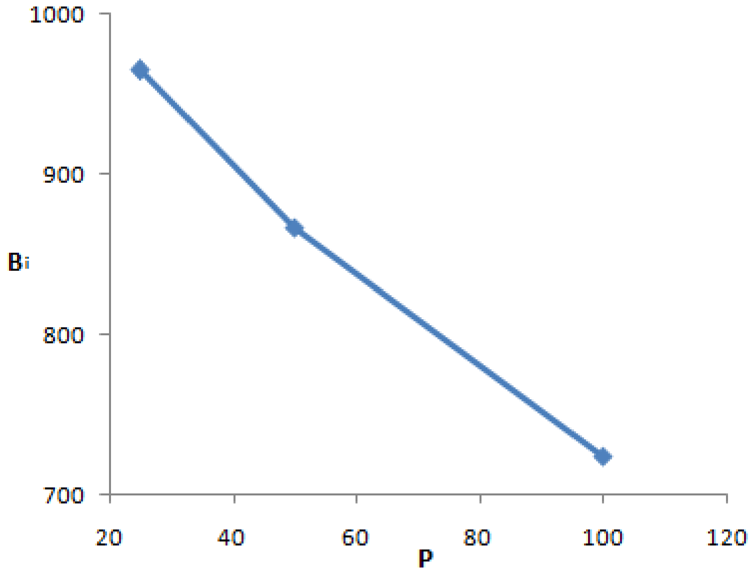


**Figure 5:*Plot between load P and $B_i$***

The microhardness value correlates with other mechanical properties such as Yield strength $(\sigma_v)$ and elastic stiffness constant $(C_{11})$. The Yield strength is an important property for device fabrication which was calculated by the relation,

$$\sigma_v = \frac{H_v}{2.9}\left\{[1-(2-n)]\left[12.5(2-n)/\left(1-(2-n)\right)\right]^{2-n}\right\} \qquad (8)$$

Where $H_v$ is the hardness number and n is the microhardenig index. The Yield strength of TPA crystal is 476.59 MPa. Figure 6 is the graph plotted between load P and Yield strength $(\sigma_v)$ , which shows the variation of $\sigma_v$ with the varying load P.

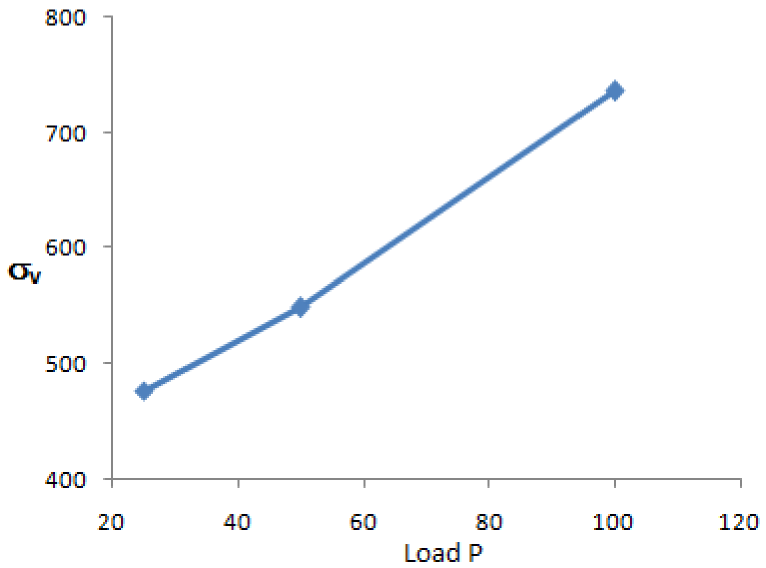


**Figure 6:*Plot between load P and $\sigma_v$***

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],

$$C_{II} = {H_v}^{7/4} \qquad (9)$$

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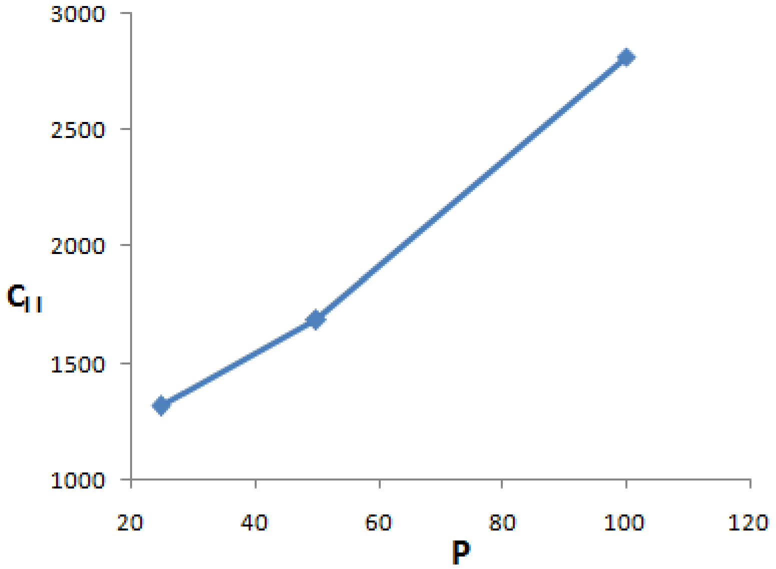


**Figure 7:** ***Plot between load P and $C_{II}$***

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 TPA crystal***

| Hardness parameters | Calculated values | | |
|---|---|---|---|
| n | 2.76 | | |
| $k_1$ in kg/m | $12.735\times10^{-3}$ | | |
| $k_2$ in kg/m | $9.5609\times10^{-6}$ | | |
| $x$ in m | $10.584\times10^{-6}$ | | |
| | P=25g | P=50g | P=100g |
| $K_c$ in $MNm^{-3/2}$ | 0.0628 | 0.0805 | 0.1293 |
| $B_i$ in $m^{-1/2}$ | 964.97 | 867.09 | 723.90 |
| $\sigma_v$ in MPa | 476.59 | 548.95 | 736.13 |
| $C_{II}$ in Pa | 1316.22 | 1685.57 | 2816.65 |

## IV. CONCLUSION

The X-Ray diffraction pattern of TPA crystal confirms the grown crystals are pure and high quality crystallinity. Mechanical properties such as Vicker's microhardness number, work hardening coefficient, Yield strength, 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) showed that the crystal is a softer material. The value of stiffness constant indicated that the binding forces between atoms and ions are quite strong.

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