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Growth and Mechanical Characterization of Thiourea Single Crystals Grown By Inexpensive Slow Evaporation Method

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Abstract: Single crystals of Thiourea(NH_2CSNH_2), an organic nonlinear optical material have been grown by simple and inexpensive slow evaporation technique at room temperature. The grown crystals are hexagonal and tetragonal in shape and average sizewere 40mm×28mm×1mm to 1.5mm×1.5mm×0.5mm.The mechanical characterization of grown crystals were studied and their Vicker's Micro hardness number(Hv), Fracture toughness (Kc),Elastic stiffness constant (C_{11}) and yield strength (σ) were determined. The classical Meyer's law and Proportional Specimen Resistance (PSR) model is used to analyze the micro hardness behavior. Indentation SizeEffect (ISE) was observed at low indentation test load variations of microhardness number (Hv) with variable loads are non systematic for samples.

Key words: Thiourea single Crystals, Vicker's Microhardness, Meyer's law, Indentation Size Effect, Proportional Specimen Resistance.

I. INTRODUCTION

The field of crystal growth has received great attention and vital importance not only for beauty of crystals but due to the applications of single crystals in various fields of science and technology. Single crystals of pure Thiourea are being used widely in electronic and optoelectronic industries. Thiourea crystals exhibit piezoelectric effect, which is utilized in Infrared (IR) and ultraviolet (UV) Scanning Electron Microscopy (SEM) detection and imaging(1). Thiourea(NH_2CSNH_2) is one of the organic material that have high potential for these applications(2-3). This material has good chemical flexibility to provide nonlinearity of organic material and strong mechanical properties of inorganic nature to form semi organic materials. Therefore the intense study on Thiourea is required.

II. EXPERIMENTAL

The Thiourea(NH_2CSNH_2) crystals have been grown from saturated solution by slow evaporation technique. The saturated solution was prepared by taking 100ml of double distilled water in a beaker and finally powdered 18.75grams of Thiourea. The substance was added slowly and continuously till it gets completely dissolved. For availing the supersaturated solution the solution was stirred well with the magnetic stirrer and process was continued until the last pinchof the substance was dissolved. Then the solution was filtered using filter paper. The filtered solution was kept at room temperature without any disturbance. The single crystals were harvested between 10 to 15 days. Photographs of pure Thiourea single crystals are shown infig1 (a) and (b).

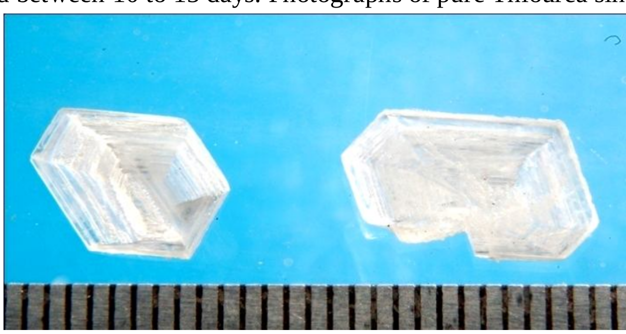

Figure:1(a)

Figure:1(b)

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III. MECHANICAL CHARACTERIZATION

Mechanical strength of the materials plays a key role in the device fabrication. Vickers's hardness is one of the important deciding factor in selecting the processing (cutting, grinding, polishing) steps of bulk crystals in fabrication of devices based on crystals. In the view of device fabrication, calculations of mechanical parameters are very important. This can be done with the help of Vicker's micro hardness studies. Microhardness measurements were done for Thiourea crystals using CLEMEX-HWMMTX7 hardness tester with a Vickers's diamond indenter at room temperature. The two diagonals of the indentation left on the surface of the material after the removal of the load were measured using software and their average was calculated. The area of the sloping surface of the indentation was calculated. The Vicker's hardness is the quotient obtained by dividing the Kg load by the square mm area of indentation.

$$H_v = \frac{2p\sin\frac{136}{2}}{d^2}$$

$$H_v = 1.544\frac{P}{d^2}$$

Where, H_v=Vickers hardness number
P= Load in Kg
d= arithmetic mean of the two diagonals

load P(10^{-3}kg)	Average d(μm)	logP	log d	Average Hv(Kg/mm^2)
25	57.16875	1.39794	1.757159	14.5
50	83.525	1.69897	1.921816	13.625
100	113.075	2	2.053367	15
200	124.5875	2.30103	2.095474	24.25

Table-1: vicker's microhardness number

For obtaining more precise values, the average microhardness values were considered for each load. 10 samples were used for observations. The results of vicker's microhardness number are summarized in Table-1. Fig:2(a) is the plot of variation in Hv with load P and Fig:2(b) is the plot of log P Vs. log d.

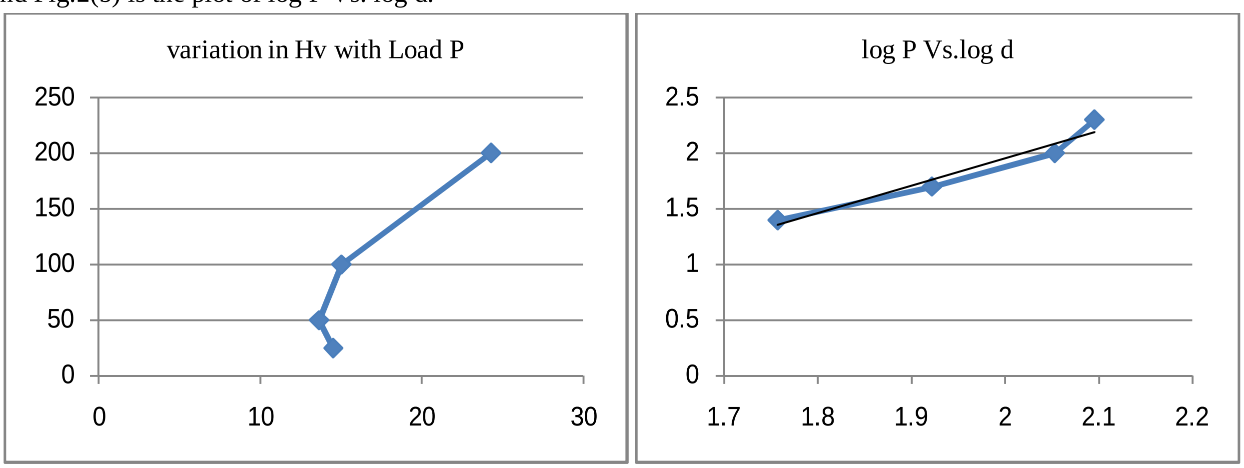

Fig:2(a):variation in Hv with Load P Fig:2(b):log P Vs.log d

The Meyer's index number **n** is calculated from the Meyer's law, which relates the load and indentation diagonal length.

$P = kd^n$

$\log P = \log k + n \log d$

Where k is the material constant and n is the Mayer's index. The above relation indicates that Hv should increase with the increase in P if n>2 and decreases with P if n<2. The Meyer's index (n) is found to be 2.478. The Thiourea material is confirmed with large amount of mechanical strength which is suitable for device fabrication.

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The elastic stiffness constant (C_{11}) was calculated by Wooster's empirical relation$C_{11}=H_v^{7/4}$. The calculated stiffness constant for different loads were tabulated and summarized in Table:2. The Yield strength for different load is calculated using the relation, σ_v =[Hv/2.9][1-(n-2)]{12.5(n-2)/1-(n-2)}$^{(n-2)}$ and is summarized in Table:2.

Yield strength σ_v(MPa)	Elastic stiffness C_{11}	Fracture toughness K_c 10^6 $Kgm^{-3/2}$
82.05992689	107.7442256	0.008262356
77.10803475	96.62486367	0.009357229
84.88957954	114.3298683	0.011880975
137.2381536	264.9998024	0.020545667

Table-2: Mechanical properties of Thiourea crystals

The crack length is measured from the centre of indentation mark to the crack end. Here, the crack length (*l*) is the average of two crack lengths for each indentation. Resistance to fracture indicates the toughness of material. The fracture mechanics of the indentation process gives an equilibrium relation for a well-developed crack extending under the centre loading condition,

$K_c = \frac{P}{\beta_o l^{3/2}}$, $l > \frac{d}{2}$

Where β_o is the indenter constant, equal to 7 for the Vicker's diamond pyramid indenter. The calculated values are summarized in Table: 2

Conclusion:A Non Linear Optical single crystal, Thiourea was grown successfully by slow evaporation solution growth method. The single crystals were harvested between 10 to 15 days. The grown crystals are hexagonal and tetragonal in shape and average size were 40mm×28mm×1mm to 1.5mm×1.5mm×0.5mm. Vicker's microhardness measurement reveals the soft nature of the crystal and shows reverse ISE. The mechanical parameters like fracture toughness (Kc), elastic stiffness constant(C11) and yield strength (σv) were calculated and summarized in table.

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