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Growth, Optical and Mechanical properties of a novel organic Single Crystal of Glycine doped with Potassium Iodide

KEYWORDS: Slow evaporation solution technique, Single crystal XRD, Optical absorption, Band gap energy, Vicker's microhardness, Meyer's index number and Elastic stiffness constant.

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ABSTRACT *Glycine doped with Potassium Iodide (GLPOI) in different ratio, a new amino acid organic single crystal was synthesized. Single crystals of Glycine Potassium Iodide (GPI) with good degree of transparency were grown from aqueous solution by slow evaporation solution technique (SEST). Good quality transparent crystal of various size was harvested over a period of 65 days. The grown crystals which were characterized by Single crystal X-ray diffraction analysis reveals that the GPI crystal belongs to hexagonal system. The optical absorption studies show that the crystal is transparent in the entire visible region and the optical band gap energy was found. In order to analyze the mechanical properties of the grown GLPOI crystal was subjected to Vicker's microhardness indentation analysis. From the mechanical analysis, it was found that the GLPOI crystal belongs to soft material category and various mechanical parameters were also calculated.*

INTRODUCTION

The family of organic or semi-organic amino acids show interesting optical properties [1,2]. Amino acids and their complexes belong to a family of organic materials that have been considered for photonic applications [3]. Out of 20 amino acids glycine is the simplest of all. Glycine family crystals have been subjected to extensive research by several researches for their efficient optical properties [4-7]. In the present study, a systematic investigation has been carried out and we report growth, X-ray diffraction study, optical, and mechanical properties of Glycine doped with Potassium Iodide (GLPOI) single crystals.

EXPERIMENTAL DETAILS

All reagents used in the synthesis were of analytical grade. Glycine Potassium Iodide (GLPOI) was synthesized by the reaction between Glycine and Potassium Iodide by slow evaporation technique. Glycine doped with Potassium Iodide in different ratio of 5:2, 5:3, 5:4. The amount of Glycine salt was calculated and dissolved in deionized water for two and half hours to get complete dissolved solution. Then the appropriate amount of Potassium Iodide was added with a few drops of H_2SO_4 to the solution. The resultant solution was continuously stirred well using a temperature controlled magnetic stirrer for 6 to 7 hours to yield a homogenous mixture of solution.

The complete dissolved solution was filtered using micro filter paper and taken in a Petri dish. It was optimally closed using a perforated polythene paper and kept in undisturbed conditions. The solution was allowed to evaporate at room temperature. After a growth period of 65 days, a well developed and optically transparent GLPOI single crystals of various size were harvested and the photo of as grown single crystals of GLPOI was presented in fig.1.

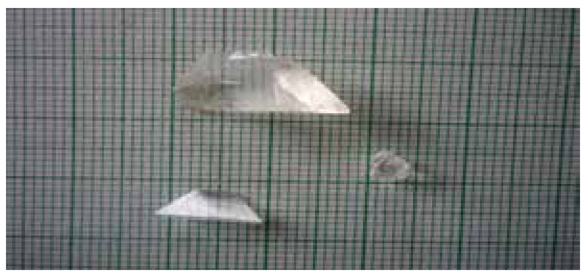

Fig.1 – Photograph of as - grown single crystal of GLPOI

RESULTS AND DISCUSSION

Single crystal X-ray diffraction

Single crystal X-ray diffraction study was carried out on the as grown GLPOI single crystal. The crystal data collection was performed by a Bruker APEX II CCD area detector diffractometer equipped with graphite-monochromatized MoKα radiation (λ = 0.7103 Å). The XRD study reveals that GLPOI belongs to hexagonal system. The unit cell parameters of grown GLPOI single crystal are listed in Table 1.

Table 1 : Unit cell information of GLPOI

Parameter	Value (in different ratio)		
	5:2	5:3	5:4
a	6.9372(7) Å	6.9477(6) Å	6.9785(5) Å
b	6.9372(7) Å	6.9477(6) Å	6.9785 (5) Å
c	5.2397(5) Å	5.4458(7) Å	5.4579(8) Å
α	90°	90°	90°
β	90°	90°	90°
ν	120°	120°	120°
Crystal System	Hexagonal	Hexagonal	Hexagonal

Optical absorption of GLPOI

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The optical absorption spectrum of GLPOI single crystal was recorded in the wavelength region ranging from 200 – 2500 nm which shows that absorption was very less in the entire visible region and part of IR region as shown in the fig.2. For optical fabrication, the crystal should be highly transparent over a considerable region of wavelength [8-9]. The UV cut-off wavelength(λ) of the GLPOI crystal of different ratio of 5:2, 5:3, 5:4 was found to be at 361, 339, and 311 nm, respectively, which makes it is a potential material for optical device fabrications. Using the relation $E_g = 1240/\lambda$, the band gap energy the GLPOI crystal of different ratio of 5:2, 5:3, 5:4 was found to be 3.43 eV. 3.66 eV, and 3.99 eV respectively.

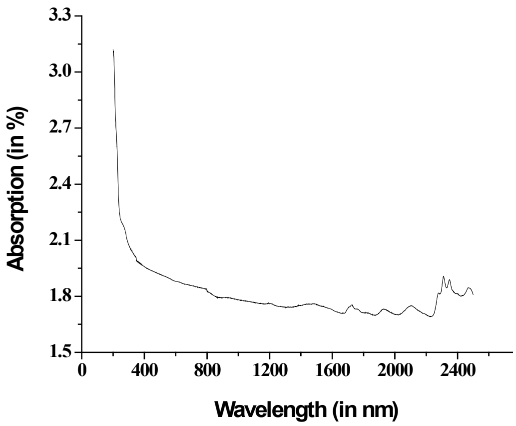

Fig. 2 UV – VIS – NIR spectrum of GLPOI single crystal (5:3 ratio)

Microhardness analysis
One of the important properties of any device material is its mechanical strength, represented by its hardness. The low load hardness (less than 200gm.) is called microhardness. Microhardness studies have been carried out on the polished GLPOI crystal using Leica, Reichert polyvar 2, MET Vickers microhardness tester fitted with a diamond indentor.

The Vickers hardness number (H_V) was calculated using the standard relation:

$H_V = 1.8544 \, P / d^2$

Where P is the applied load in Kg, d is the diagonal length of the indentation impression in micrometer and H_V is in Kg / mm². All the readings are recorded and listed in table 2. The relation between hardness number (H_V) and load (P) for GPI crystal is as shown in fig.3.

Table 2: Values of H_V of GLPOI for various load

Load (P) gm	Vickers Hardness number (H_V) Kg / mm²		
	5:2	5:3	5:4
10	11.55	13.86	16.65
15	20.76	23.56	26.06
20	28.69	30.37	33.03
25	34.57	36.45	39.80
30	46.13	48.07	52.03
35	51.06	54.24	57.66
40	57.62	58.34	61.57
45	---	--	68.20

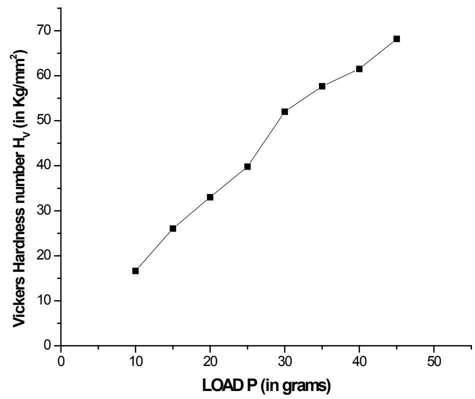

Fig. 3 Hv Vs Load

Higher hardness value of a crystal indicates that greater stress is required to form dislocation thus confirming greater crystalline perfection. [10]. So we have taken the GLPOI crystal of ratio 5:4 for further studies.

The Meyer's index number or work hardening coefficient (n) of the material was calculated from the Meyer's law, which relates the load and size of the indentation or indentation diagonal length.

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

Where k_1 is an arbitrary constant which depend on material, so it is also called as material constant. In order to estimate the value of 'n', the graph is plotted against log P Versus log d as shown in fig.4, which gives a straight line curve, the slope of this straight line gives the value of 'n'. The calculated value of 'n' is 5.99. The material constant k_1 was calculated using the relation

$k_1 = P/d^n$ and it was found to be 25.42×10^{-9}.

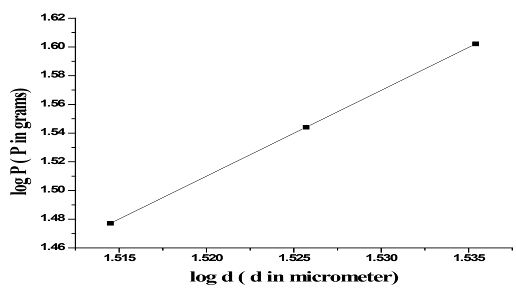

Fig. 4 log P Vs log d

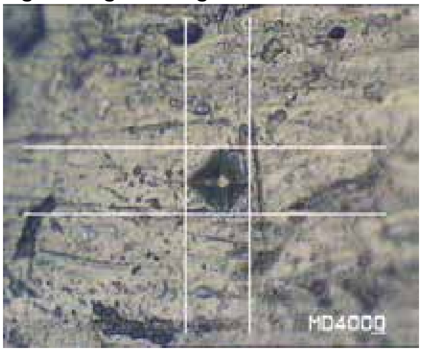

Fig.5 Indentation mark for 25 gm

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From the expression $H_V = b\ P^{(n-2)/n}$, it is inferred that H_V should increase with increase of P if n > 2 and decrease with same if n < 2. The calculated value of n for GLPOI crystal agrees well with the expression. The indentation mark of GLPOI Crystal for 25 gm was recorded as shown in fig. 5. According to Onitsch[11] and Hanneman [12], if n should lie between 1 to 1.6 for hard materials and above 1.6 for soft materials[13]. Thus the grown GLPOI crystal belongs to the soft material category.

The elastic stiffness constant (C_{11}) was calculated by using Wooster's empirical relation as

$C_{11} = H^{7/4}$ [14]. The calculated stiffness constant was found to be 1.6185 Kg / mm^2 for the load of 45gm.

The resistance pressure is defined as a minimum level of indentation load (W) below which there is no plastic deformation [15]. Hays and Kendall proposed a relationship to calculate the 'W' by the equation: $d^n = (W/k_1) + (k_2 / k_1)\ d^2$

The plot between d^n and d^2 in fig.6, gives a straight line having slope (k_2 / k_1) and intercepts

(W / k_1). From the graph, the slope and intercept were found to be 3.83×10^6 and 1.12×10^9. From these values, the minimum level of indentation load W was calculated as 28.5grams and the value on n was found to be 6.03. From this analysis, it is clear that below 28.5grams, the crystal exhibits elastic property and from 28.5grams, it starts to exhibit plastic property. Beyond 45gm, cracks develop on the smooth surface of the crystal.

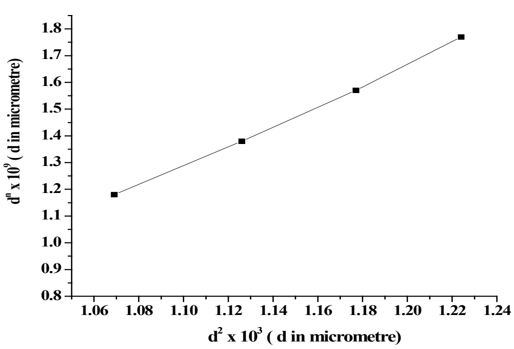

Fig. 6 d^n Vs d^2

The micro-indentation test is a useful method for studying the nature of plastic flow and its influence on the deformation of the material. Also, the hardness of the crystal is dependent on the type of chemical bonding, which may differ along the crystallographic direction [16]. Hardness is one of the important factors in selecting the processing (cutting, grinding and polishing) steps of bulk in fabrication of devices based on the crystals. It is therefore important to study the mechanical properties of NLO or organic crystals [17].

Conclusion

Good quality and optically transparent single crystals of GLPOI of different ratio were grown by slow evaporation technique at room temperature. Single crystal XRD analysis confirmed that the crystals belongs to hexagonal system. Optical absorption study reveals high transparency of the crystal with a UV cut off wavelength and the band gap energy of the crystal was found. Microhardness analysis reveals that the higher hardness value of a crystal indicates that greater stress is required to form dislocation thus confirming greater crystalline perfection. The Meyer's index number (n = 5.99) infers that the grown GLPOI crystal belongs to the soft material category. The elastic stiffness constant (C_{11}) and the minimum level of indentation load (W) of GLPOI single crystal were calculated.

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