Synthesis and Characterization of Potassium Thiourea Nitrate doped L- Alanine Single Crystals

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***Abstract* - Single crystals of Potassium Thiourea Nitrate (KTN) and KTN doped L-Alanine crystals were grown from aqueous solutions by slow evaporation technique. The cell parameters and crystallinity of KTN and KTN doped L-Alanine crystals were analyzed by single crystal X- Ray Diffraction. The grown crystals were subjected to Powder X-Ray Diffraction technique to identify the diffraction phase of the grown crystals. The functional groups present in the KTN and KTN doped L-Alanine crystals were confirmed by FTIR analysis. The UV-Visible transmittance spectrum of KTN and KTN doped L-Alanine crystals were recorded to analyses the optical transparency and optical energy gap. The mechanical properties of the grown crystals were studied using Vickers Micro Hardness tester.**

***Keywords* - L-Alanine, Thiourea, XRD, FTIR, UV-Vis.**

I. INTRODUCTION

Crystal growth plays an important role in modern technology. Crystals are the solids in which the atoms are arranged regularly in a space lattice with specific geometry. Crystal growth is an interdisciplinary subject covering physics, chemistry, material science, chemical engineering, metallurgy, crystallography, mineralogy, etc. [1].The strong influence of single crystals in the present day technology is evident from the recent advancements in the above mentioned fields. Hence, in order to achieve high performance from the device, good quality single crystals are needed. Growth of single crystals and their characterization towards device fabrication have assumed great impetus due to their importance for both academic as well as applied research. [2].

In the present work, Potassium Thiourea Nitrate (KTN) and Potassium Thiourea Nitrate doped L-Alanine single crystals were grown by slow evaporation method. The grown crystals were characterized by Single crystal and Powder X-Ray Diffraction studies (XRD), Fourier Transform Infrared spectroscopy (FTIR), UV-Visible Spectroscopy and Vickers Microhardness tester.

II. MATERIALS AND METHODS:

The single crystals of KTN and KTN doped L-Alanine were grown by slow evaporation technique at room temperature. The ratio in which the constituent compounds were added in each case was 2:1:0.5 respectively. A saturated solution of the salts were prepared and filtered. The filtered solutions were transferred to Petri Dishes which were closed with thick papers with fine pores in order to minimize the rate of evaporation [3]. The solutions were allowed to evaporate completely and the single crystals of KTN and KTN doped L-Alanine single crystals were harvested. The harvested crystals were shown in Fig.1,2.

Fig 1. KTN Crystal

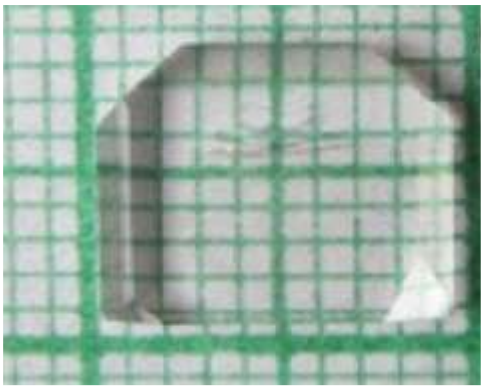

Fig 2. KTN doped L-Alanine Crystal

III. RESULT AND DISCUSSION

The grown crystals were characterized by various characterization viz, Single crystal and Powder X-Ray Diffraction (XRD) analyses, FTIR analysis, UV-Visible spectral study and Vickers Microhardness study.

3.1.Single Crystal XRD Analysis

Single crystal X-Ray Diffraction (XRD) is a non- destructive tool to analyze crystal structure of compounds, which can be grown as single crystal. From the studies, it was found that KTN crystals and KTN doped L-Alanine single crystals belong to Orthorhombic system[4].The lattice parameter values of the grown crystals are listed in table 1.

Table 1: Lattice Parameter values of KTN and KTN doped L-Alanine crystals

CRYSTAL	a (Å)	b (Å)	c (Å)	α =β =γ
KTN	5.55	7.76	8.67	90°
KTN doped L-Alanine	13.756	13.760	9.217	90°

3.2 Powder XRD

X-Ray Diffraction technique is a powerful tool to analyze the crystalline nature of the materials. If the material to be investigated is crystalline in nature, well defined peaks will be observed. The grown crystals were finely powdered and had been subjected to X-Ray Diffractometer employing Cu Kα (1.5405Å) with a scan speed of 1°/min. The prominent peaks obtained in the Powder X-Ray Diffraction patterns (Fig. 3, 4) of the grown crystals confirmed the good crystalline nature.

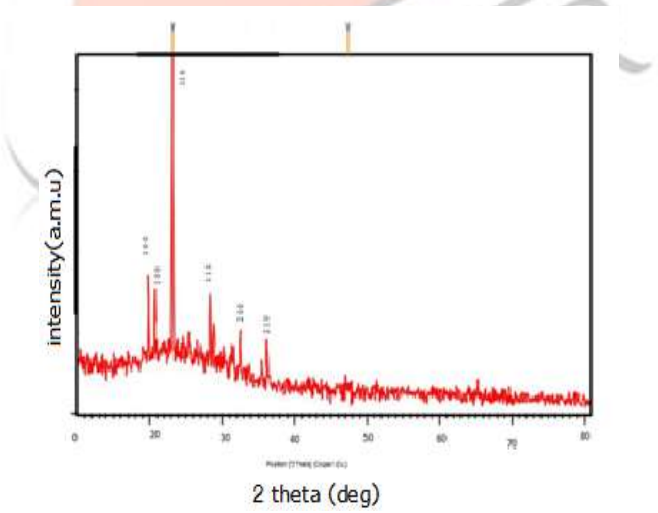

Fig 3.XRD Pattern of KTN Crystals

Table 2: Structural Parameters of KTN Crystals

2θ degree	FWHM degree	hkl	D nm	ρ $\times 10^{14}$ lines/m^2	ε $\times 10^{-4}$
19.8556	0.1476	100	55.378	3.26	6.34
23.1851	0.2952	100	54.845	3.32	6.27
28.3508	0.1476	110	54.507	3.37	6.24
32.5007	0.1968	111	53.745	3.46	6.18
36.2220	0.1476	200	53.433	3.50	6.12
47.3505	0.5904	210	53.215	3.53	5.87

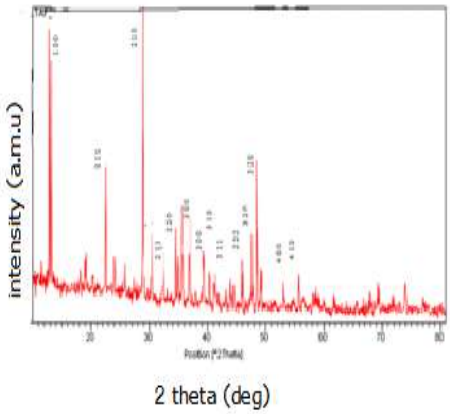

Fig 4.XRD Pattern of KTN L-Alanine doped crystal

2θ Degree	FWHM Degree	Hkl	Dnm	ρ×10^{14} lines/m^2	ε×10^{-4}
19.0587	0.2952	110	55.574	3.24	6.54
24.0379	0.1968	111	55.246	3.28	6.50
28.9607	0.1476	210	54.435	3.37	6.45
30.3756	0.1968	210	54.259	3.40	6.39
32.3743	0.1968	211	54.185	3.41	6.34
36.8682	0.1476	220	52.777	3.43	6.26
40.3207	0.1968	300	52.145	3.59	6.24
44.5286	0.1476	311	51.654	3.60	6.17
46.0108	0.1968	222	51.264	3.68	6.14
47.6565	0.1476	320	51.654	3.75	6.07
48.4804	0.1476	320	51.264	3.81	5.99
53.1145	0.1476	400	50.284	3.95	5.97
55.7110	0.1968	410	50.124	3.98	5.89

Table 3: Structural Parameters of KTN doped L -Alanine Crystals

From the results of the powder XRD of KTN and KTN doped L-Alanine Crystals, the Crystallite Size, Strain and Dislocation density were found and they were tabulated above.

3.3 FTIR Analysis

Fourier Transform Infrared Spectroscopy (FTIR) is an analytical technique used to determine the molecular structure of a material. In order to qualitatively analyze the presence of functional groups in the grown crystals, the FT-IR spectrum were recorded in the range 400-4000 cm^{-1}using FTIR-8400 resolution of 4 cm^{-1}and with the scanning speed of 2 mm/sec with KBr pellets method [5].The functional groups present in the single crystals of KTN and KTN doped L-Alanine single crystals were identified with the help of available data.

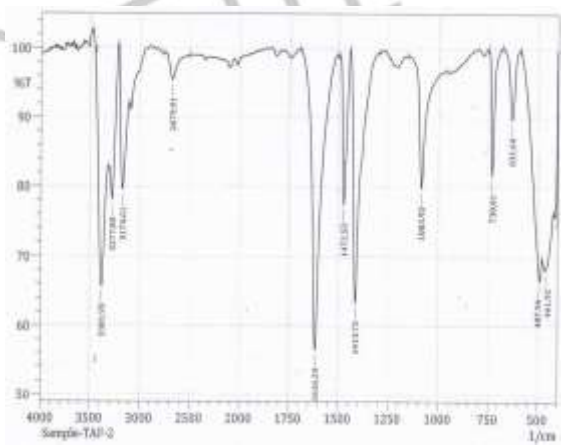

Fig 5. FTIR Spectrum of KTN crystal

The Spectrum of KTN crystal was shown in fig 5.The Vibrational modes of KTN Crystals appearing at wave numbers 3382, 3278, 3175, 2680, 1616, 1473, 1414 and 1084 cm^{-1} was a strong and wide peak which indicates that there was a –OH on the KTN Crystals. The absorption at 3175 cm^{-1} was attributed to N-H stretching. The peak observed at 3382 and 3278 cm^{-1} were ascribed to NH_2 asymmetric stretching and –OH bending respectively. The peak observed at 1616 and1473 cm^{-1} are attributed to bending vibration of NH_3 and C=C stretching vibrations. The peaks of 488 and 730 cm^{-1} were attributed to aliphatic chloro compounds and Aryl disulfide respectively.

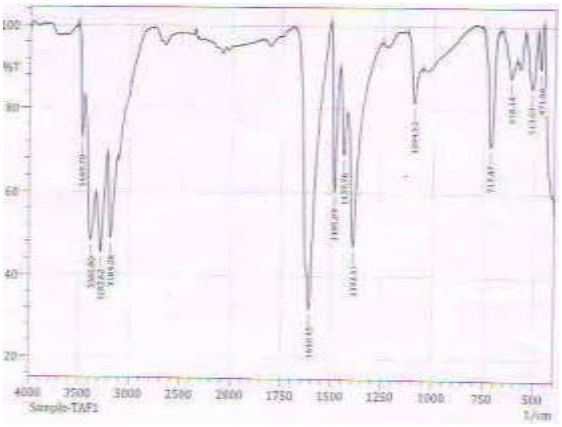

Fig 6. FTIR Spectrum of KTN doped L -Alanine crystal

Figure 6 is the Spectrum of KTN doped L-Alanine crystals contains strong intense peaks at 3386 and 3283cm^{-1} which were attributed to NH_2 bending of hydrogen group and =C-H stretching vibration respectively. The absorption at 3184cm^{-1} was attributed to N-H stretching vibration of amines. The Peaks observed at 1610 and 1485cm^{-1}were assigned to C=N stretching and bending vibration of C-N stretching respectively. The peak of 1439 and 1095 cm^{-1} were ascribed to deformation of C-H and presence of alkyl substituted ether. The absorption peak at 717cm^{-1} was attributed to C-S symmetric stretching respectively.

3.4. UV-Visible Analysis

Optical absorption analysis was studied at room temperature by Shimadzu UV-1800 UV-Vis-NIR spectrometer. The optical absorption spectrum of KTN Crystals and KTN doped L-Alanine Crystals was recorded in the range 190 – 900 nm. Fig 5 assertains that the crystal has a wide transmission of about 100% in the entire range without any absorption peak. The lower cutoff wavelength of KTN crystal and KTN doped L-Alanine Crystal were found to be 317nm and 308nm.The crystal has good optical transmission in the visible region. The measured transmittance (T) was used to calculate the absorption coefficient (α) using the relation

$$\alpha = \left\{2.3026 \log\left(\frac{1}{T}\right)\right\}/t \text{- -------- (1)}$$

Where t is the thickness of the sample. The optical band gap (E_g) is related to optical absorption coefficient (α) and energy (hν) of the incident photon given by [6]

$$\alpha = \{A(h\nu - E_g)1/2\}/\ h\nu \text{ ----- (2)}$$

Where A is a constant, E_g is the optical band gap, h is the Planck's constant and ν is the frequency of the incident photons. The band gap of KTN and KTN doped L-Alanine crystals were estimated by plotting $(\alpha h\nu)^2$ vs Proton energy hν as shown in figure6. From the fig 6, the value of band gap was found to be 4.5ev and 4.75ev.

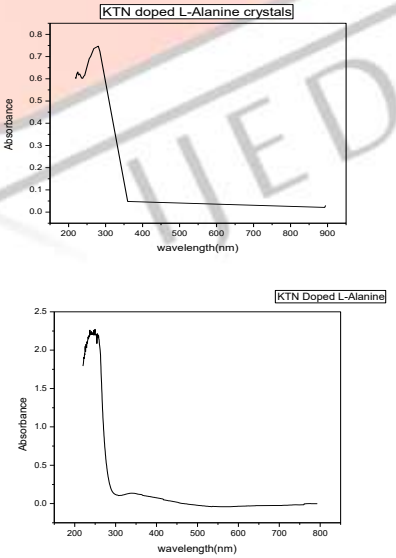

Fig 5.Optical absorption spectrum

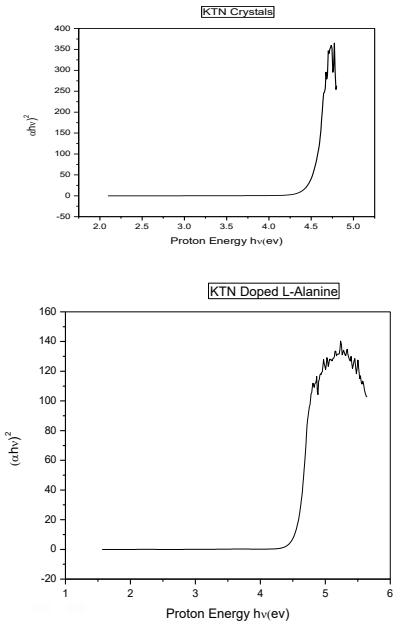

Fig 6.Tauc Plot

IV. CONCLUSION

Good quality single crystals of KTN and KTN doped L-Alanine crystals were successfully grown by slow evaporation method. Single crystals X-ray Diffraction analysis confirms that the crystal belongs to orthorhombic system. The FTIR analysis confirms the presence of functional groups in the crystal. UV-Vis spectrum analysis reveals that high absorbance of the grown crystal with lower cutoff wavelength at 317nm and 308nm. The band gap for the grown crystal was found to be 3.98eV and 4.03eV. Mechanical hardness studies reveals that Vickers Micro Hardness number increases with load and it is also confirmed that the grown crystals belong to soft material category.

V. ACKNOWLEDGEMENT

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V. REFERENCE

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