STUDIES ON GROWTH AND CHARACTERIZATION OF L-HISTIDINESINGLE CRYSTAL

Dr.K.N.Chadambarakumar[1].,Dr.G.Vengatesan[2].,Dr.S.Rajesh[3].,Ms.C.Elakkiya[4].

Assistant professor Department of physics

Dhanalakshmi Srinivasan College of arts and science for women (autonomous)

Perambalur- 621212

I.Abstract

Single crystals of pure L-Histidine Di Picrate (LHDP) have been grown by slow evaporation technique at room temperature. The crystalline nature of grown crystal was established by powder X-ray diffraction analysis (XRD). The structure of the grown crystal determined by X-ray diffraction analysis reveals that it belongs to the monoclinic system. The functional group of the mature crystals was found by FTIR analysis. The spectral bands have been compared with similar complexes using the FTIR spectrum in the range 400-4000 cm-1. The UV-Vis study was performed to know the optical behavior of the grown crystals. The optical transmittance of the spectrum of solid material was measured in the range of 190 to 800 nm. The crystal shows a good transmittance in the visible region. The transmittance above 74% is observed from 354 to 800 nm, which clearly shows the crystal possesses good optical transparency for the second harmonic generation.

KEYWORDS: pure L-Histidine, FTIR, UV, second harmonic generation.

II. INTRODUCTION

Producing good quality crystals of a suitable size is the first and most important step in determining any crystal structure. Crystallization is the process of arranging atoms or molecules that are in a fluid or solution state into an ordered solid state. This process occurs in two steps nucleation and growth. Nucleation may occur at a seed crystal, but in the absence of seed crystals usually occurs at some particle of dust or some imperfection in the surrounding vessel. Crystals grow by the ordered deposition of material from the fluid or solution state to a surface of the crystal.

Kuang-Chich Lai et al, have prepared and reported and reported for crystal at first time by conventional methods [I]. They used modified and as-deposited surface properties of thin-film for back contacts on a-Si solar cells. Texturing was performed by simple dry plasma etching in a CVD process chamber. When power is 100W substrate temperature is 190°C, the pressure is 400 Pa and process gas H2 flow is 700 cm. SEM and TEM were used to evaluate the morphological treatment and induced changes in the deposited films. Comparison of the Si solar cells with/without texturing showed both increases in short-circuit current density and fill factor. Consequently, the Si solar cell efficiency was relatively improved by 4.6%.

Li Wei Change et al .have reported SnO2 nanowires synthesized by thermal evaporation with ZnO metal as the dopant source [2]. The investigator had confirmed, the nanowires are quartzite hexagonal structures. These doped nanowires have the diameters in the range 30-7- nm and several hundred nanometers of length with growth direction along the (100) crystal plane. They found optical properties from the cathode luminescence (CL) and photoluminescence (PL) spectrum showed that nanowires exhibit a relative weak ultraviolet emission (UV) and a strong green emission. Field emission measurements demonstrated that the possesses good performance with a turn-on field of 3.4 V/μm at a current density of 10μm/cm2, a threshold field of 5.4V/μm at a current density of I μa /cm2 and a field-enhancement factor of 5945. These results are very helpful for the fabrication and optimization of integrated optoelectronic nanodevices using nanowires.

Xun Bie and his co-worker have reported transparent conductive films were deposited on glass substrates by DC reactive magnetron sputtering [3]. Under the optimized deposition conditions, the films showed acceptable crystal quality, lowest electrical resistivity 2.61X 10-4Ωcm and high transmittance of 90%in the visible region. The surface root-mean-square (RMS) roughness value was about 7.5nm in a scanning area of 10μm X 10μm for the film was observed by them.

Bie et al .have been reported transparent conductive films were deposited on glass substrates by DC reactive magnetron sputtering method [4]. The structural, optical; and electrical properties of films were investigated in a wide temperature range from room temperature up to 400°C. The crystalline and surface morphology of the electrical and optical properties of the films are studied by them. The observed films deposited at 350°C exhibited the relatively well crystalline and the lowest resistivity of 3.4 X 10-4Ω cm. More importantly, the low-resistance and high-transmittance films were also obtained at 150°C and by changing the sputtering powers, having acceptable properties for application as transparent conductive electrodes in LCDs and solar cells are also studied.

Yun-yan Liu et al [5] have reported the films were deposited on quartz substrates by a laser deposition system using a KrF excimer laser at a wavelength of 248nm (200mJ per pulse, 10 ns, and 5Hz). A ceramic with a content of 2.5 at % was used as a target. The XRD pattern was obtained for the films deposited on quartz substrates for 2,4,10 and 30 min and AFM images had been scanned for different films with deposition times=1,4,10 and 30 min. The electrical and optical property, the optimal film prepared in their experimental condition was 10min with a thickness of 200nm, corresponding to the resistivity of 4.8 X 10-4Ω cm, and its average optical transmittance was 85%.

Keun Jung et al studied thin films were deposited on glass substrates at room temperature by continuous composition spread (CCS) method [6]. In their study, they optimized thin films with a low resistivity of 1.46 X 10-3Ω cm and an average transmittance above 90% in the 550nm wavelength region was formed at an Ar pressure of 2.66 Pa and room temperature.

They optimized the composition of the thin film which had the lowest resistivity and high transmittance was found at 0.8 wt of %

Quan-Bao Ma and his team have explored Gallium-dopes tin oxide() transparent conductive films were deposited on glass substrate by DC reactive magnetron sputtering [7] They observe the lowest resistivity of the film as 4.48 X10-4Ωcm and the average transmittance of the films' as over 90% in the visible range. The obtained optical band gaps of these films were much larger than pure (~3.34eV).

Seung Wook Shin et al have reported thin films [8]. The thin films were deposited by RF magnetron sputtering at different growth temperatures ranging from 350°C to 750°C. XRD and TEM showed that the thin films deposited below a growth temperature of 450°C grew epitaxial with an orientation relationship of (0001). The thin film deposited at 350c showed the lowest electrical resistivity of 1.13 X 10-4Ωcm. The electrical properties of the thin films also deteriorated with increasing growth temperature. UV-visible spectroscopy showed that thin films are highly transparent (from 75%to 90%) in the visible region. Besides, the bandgap of the deposited thin films decreased from 3.5 to 3.2eV with increasing growth temperature.

III. EXPERIMENTAL TECHNIQUES

Properties Picric acid

Picric acid is an organic compound with the formula (O2N)3C6H2OH. Its IUPAC name is **2,4,6-trinitrophenol** (**TNP**). The name "picric" comes from the Greek πικρός (*pikros*), meaning "bitter", reflecting its bitter taste. It is one of the most acidicphenols. Like other highly nitrated organic compounds, picric acid is an explosive, which was once its primary use. It has also been used in medicine (antiseptic, burn treatments) and dyes.

Chemical formula	**$C_6H_3N_3O_7$**
Molar mass	229.10 $g \cdot mol^{-1}$
Appearance	Colorless to yellow solid
Density	1.763 $g \cdot cm^{-3}$, solid
Melting point	122.5 °C (252.5 °F; 395.6 K)
Boiling point	> 300 °C (572 °F; 573 K) Detonates
Solubility in water	12.7 $g \cdot L^{-1}$
Vapor pressure	1 mmHg (195 °C)[2]
Acidity (pK_a)	0.38
Magnetic susceptibility (χ)	$-84.34 \cdot 10^{-6}$ cm^3/mol

Properties L-Histidine

Histidine (symbol His or H, encoded by the codons CAU and CAC) is an α-amino acid that is used in the biosynthesis of proteins. It contains an α-amino group (which is in the protonated $–NH_3^+$ from under biological conditions), a carboxylic acid group (which is in the deprotonated $–COO^−$ form under biological conditions), and an imidazole side chain (which is partially protonated), classifying it as a positively charged amino acid at physiological pH. Initially thought essential only for infants, longer-term studies have shown it is essential for adults also.

Histidine was first isolated by German physician Albrecht Kossel and Sven Hedin in 1896. It is also a precursor to histamine, a vital inflammatory agent in immune responses. The acyl radical is histidyl.

For preparing the title compound of Picric acid ($C_6H_3N_3O_7$)and L-Hiustidine ($C_6H_9N_3O_2$), water is taken as a solvent. L- Histidine and Picric acid were mixed 1:5 ratio in 50 ml Ethanol and stirred well to form a homogeneous solution. The solution was maintained at a constant temperature of 333K for 30 hours till the solvent get evaporated and a yellow salt was extracted. The purity of the synthesized yield was increased by the successive recrystallization process.

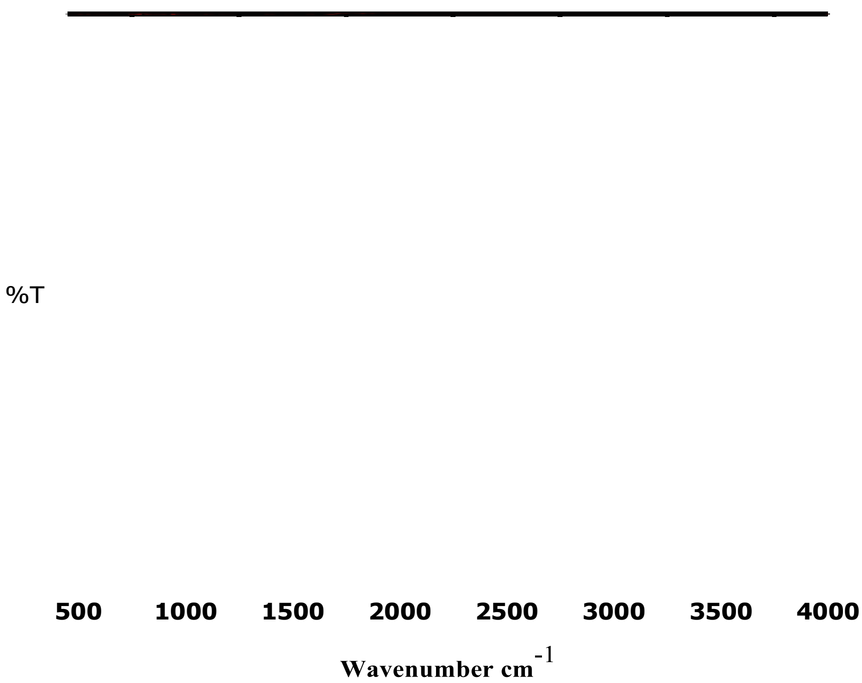
The saturated solution of the synthesized salt was prepared by dissolving it in 20 ml Ethanol and stirred well for about 6 hours with a temperature-controlled magnetic stirrer to give a homogeneous mixture of solution. The solution was filtered with high-quality filter paper and kept undisturbed. Colorless crystals were harvested after 30 days and shown in Fig. The good quality crystals were selected for further studies.

IV. RESULTS AND DISCUSSION

Fourier Transforms Infrared (FT-IR) analysis:

FTIR spectrum show changes in the absorption wavenumber due to change in the bond length between O-H and P=O. It is found that the optical properties of pure and doped ADP changed due to the weak force of attraction of the bond between O-H and P=O. This force of attraction may not be only due to hydrogen bonding but also due to the substitution of $NH4^{+}$ ion in the crystal lattice of tetragonal ADP crystal, which is in good agreement with the result of Ananda Kumari at el (2009).

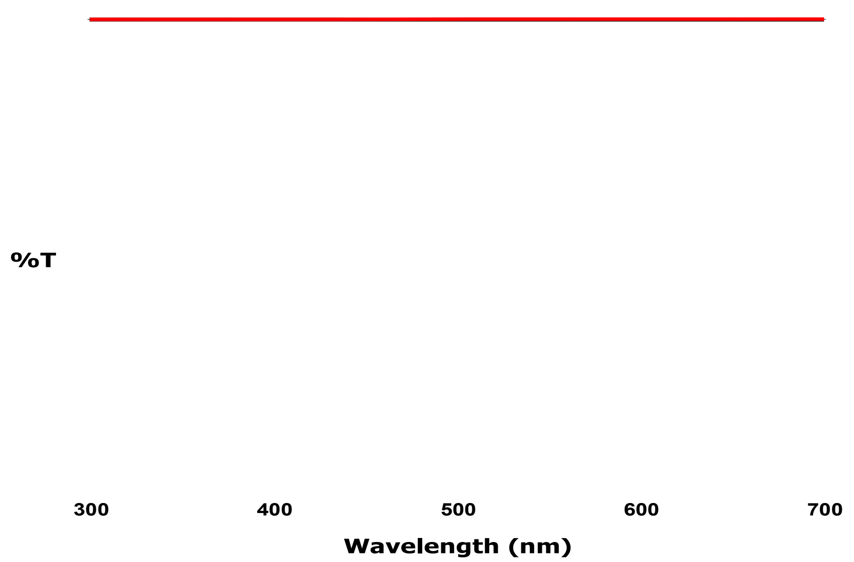
The characteristics absorption frequencies of various functional groups are given in the following table1.



FT-IR of LHP Crystal

UV-Visible Spectrum

The UV-Vis-NIR transmittance spectrum was recorded for the grown crystal using a UV-Vis-NIR spectrophotometer in the range 420-700 nm, to find the suitability of LHP crystal for optical applications. The recorded spectrum is shown in fig.3. The crystal shows good transmittance in the visible region which enables it to be a good material for optoelectronic applications.



UV-Visible Transmittance Spectrum of LHP Crystal

V. Conclusion

L- histidine doped picric acid (LHP) crystals were grown by slow evaporation technique. The powder X-ray diffraction studies of pure and L- Histidine doped picric acid showed that crystal posses tetragonal structure having symmetry space group, with lattice parameter has good agreement with JCPDS data card no. 085-0815. Even after the doping crystal system remains unchanged. Intensity peaks of LHistidine doped picric acid crystal resemble with diffraction angle of pure picric acid crystal with negligible small variation, while intensity variation observed. The FT-IR spectrum confirms the presence of all functional groups and L- Histidine. As the concentration of L- Histidine increases the peaks shifted towards higher wavenumber side. The UV-Vis-NIR spectral studies confirmed that the grown crystal has wide transparency in the visible region the good transparency shows that LHP crystal can be used for Nonlinear optical applications.

VI.REFERENCES

[1] Gilman, J.J Ed., (1963), "The Art and Science of Growing Crystals", John Wiley & Sons, Inc, New York, London.

[2] Brice, J.C (1965), "Crystal Growth Processes", John Wiley & Sons, New York.

[3] Brice, J.C (1965), "The growth of crystals from the Melt", (North Publishing company, Amsterdam.

[4] Ramasamy, P. C.Subramaniam and R. Dhanasekaran (1986), "Proceedings of UGC summer school on crystal growth and characterization of Advanced Materials for solid-state applications".

[5] Bulakh, B.M (1970), J. Cryst. Growth. **7**,196.

[6] Henisch, H.K (1980, "Crystals in Gels and Lisegang Rings", (Cambridge University Press, Cambridge)

[7] Arora, S.K. (1982)," Prog.Crystal Growth Charact.", **4**,345.

[8] Patel, A.R and A.Venkateswara Rao (1982), "Bull Mater.Sci.",**4**,527.