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Synthesis, Growth and Characterization of Trifluoroacetyl Glycine (TFAG) Single Crystal

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Abstract: Trifluoroacetyl Glycine (TFAG) Single crystal have been grown by slow evaporation solution growth technique. The presence of functional groups is identified by Fourier transform infrared (FTIR) analyses. UV–vis-NIR spectroscopy shows that minimum absorption in the entire visible region. The optical band gap (E_g) value of the grown crystal is obtained from the Tauc's plot also transparency, band gap, extinction coefficient and the refractive index were calculated. The mechanical properties have been studied by vicker's microhardness test.

Keywords: TFAG, FTIR, UV–vis-NIR, Band gap (E_g), Tauc's plot

1. Introduction

Glycine is a sweet tasting, non-essential amino acid that was first isolated in 1820 from gelatin and is also found in good quantity in silk fibroin. Also glycine is 'crystal friendly' since it easily crystallizes along with the added inorganic constituents like acids or salts [1–6]. Glycine is the simplest amino acid. Unlike other amino acids, it has no asymmetric carbon atom and is optically inactive. It has three polymeric crystalline forms α, β and γ. Glycine and its methylated analogues form complexes with mineral acids exhibiting interesting physical properties like ferroelastic, ferroelectric or antiferroelectric behaviour often associated with transitions to commensurate or incommensurate phases. According to the structural analysis of ferroelectric triglycine sulphate [7] there are two kinds of glycine groups, glycinium ions and zwitter ions. Such configurations of glycine ions interconnected by short O–H…O hydrogen bonds are regarded as particularly important for the ferroelectric behaviour of this crystal. In the ferroelectric and paraelectric structure of (glycine) $2HNO_3$, one of the glycine molecules has the zwitterionic configuration and the other is monoprotonated [8,9]. In the present study, we have grown single crystals of Trifluoroacetyl Glycine (TFAG) Single crystal and characterized by carrying out X-ray diffraction (XRD), FTIR, UV–Vis-NIR spectroscopic and microhardness measurements.

2. Synthesis of TFAG crystals

For preparing the title compound of Trifluoro acetic acid (CF_3COOH) and Glycine (NH_2CH_2COOH), water is taken as a solvent. Trifluoro acetic acid and Glycine were mixed 1:1 ratio in 50 ml doubly distilled water and stirred well to form homogeneous solution. The solution was maintained at constant temperature of 333K over a period of 30 hours till the solvent get evaporated and a white coloured salt was extracted. The purity of the synthesized yield was increased by successive recrystallization process. The saturated solution of the synthesized salt was prepared by dissolving it in 20 ml of doubly distilled water 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

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filter paper and kept undisturbed. Colorless crystals of dimensions 40 x11 x 8 mm^3 were harvested after 25 days and shown in Fig.1. The good quality crystals were selected for further studies.

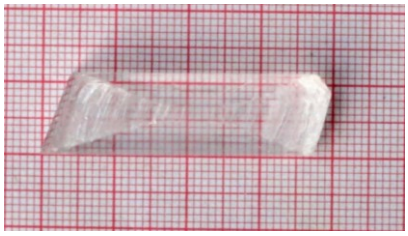

Figure 1. Photograph of TFAG crystal

3. X-Ray Analysis

The grown crystals have been subjected to single crystal X-ray diffraction studies using an ENRAF NONIUS CAD4 automatic X-ray Diffractometer. The crystallographic details are given in Table.1.

Molecular Formula	$C_4H_4F_3NO_3$
Crystal system	Orthorhombic
a (A°)	10.651
b (A°)	8.891
c (A°)	21.452
α (°)	90
β (°)	90
γ (°)	90
V(A^{o3})	2031.462

4. FT-IR Analysis of TFAC

The FTIR spectrum of grown crystal was revealed at room temperature in the range of 400–4000 cm^{-1} and shown in Fig 2. Grown crystals were crushed and few mg of the powder was used for making pellets with KBr. The pellets were then exposed to IR radiation. The FTIR spectrum was taken using Perkin Elmer FTIR spectrometer. The tentative frequency assignments were made by comparing it with standard values of frequency. The following table reveals the absorption peaks corresponds to TFAG crystal.

The asymmetric stretching mode of NH_3^+ present at 3091 cm^{-1}. The C=O stretch of carbonyl groups displays its characteristic peak at 1758 cm^{-1}. The CH_2 bending mode of glycine is located at 1447 cm^{-1}. The NH_3^+ displays its characteristic bending modes at 1534 and 1659 cm^{-1}. The intense sharp peaks position between 1000 and 1200 cm^{-1} are assigned to stretching modes of carboxyl group. The peaks due do NH_3^+ oscillation are seen at 601 and 513 cm^{-1}. The peaks at 2655, 1173 and 1123 cm^{-1} and the medium are assigned to F-C stretching vibrations.

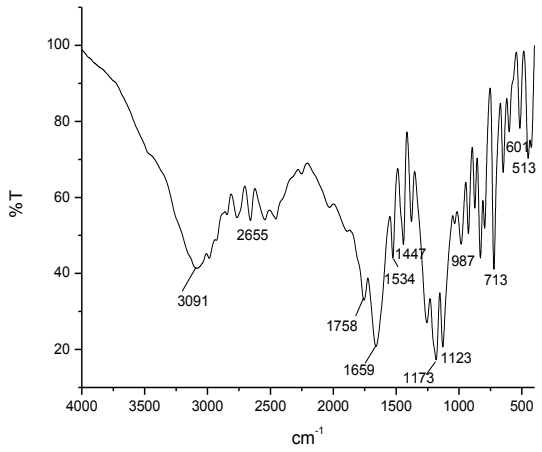

Figure 2. FTIR spectra of TFAG single crystal

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5. UV-Visible Spectral Analysis of TFAG

In a crystalline material, the region of transparency to electromagnetic radiation defines the intrinsic loss mechanism and also theoretical transmittance achievable within the region. The transmission range of Trifluoroacetyl Glycine crystal was determined by recording an optical transmission spectra in the wavelength region 200 – 1200 nm. A graph of transmission Vs wavelength ignoring the loss due to absorption is shown in Fig.3. For TFAG the lower cutoff is at 273nm and it has the large transmission in the entire visible region. Hence it may be a suitable material for up to electronic applications.

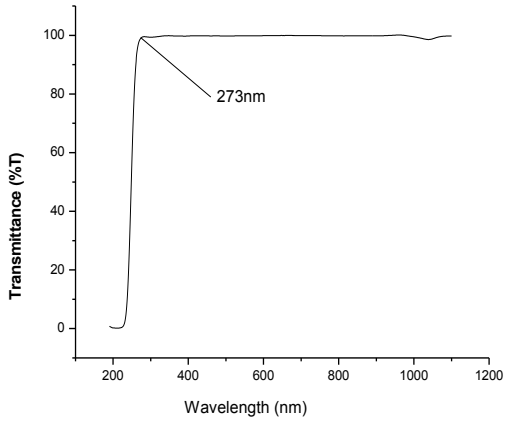

Figure 3. UV-Visible spectra of TFAG crystal

6. Optical Band Gap

The dependence of optical absorption coefficient with the photon energy helps to study the band structure and the type of transition of electrons. The absorption coefficient (α) were determined using Beer's law

$$\alpha = \frac{1}{d}\log(\frac{1}{T})$$

where T is the transmittance and d is the thickness of the cell. As the indirect band gap, the crystal under study has an absorption coefficient (α) obeying the following relation for high photon energies,

$$\alpha = \frac{A(h\nu - E_g)^2}{h\nu}$$

where E_g is optical bandgap of the crystal and A is a constant. The plot of variation of Photon energy (E) in eV and $(\alpha h\nu)^{1/2}$ is shown in Fig.4. The linear region of the curve was extrapolated to find the x-intercept which gives the Band gap of the compound.

The band gap is found to be 4.77eV. As a consequence of wide band gap, this crystal can be a suitable material for the optoelectronic devices like LED and Laser diodes.

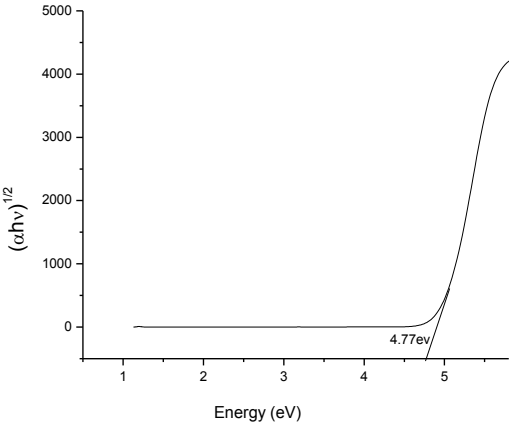

Figure 4. Bandgap curve of TFAG crystal

7. Optical Constants

The optical properties of a crystal can be evolved mainly from its optical transparency, band gap, extinction coefficient and the refractive index. The optical properties of the crystals are governed by the interaction between the crystal and the electric and magnetic fields of the electromagnetic wave. Extinction coefficient is the fraction of light lost due to scattering and absorption per unit distance in a participating medium. In electromagnetic terms, the extinction coefficient can be explained as the decay or damping of the amplitude of the incident electric and magnetic fields. The extinction coefficient can be calculated from the following relation,

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$$K = \frac{\lambda \alpha}{4\pi}$$

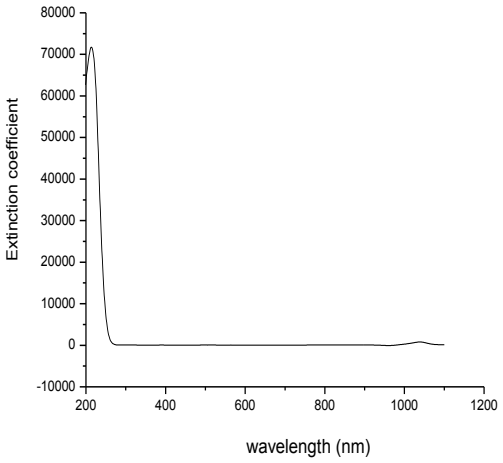
The reflectance gives the ratio of the energy of reflected to incident light from a crystal. The reflectance in terms of the absorption coefficient can be calculated using

$$R = \frac{1 \pm \sqrt{(1 - \exp(-\alpha t) + \exp(\alpha t)}}{1 + \exp(-\alpha t)}$$

The refractive index can be determined from the reflectance (R) data using the relation

$$n = \frac{-(R+1) \pm \sqrt{-3R^2 + 10R - 3}}{2(R-1)}$$

A profile on the above calculated optical constants as a function of wavelength is graphically illustrated in fig.5. From the graph it is clear that the extinction coefficient (k), reflectance(R) and refractive index (n) vary with wavelength and hence depend on the photon energy. The internal efficiency also depends on the photon energy. Hence by tailoring the photon energy, one can achieve the desired material for the device fabrication.



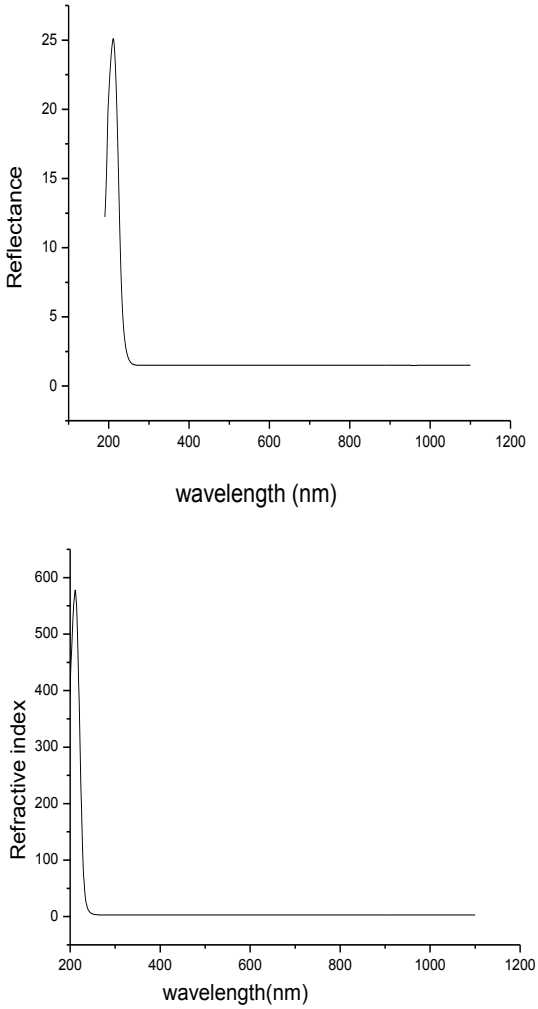


Figure 5. Profile of the variation of optical Constants of TFAG crystal

8. Mechanical Analysis of TFAG

The mechanical strength of a material is primarily studied from its micro hardness. The micro hardness studied have been carried out on the grown crystals using Leitz Weitzler tester fitted with Vickers diamond pyramidal indenter. Vickers micro hardness as a function of the applied load is shown in Fig. 6. From the graph it is observed that the hardness number increases with increase of load. The value of the work hardening coefficient n (Fig. 7) is found to be 3.996. According to Onitsch and Hanneman, 'n' should be between 1 and 1.6 for

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hard materials and above 1.6 for softer ones. Hence TFAG single crystal belongs to soft material category.

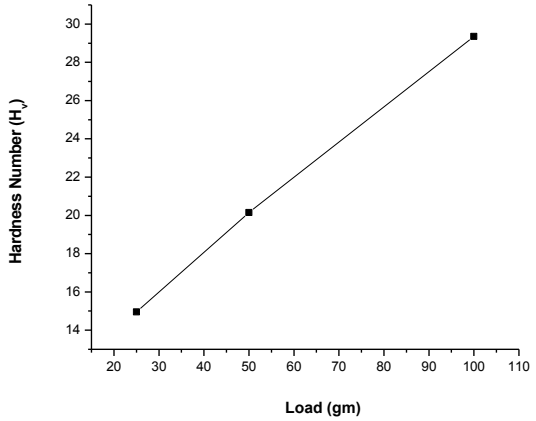

Figure 6. Hardness variation of TFAG crystal

The toughness of the material is the resistance to fracture. The fracture toughness K_c of the material is dependent on the micro structural features and is generally insensitive to the chemical species in the surrounding environment.

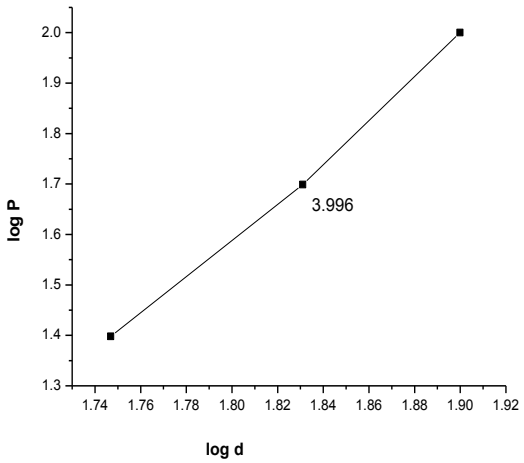

Figure 7. Work hardening coefficient curve of TFAG crystal

9. Conclusion

Trifluoroacetyl Glycine (TFAG) Single crystal have been grown by slow evaporation solution growth technique. The presence of functional groups is identified by Fourier transform infrared (FTIR) analyses. UV–vis-NIR spectroscopy shows that minimum absorption in the entire visible region. The optical band gap (E_g) value of the grown crystal is obtained from the Tauc's plot also transparency, band gap, extinction coefficient and the refractive index were calculated. The mechanical properties have been studied by vicker's microhardness test.

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