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GROWTH AND CHARACTERIZATION OF ZTS SINGLE CRYSTAL AND ITS ANALYSIS OF OPTICAL, STRUCTURAL, MECHANICAL, THERMAL AND DIELECTRIC STUDIES

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ABSTRACT: The growth of Zinc Thiourea Sulphate (ZTS) Single Crystals is analysed in this study. The crystals were grown by slow evaporation technique. The X-Ray diffractions study indicates that the prepared grown crystals confirm the crystal structure for ZTS crystal. In Good quality crystals of size 12x5x7mm^3 single crystals were grown. The presence of Sulfur- to- Zinc bonds in the complex has been revealed by Fourier transform infrared analysis. The optical characterization shows that the grown crystal having ZTS crystal has low UV cutoff of 220nm and has a good transparency in the visible region. The thermal analysis shows that the Zinc Thiourea Sulphate can be expoliated up to 240°C of ZTS Single Crystal. The Vicker's microhardness number was found to increase ZTS crystal which studies the mechanical stability of the grown crystal. The dielectric properties of dielectric constant as function of frequencies 80KHz room temperature for electro optic modulators of ZTS Single crystal.

Key word: FTIR, UV-Visible, Micro hardness test, Powder XRD, Thermal analysis, electrical studies.

1.INTRODUCTION:

Zinc thiourea sulphate (ZTS) is a semi organic nonlinear optical material (NLO) [1-2]. In semi organic nonlinear optical materials, metal complexes of thiourea which have low UV cutoff wavelengths, and high power frequency conversion [3]. ZTS crystals have thermal stability is less and more over nlo efficiency is high. In inorganic crystals, NLO composition is very low but thermal stability is high [4]. In semi organic nlo material non-linearity is very high and it has laser induced damage, low angular sensitivity and good mechanical hardness. The ZTS possess is 1.2 times more SHG efficiency than KDP [5]. Nonlinear optical crystals has a significant impact on laser technology. Optical communication and electro optical modulation [6]. The grown crystal has been characterization by carrying out FTIR,UV-Visible spectrometer, Vickers hardness test, thermo gravimetric (TG/DTA), powder X-Ray diffraction, and dielectric measurements.

2.MATERIALS AND METHODS:

The compound is synthesized from purified zinc thiourea sulphate in the stoichimetric ratio 1:3. By recrystallization process the synthesized material is purified using water as a solvent.

$$ZnSO_4+3[CS(NH_2)_2]\rightarrow Zn[CS(NH_2)_2]_3.O_4$$

Purified compound is dissolved in double distilled water to obtain a homogenous solution. The solution is kept under a constant temperature to obtain seed crystals by spontaneous nucleation. The temperature was reduced at a rate of 0.05-0.5^{0}C/day growth progressed. The seeds obtained from slow evaporation were employed for the growth. The period of growth ranged from 40-60 days. Good quality crystals of size 12x5x7mm^3 were grown single crystal.

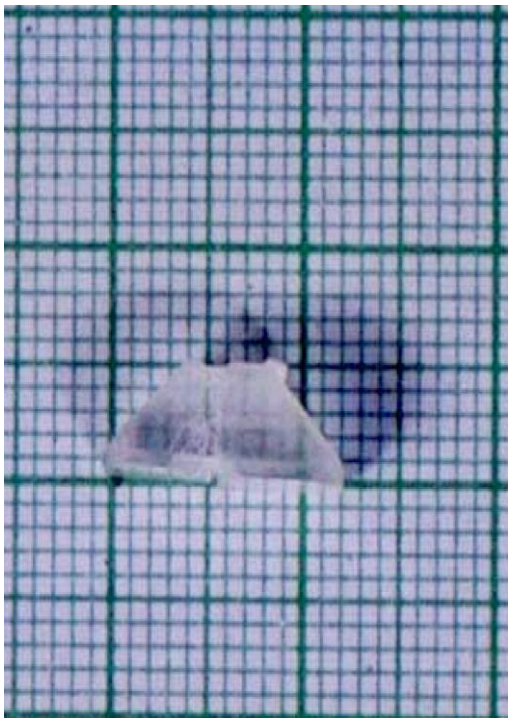

Fig1. The Photograph of ZTS single crystal.

3. CHARACTERIZATION:

3.1 Powder X-Ray Diffraction:

The powder X-Ray diffraction analysis of pure ZTS was subjected. The samples were scanned over the range 11 to 52 (2θ). The recorded X-ray spectra of pure ZTS is shown in figure 2(a). The interplaner d spacing was calculated using bragg's equation. Using the value of d, the hkl values for all the reflections were obtained. The experimental and calculated d-values along with the hkl indices of the corresponding reflecting planes for pure ZTS crystals are tabulated respectively. All the peaks could be indexed to the orthorhombic structure and cell parameters for ZTS crystal are a=11.12Å, b=7.773Å, c=15.49Å for ZTS crystal.

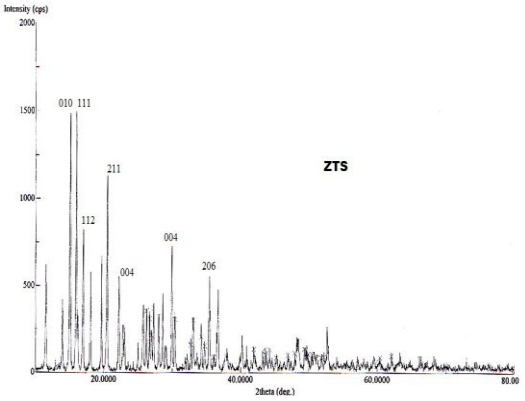

Fig 2(a) Powder X-Ray spectrum of ZTS

3.2 Uv-Visible Transmittance Study:

The UV-Visible spectrum of pure ZTS crystals were recorded in the range of 190nm-1100nm using LAMBDA-35 uv-visible spectrophotometer. The optical transmission spectra shown in fig 2(b) of ZTS has a good transparency in between 200 and 1100nm. The lower cutoff frequency of pure ZTS crystals occur 220nm. The ZTS crystals has minimum of 55% transmission increasing transparency in visible region for pure ZTS single crystal.

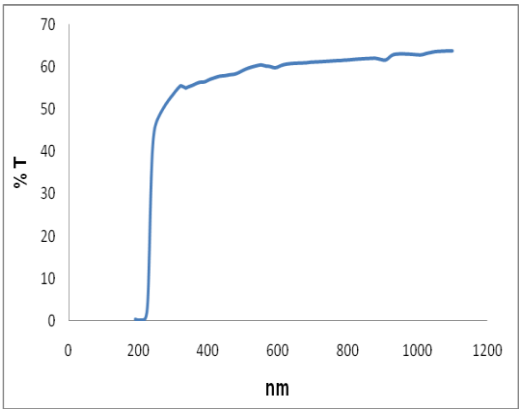

Fig 2(b) UV-Vis spectrum of ZTS

3.3 FTIR Analysis:

The presence of ZTS crystal was analyzed from the FTIR spectra of the grown crystals and compared with each other. The figure 2(c) shows that the FTIR spectra of pure ZTS crystals. The spectrum was recorded using KBr pellet technique in the frequency region 400 - 4000cm^{-1} using PERKIN ELMER fourier transform infarad spectrometer. The FTIR spectrum contains the bands corresponding to different molecular groups present in the sample. The ZTS show a broad envelope lying in between 2712cm^{-1} and 3585 cm^{-1} arising the symmetric and asymmetric modes of the NH_2 group of Zinc coordinated thiourea. The absorption band at around 1626cm^{-1} in the spectra of NH_2 bending vibration. The absorption observed at around 1508cm^{-1} corresponds to the N-C-N stretching vibration. The absorption band at about 1400cm^{-1},1030cm^{-1},948cm^{-1},746cm^{-1}corresponds to the N-H stretching vibration. The presence of sulphate ion in the coordination sphere of pure ZTS from the peaks FTIR spectrum in the frequency region.

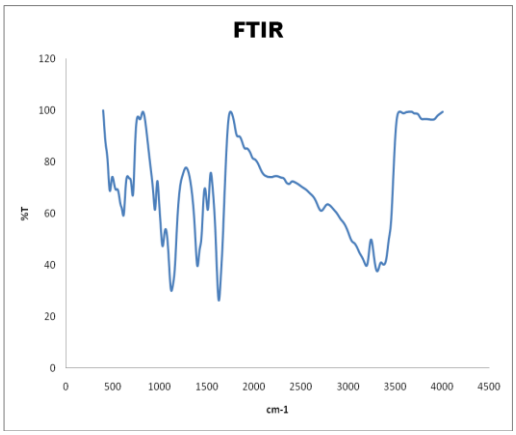

Fig 2(c) FTIR Spectrum of ZTS

Table1: Wavenumber for FTIR Spectrum of ZTS

Thiourea	Pure ZTS	Assignment
3585	2712	NH_2 stretching
1626	1508	Asymmetric N-C-N stretching
1400	1030	N-H stretching
948	746	N-H stretching

3.4 Microhardness Measurement:

Microhardness testing is performed on single crystals to evaluate the mechanical properties to the suitability of the material by measuring the resistance of the lattice was applied load. The hardness measured using the leitz - wetzler hardness tester. The ZTS crystal shown in fig 2(d) grown and load P was varied between 25g to 100g by the

time of constant (10s) for the all trials and the diagonal length was measured.

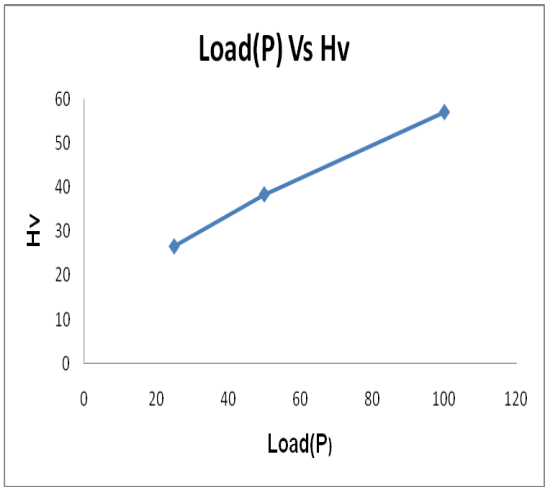

Fig 2(d) Microhardness test of ZTS

3.5 THERMAL ANALYSIS OF ZTS:

The thermo gravimetric analysis of pure ZTS crystals were analyzed of SDT Q600. In TGA/DSC figure 2(e) shows that the sample pure ZTS crystals are carried and between 25°C is stable till 240°C in nitrogen atmosphere at a heating rate 10°C/min with 37.78% weight loss of observed from second endothermic peak is increasing to 347.84°C. this ZTS crystal due to the sulphur of thiourea from zinc coordination in thermo gravimetric analysis.

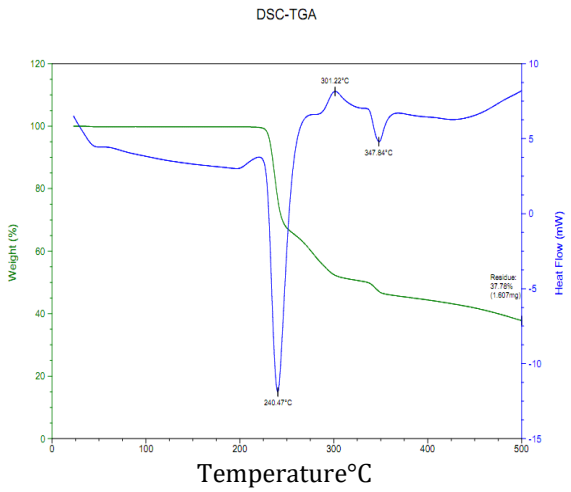

Fig 2(e) TGA-DSC crystal of ZTS

3.6 Dielectric Properties:

Dielectric properties are correlated with electro-optic property of the crystal. The figure 2(f) shows that the relative dielectric constant (ε_r) for ZTS crystals. The room temperature values of ε_r gradually decreases as the frequency increases ε_r decreases upto 80 KHz with respect to the room temperature. These values are low frequencies the dipolar contribution decreases the electronic contribution increases at high frequencies to the total polarizability of ZTS temperature increases.

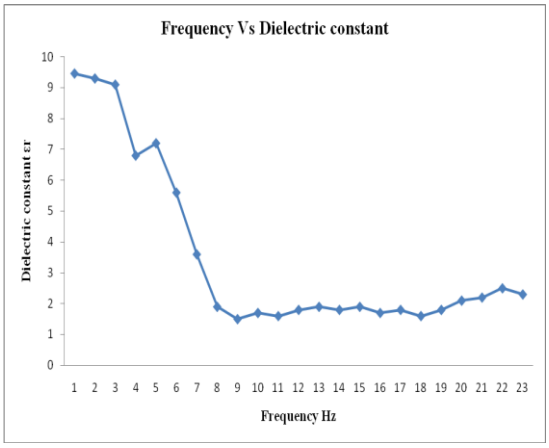

Fig 2(f) dielectric studies of ZTS

CONCLUSION:

The growth and characterization of pure ZTS crystals were grown by slow evaporation method. The crystal is confirmed with X-Ray diffraction were determined to the orthorhombic system for PC2a1. The FTIR spectra revealed all the characteristic bands corresponding to different molecular groups present in the sample. The transmission spectra revealed that the crystals have an extended transparency down to UV-Visible spectrometer. The thermo gravimetric (TGA/DSC) analysis shows that the thermodynamically more stable than pure ZTS crystal. The mechanical property of the grown crystal has been studied by microhardness number values is increased. The frequency of variation dielectric constant at room temperature and above shows the decrease with increase in frequency 80 KHz is a characteristic of a normal dielectric in ZTS single crystal.

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