

Regular Article

Growth, Optical, Electrical and Microhardness Studies of Pure and Strontium Chloride-Doped Zinc Tris-Thiourea Sulfate (ZTS) Crystals

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Abstract

Single crystals of pure and strontium chloride-doped zinc tris-thiourea sulfate (ZTS) were grown from aqueous solution by the slow evaporation method. The grown crystals were transparent and colourless. The grown crystals have been subjected to single crystal X-ray diffraction to determine the unit cell dimensions. Second Harmonic Generation (SHG) for the materials was confirmed using Nd:YAG laser. The UV-visible spectra show that the grown crystals have wide optical transparency in the entire visible region. Atomic absorption study reveals the presence of strontium in the doped ZTS crystals. The density of the grown crystals was measured and it was compared with X-ray data. The DC conductivity was measured in the temperature range 30-130°C along c-direction. DC conductivity of the grown crystals increases with increase in temperature and with concentration of dopants. The micro hardness test was carried out in (100) plane and the hardness coefficient was calculated. The hardness number is found to be increasing with increase in the concentration of dopants and loads.

Keywords: ZTS crystals; Doping; Single Crystal; Solution Growth; Characterization; NLO material

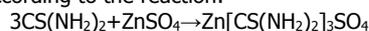
Introduction

Nonlinear optics plays an important role in the emerging photonic and optoelectronic technologies. Nonlinear optical (NLO) materials find wide applications in the area of laser technology, optical communication, data storage technology and the electro-optic modulation [1-4]. Zinc Tris-thiourea Sulfate (ZTS), $Zn[CS(NH_2)_2]_3SO_4$, crystal continues to be an interesting semi-organic material for academic research and industry [5]. ZTS belongs to orthorhombic system with point group $mm2$ and space group $Pca2_1$. The lattice parameters are reported to be $a = 11.261 \text{ \AA}$, $b = 7.773 \text{ \AA}$ and $c = 5.491 \text{ \AA}$ [6]. A wide acceptance angle, good transparency down to 290 nm and a single shot laser damage threshold of $3GW/cm^2$ at 1064 nm, makes it a good candidate, for nonlinear optical applications [7-9]. High damage threshold and wide transparency make it a better alternative for KDP crystals in frequency-doubling and laser fusion [10,11]. The Curie temperature (FE-PE phase transition) of ZTS is $T_c = 323K$ [12]. P.U.Sastry [13] has reported that the linear susceptibility for the ZTS crystal is 0.16. Venkataramanan et al [14] have determined the melting point of ZTS accurately by direct observation method as 234°C. Bhagavannarayana and his co-workers [15,16] have recently found that organic dopants like EDTA are better candidates to influence the crystalline perfection and nonlinear optical properties of ZTS single crystals. Several authors have reported the growth rate and influence of solution pH on the morphology of ZTS crystals [17-19].

Experimental Methods

Growth

Analytical Reagent (AR) grade thiourea, zinc sulfate and strontium chloride along with doubly distilled water are used for the synthesis and growth. Zinc Tris-thiourea sulfate (ZTS) salt was synthesized [20-22] according to the reaction:



Pure ZTS salt was synthesized by stoichiometric incorporation of thiourea and zinc sulfate in the molar ratio 3:1. The component salts were very well dissolved in de-ionized water and it was thoroughly mixed using a magnetic stirrer and the mixture was heated at 50°C

till a white crystalline salt of ZTS was obtained. Temperature was maintained at 50 °C to avoid decomposition. To obtain the doped ZTS salts, different concentrations of strontium chloride such as 1 mole %, 2 mole % and 3 mole % were added to the solution of ZTS separately. Single crystals of pure and strontium chloride-doped ZTS were grown by solution growth employing slow evaporation technique at room temperature [23-29].

In the present work, the supersaturated concentration used for the preparation of supersaturated solution is 1 M. The solution was prepared at a temperature slightly higher than the room temperature and it was cooled naturally to 31°C. The solution was allowed to equilibrate at the same temperature. For the formation of ZTS pure crystals, the amount of solute i.e. (ZTS salts) required to prepare the supersaturated solution is given by the form

$$m = (M \times X \times V) / 1000 \text{ (in gram units)}$$

where M is the molecular weight of the solute, X is the supersaturated concentration in molar units (1 M in the present work) and V is the required volume of the solution. Transparent and colourless ZTS crystals of size $7 \times 7.5 \times 5 \text{ mm}^3$ were harvested in 15-20 days. To grow big-sized crystals, seed crystal technique has been used.

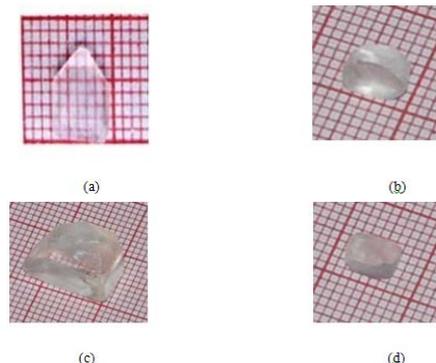
Zinc Tris-thiourea Sulfate (ZTS) was doped with strontium chloride in 3 different molar ratios. For the formation of doped crystals, the supersaturated solution was prepared by dissolving the dopant along with the pure solute. If the molecular ratio of the pure substance and dopant is 1: P , then the amount of dopant (solute) to be added is calculated using the formula.

$$m_1 = (M_1 \times X \times V \times P) / 1000 \text{ (in gram units)}$$

where M_1 is molecular weight of the dopant.

All the harvested crystals of this work are observed to be white. During the growth, very small crystals appeared at first which then grow bigger on slow evaporation. Best crystals free from imperfection are selected for the present study. The grown crystals of this work are displayed in the photograph 1. Morphological changes have been observed when ZTS crystals are doped with strontium chloride. Dimensions of $5.5 \times 5.5 \times 14$, $14 \times 13 \times 17$ and $5 \times 5 \times 12 \text{ mm}^3$ were harvested in 25-30, 33-36 and 38-41 days for 1 mole %, 2 mole % and 3 mole % respectively of strontium chloride-doped ZTS crystals. The external appearance or morphology of the grown crystals seems to be polyhedron in shape. The morphology of ZTS crystal is found to be different when strontium chloride is added ZTS.

Fig.1: Grown crystals (a) Pure ZTS crystal, (b) ZTS +1 mole % of $SrCl_2$, (c) ZTS + 2 mole % of $SrCl_2$ and (d) ZTS +3 mole % of $SrCl_2$



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Characterization techniques

The single crystals of pure and strontium chloride-doped ZTS crystals were subjected to single crystal XRD studies using an ENRAF NONIUS CAD4 diffractometer with MoK_α radiation ($\lambda=0.71073\text{\AA}$) to determine the unit cell dimensions and morphology.

The Second Harmonic Generation (SHG) test for the grown pure and the strontium chloride-doped ZTS was performed by the powder technique of Kurtz and Perry [30] using a pulsed Nd:YAG laser. The grown crystals were ground to powder of grain size 1500-1800 μm and the SHG was confirmed by the emission of green radiation (532 nm) which was detected by a photomultiplier tube. In this experiment, potassium dihydrogen phosphate (KDP) was used as the reference. For single crystals mainly used in optical applications, the optical transmittance range and the transparency cut-off are important. A Varian Cary 5E UV-Vis-NIR spectrophotometer was used for spectral transmission studies.

Atomic absorption studies of strontium chloride-doped ZTS crystals were carried out using an atomic absorption spectrometer (Model: AA 6300). The density of the grown crystals was determined by floatation method. The floatation method is employed for the precise determination of density and this method is sensitive to point defects and insensitive to dislocation of crystals unlike other methods of density measurements [31-35]. Bromoform (density: 2.86 g/cc) and carbon tetrachloride (density: 1.59 g/cc) were used for the experiment. After mixing the bromoform and carbon tetrachloride in a suitable proportion in a specific gravity bottle, a small piece of crystal was immersed in a mixture of the liquids. When the sample was attained in a state of mechanical equilibrium, the density of the crystal would be equal to the density of mixture of liquids. The density was calculated using the relation $\rho=(w_3-w_1)/(w_2-w_1)$, where w_1 is the weight of empty specific gravity bottle, w_2 is the weight of the specific gravity bottle with full of water and w_3 is the weight of the specific gravity bottle full of the mixture of bromoform and carbon tetrachloride.

DC electrical conductivity measurements were carried out along the c- direction using the conventional two-probe technique at different temperatures ranging from 30-130°C in a way similar to that followed by Selvarajan and Mahadevan [36]. Crystals with high transparency and large defect-free size were selected and used for the DC electrical conductivity measurements. The extended portions of the crystals were removed completely. The resistance of the crystals was measured using a million megohm meter. The observations were made while cooling the samples. The samples were cut into rectangular shapes to the desired thickness of 2-3 mm and polished. For good ohmic contact, opposite faces of the sample crystals were coated with good quality graphite. The samples were annealed in the holder assembly at $\sim 130^\circ\text{C}$ before making observations. The dimensions of the crystals were measured using a traveling microscope (L.C=0.001 cm). The DC conductivity (σ_{dc}) of the crystal was calculated using the relation $\sigma_{dc} = d/RA$ where R is the measured resistance, d is the thickness of the sample and A is the area of cross section of the crystal. Microhardness measurements were done using a Vickers microhardness indenter (Leitz Weitzier hardness tester).

Results and discussion

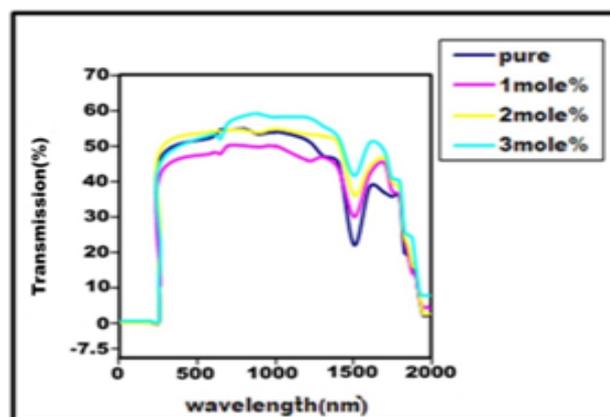
Structural and UV-visible transmittance studies

The obtained unit cell dimensions by single crystal XRD studies are $a=7.858\text{\AA}$, $b=11.255\text{\AA}$, $c=15.529\text{\AA}$, $\alpha=\beta=\gamma=90^\circ$ for pure ZTS crystal and $a=7.823\text{\AA}$, $b=11.241\text{\AA}$, $c=15.511\text{\AA}$; $a=7.833\text{\AA}$, $b=11.210\text{\AA}$, $c=15.544\text{\AA}$; $a=7.831\text{\AA}$, $b=11.283\text{\AA}$, $c=15.584\text{\AA}$ and $\alpha=\beta=\gamma=90^\circ$ for strontium chloride-doped ZTS crystals for 1 mole%,

2 mole% and 3 mole% respectively, which belong to orthorhombic system. The obtained values for pure ZTS crystals are in good agreement with the reported values [39, 6]. The space group and number of molecules per unit cell for the grown crystals were found to be Pca2₁ and 4.

The recorded transmittance spectra of pure and strontium chloride-doped ZTS crystals in the wavelength range 200-2000 nm are shown in figure 2. At about 251 nm [40], a sharp fall in the transmittance to zero is observed and the crystals have sufficient transmission in the entire visible and near IR region and hence the crystals of this work are the potential candidates for optoelectronics. However, there is some significant absorption around 1040 nm due to N-H vibrational overtones [41]. An absorption at the Nd:YAG laser fundamental wavelength is a disadvantage since it reduces the resistance to laser induced damage. The obtained values for cut-off wavelength of the grown crystals in this work are in good agreement with the literature values (below 300 nm [41], 250 nm [42] and 268-272 nm [43]), confirming the NLO property of the substance. From the Figure 2, it is noticed that the cut-off wavelength for pure and strontium chloride-doped ZTS crystals are almost same. The forbidden band gap for the grown crystals of this work was calculated using the relation $E=hc/\lambda$ where h is the Planck's constant, c is the velocity of light and λ is the cut-off wavelength. The obtained value for the forbidden band gap for all crystals is 4.948 eV. This indicates that all the grown crystals are insulators. Since the cut-off wavelength occurs in UV region, the grown crystals are useful to be NLO materials.

Fig.2: The UV-visible transmittance spectra for pure and strontium chloride-doped ZTS crystals



Nonlinear optical (NLO) studies

The Second Harmonic Generation (SHG) test has been carried out to confirm NLO property. When ZTS crystals are doped with strontium chloride, the transmission is altered in the entire region. Increase of transparency indicates improvement of quality of crystals with the incorporation of dopant which suppress the inclusions during growth of the crystals

The values of SHG relative efficiency of pure and strontium chloride-doped ZTS crystals with reference to KDP are tabulated in the table 1. The powder SHG test confirms the NLO property of pure and doped ZTS crystals. When ZTS crystals are doped with strontium chloride, it is observed that SHG efficiency relative to that of KDP increases. This shows that the strontium chloride-doped ZTS crystals will be better candidates for NLO applications.

Density measurement and atomic absorption studies

The density of pure ZTS crystals was also calculated from the crystallographic XRD data using the relation $\rho=(MZ)/(NV)$ where M is the molecular weight of the ZTS crystal, Z is the number of molecules per unit cell, N is Avogadro's number and V is volume of the unit cell. The density of pure ZTS crystals was found to be 1.9114 g/cc [44] which is in good agreement with the literature values (1.923 g/cc [45], 1.910 g/cc [46]). From the crystallographic XRD data, density of pure ZTS crystal was also found to be 1.9084

Table 1: Values of relative SHG efficiency Reference: KDP sample

Sample	Relative SHG efficiency
Pure ZTS crystal	1.2
ZTS+1 mole% SrCl ₂	1.23
ZTS+2 mole% SrCl ₂	1.29
ZTS+3 mole% SrCl ₂	1.31

g/cc. The density of 1 mole % of SrCl₂ doped ZTS crystals is 1.922 g/cc. The density of 2 mole% of doped ZTS crystals is 1.931 g/cc and the density of 3 mole% of doped ZTS crystals is 1.947 g/cc. Density of the grown crystals measured by floatation method was observed in agreement with the crystallographic data. The change of density also indicates the incorporation of impurity in ZTS crystals. The measured values of the density of the doped ZTS crystals are found to be increasing with increase in the concentration of dopants. The presence of strontium in ZTS crystals was confirmed by atomic absorption studies. The concentration of impurity in the doped ZTS crystals is found to be 121 ppm, 125 ppm and 175 ppm for 1 mole%, 2 mole% and 3 mole% of the doped crystals respectively.

DC Conductivity measurement

The DC conductivity (σ_{dc}) values for the grown samples are presented in tables 2. The obtained values are in good agreement with the reported values [47]. From the measurements of DC conductivity, it is observed that DC conductivity increases with the increase in temperature and the concentration of dopants. The increase of DC conductivity with the increase in temperature observed for pure and doped ZTS in the present study is similar to that observed for system like KDP [48]. The defect concentration will increase exponentially with temperature and consequently, the electrical conduction also increases. The conduction region considered in the present study seems to be connected to mobility of vacancies.

Table 2: DC Electrical conductivities of pure and strontium chloride-doped ZTS crystals along c-direction

Temperature (°C)	Conductivity, σ_{dc} (X 10 ⁻⁹ mho m ⁻¹)			
	pure ZTS	Strontium chloride-doped ZTS		
		1 mole%	2 mole%	3 mole%
30	1.227	1.631	1.853	1.964
40	1.392	1.793	2.041	2.174
50	1.402	2.035	2.194	2.397
60	1.420	2.313	2.445	2.595
70	1.456	2.471	2.626	2.749
80	1.743	2.790	2.937	3.295
90	2.003	3.352	3.524	3.959
100	2.453	3.751	4.026	4.474
110	2.845	4.540	4.744	5.588
120	3.354	5.762	6.073	6.835
130	5.446	6.533	7.321	7.867

Microhardness measurement

Microhardness of a crystal is its capacity to resist indentation. In the present work, indentations are made on (100) plane of pure and strontium chloride-doped ZTS crystals for five loads 15, 20, 25, 40 and 50 g and indentation time given is 10 s. For each load, several indentations are made and the average diagonal length (d) is used to calculate the microhardness number. Vicker's hardness number H_v is calculated using the relation

$$H_v = 1.8554 P/d^2 \text{ Kg mm}^{-2}$$

where P is the load applied in Kg and d is the diagonal length of the indented impression in mm. Plots between the hardness values and the corresponding loads for pure and doped ZTS crystals are drawn and they are provided in the figure 3. From the results, it is observed that hardness number increases as the load increases for all the samples of this work. Hardness number is found to be increasing with the doping concentration of SrCl₂ in the lattice of ZTS crystals. The increase of hardness number for the strontium chloride-doped ZTS crystals is due to the presence of dopants in ZTS crystals. It is observed that scratching occurs in the crystals when the load is applied beyond 50 g. Reports of microhardness studies for various systems at low loads were published by a number of researchers [49-54]. Since the hardness number for doped crystals is more than that of pure ZTS crystal, strontium chloride-doped ZTS crystals are harder than pure ZTS crystal. The increase of hardness for the doped ZTS crystals is due to incorporation of dopant in the form of positive and negative ions. These impurity defects in the lattice of ZTS crystals may act as obstacles to dislocation motion thus increasing the hardness of the crystals.

Fig. 3: Variation of microhardness with load for pure and strontium chloride-doped ZTS crystals

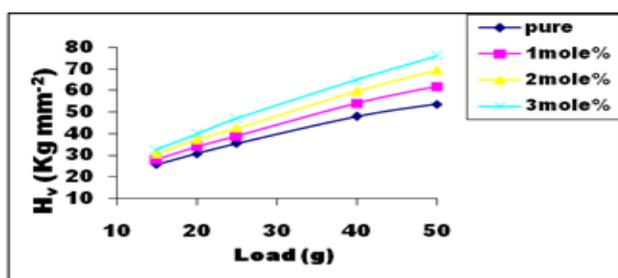
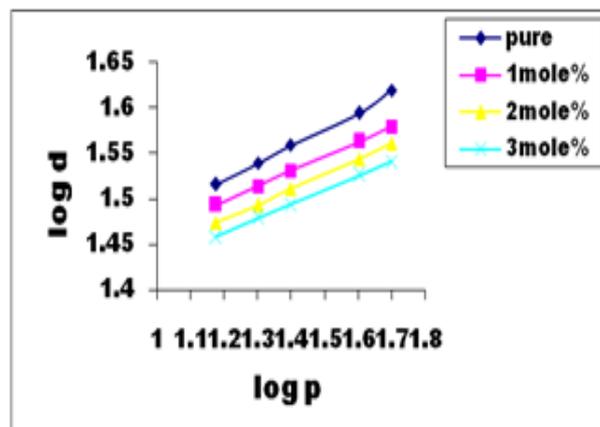


Fig.4: Plots of log P versus log d for pure and strontium chloride-doped ZTS crystals



The relation between the load and size of indentation is given by Meyer's law as $P = a d^n$ where P is load in Kg, d is the diameter of recovered indentation in mm, a is a constant and n is the work hardening coefficient. The plot log p against log d is a straight line, which is shown in figure 4. The slope of the straight lines of the figure gives the work hardening coefficient n [42]. The work hardening coefficients (n) for pure ZTS crystals are found to be 5.189 which is in good agreement with the reported value [42]. Work hardening coefficients of strontium chloride-doped ZTS crystals are 5.933, 6.035 and 6.104 for 1 mole%, 2 mole% and 3 mole% respectively.

Careful observations of Onitsch [55], Hanneman [56] and Selvarajan et al [57] on various materials have pointed out that n lies between 1 and 1.6 for hard materials and it is more than 1.6 for soft materials. According to Onitsch, if n is greater than 1.6, the microhardness number increases with increase in load. Since the obtained values of n for the grown undoped and strontium chloride-doped ZTS crystals are more than 1.6, the grown crystals of this work belong to the category of soft materials.

Conclusions

Single crystals of pure and strontium chloride-doped Zinc Tris-thiourea Sulphate (ZTS) were grown by solution growth. All the grown crystals are transparent with well-defined external appearance. The unit cell parameters have been evaluated by single crystal XRD method. The powder SHG test confirms the NLO property of pure and strontium chloride-doped ZTS crystals. The UV cut-off wavelength was found to be 251 nm and thus the material is a potential candidate for optoelectronics. Atomic absorption studies reveal the presence of strontium in strontium chloride-doped ZTS crystals. Density of the grown crystals measured to be in agreement with crystallographic data. The DC conductivity was found to be increasing with temperature and concentration of dopants. The microhardness studies reveal that all the grown crystals have a relatively high value for its work hardening coefficient. Also it was found that the strontium chloride-doped ZTS crystals are harder than pure ZTS crystals.

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