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Dielectric Properties of Proline Doped Triglycine Sulpho Phosphate (TGSP) Crystals

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Abstract

Effect of amino acid (proline) doping on the dielectric properties of triglycine sulphophosphate(TGSP) has been studied. Pure and proline doped TGSP were grown from aqueous solution by solution growth technique at room temperature. The dielectric studies were carried out to identify the phase transition temperature and the dielectric behavior. The present study shows that there is an increase in the Curie temperature for the doped crystals.

Key Words: Solution growth; TGSP crystals; Proline dopage; Dielectric constant; Dielectric loss; AC conductivity; DC conductivity

Introduction

Amino acid family of crystals over the years been subjected to extensive investigation by several researchers for their non linear optical properties [1,2]. The Crystal of many inorganic derivatives of amino acids provides excellent crystals such as triglycine sulphate, L-arginine phosphate accepted for the fabrication of devices [3]. Triglycine sulphate(TGS) is well known ferro electric material and it is widely used in fabricating thermal detectors, vidicons and image systems due to its second-order phase transition at room temperature and higher pyroelectric co-efficients as well [4,5]. The major drawback in TGS crystal in to depolarization of the device performance. To overcome this, organic and inorganic dopants have been tried. The organic and inorganic dopants in TGS have solved the problem of depolarization. In L-alanine added TGS, the presence of extra methyl group in the place of a hydrogen atom resulted in stabilization of the structure [6-9]. The added advantage of doping results in reduced dielectric permittivity and hence the pyroelectic figure of merit was higher that's the undoped TGS[10,11]. Even though doping enhances the characteristics of TGS, it could not alleviate the proliferation of microbes. Since glycine and co-substituted amino acids are rich in nutrient and hence reducing their shelf life. In order to overcome this microbial problem, the partial substitution of phosphate in TGS was tried and it gives rise to enhanced characteristic and shelf life time of the solution.

The phosphate incorporated triglycine sulphate(TGSP) crystal belongs to monoclinic system with space group of $2P_1$, in the ferroelectric phase. The lattice parameter values of TGSP are a=9.15Å, b= 12.69 Å and c=5.73 Å [12]. It has a wide ferroelectric phace and offers the and offers the possibility of fabricating with single element detectors [10]. It was also observed that the dielectric permittivity of TGSP was reduced as compared to pure TGS due to the extra proton available for charge compensation in TGSP[13-15]. The aim of the present work is to study the contribution of amino acid proline on the TGSP characteristics. Pure and proline (5 wt%, 10 wt%) added TGSP were grown from aqueous solution using solution growth method. Dielectric studies were carried out for all the grown crystals.

Experimental

Triglycine Sulphophosphate (TGSP) was synthesized according to the following chemical reaction.

$$3(NH_2CH_2COOH) + x\ (H_2SO_4) + (1-x)\ (H_3PO_4) \rightarrow (NH_2CH_2COOH)_3.\ (H_2SO_4)x\ (H_3PO_4)\ 1\text{-}x,$$

Where x was varied from 0 to 1 in steps of 0.25. Due to the aggregation of tiny poly crystals at higher molar concentration of orthophosphoric acid in TGS mother solution, the growth was restricted upto 50% of H_2SO_4 and 50% H_3PO_4 . For the synthesis of amino acid mixed TGSP proline (5 wt%, 10 wt%) were added separately in TGSP solution. After fully evaporating water, dried salts of pure and proline mixed TGSP were obtained and used for growth. After saturating the solutions, filtration was done using 0.2µm porosity nuclear filter after due preheating. Fifty milliliter of solution was poured into two glass vessels covered with perforated filter paper [16,17] and allowed to nucleate spontaneously at room temperature so as to get transparent seeds for the growth runs. To grow optical quality and strain free bulk crystals, the seeds obtained were tested under microscope. These seeds were placed in the respective saturated solutions at room temperature.Here the crystal growth began after 3 days of solvent evaporation and attained the maximum size. The grown crystals were carefully harvested and subjected to dielectric studies. The grown crystals were shown in figure 1.

The sample crystals cleaved perpendicular to the polar axis with thickness about 4 mm having graphite coating on the opposite faces was placed between two brass electrode and thus parallel plate capacitor was formed. The capacitance and dielectric loss (tan δ) were measured using the conventional two probe technique [18-20] at various temperatures ranging from 300 to 350 K using an LCR meter (Model APLAB) with frequency 1 kHz. The dielectric constant was calculated using the relation

$$\varepsilon_r = \frac{C_{cry} - C_{air}(1-(A_{crys}/A_{air}))}{C_{air}} \times (A_{air}/A_{crys}),$$

where C_{crys} is the capacitance with crystal (including air), C_{air} is the capacitance of air, A_{crys} is the area of the crystal touching the electrode and A_{air} is the area of electrode. The AC electrical conductivity (σ_{ac}) was calculated using the relation $\sigma_{ac} = \varepsilon_o \varepsilon_r \omega \tan \delta$ where ε_o is the permittivity of free space(8.85×10^{-12} Farad/m) and ω is the angular frequency ($\omega = 2 \pi f$, f = 1000).

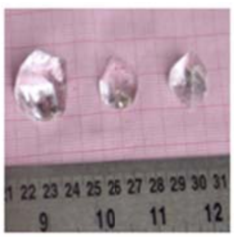

Figure 1: Photograph showing the pure and proline added TGSP crystals grown. (From left are : pure TGSP, 5 and 10 wt% proline added)

The resistance of the grown crystals were measured using a thousand meg ohmmeter. The observations were made while cooling the sample. The dimensions of the crystals were measured using a traveling microscope. The DC conductivity (σ_{dc}) of the crystal was calculated using the relation $\sigma_{dc} = d / (RA)$ where d is the thickness of the sample , A is the area of the sample and R is the measured resistance.

Results and Discussion

Dielectric studies are an essential parameter for all ferroelectric crystals to study their exact phase transition temperature. The effects of dopants on the phase transition temperature were identified through dielectric measurements[21]. The magnitude of the dielectric constant depends on the degree of polarization charge displacement in the crystals. The electronic exchange of the number of ions in the crystals gives local displacement of electrons in the direction of applied field that gives polarization. Ferroelectric domains are areas of such local dipole alignment with an associated net dipole moment and net polarization[16].In presence of an applied electric field, domains that are aligned with the direction of the field will grow at the expense of the less well aligned domains. Thus the observed enhancement in the dielectric constant at low frequency could be attributed to the multi-domain state of the proline doped sample.

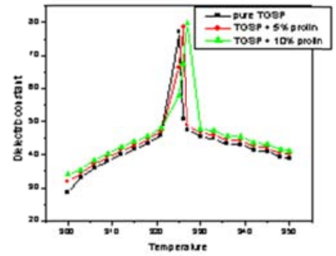

Figure 2 Variation of Dielectric constant with temperature.

The dielectric constant value increased and reached a maximum at the phase transition temperature after the permittivity decreased continuously to a constant value. Dipole ordering appeared at the phase transition temperature due to the domain nature of the as grown crystalline specimen. The sharp peak at the phase transition temperature reveals that the grown crystal was of the continuous photon transition nature[21].

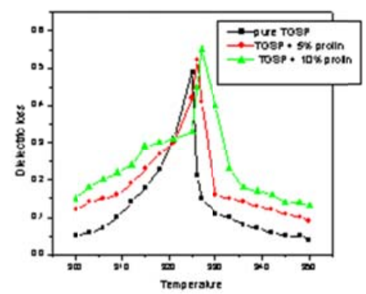

Figure 3 Variation of Dielectric loss with temperature

Figures 2 and 3 show the temperature dependence of dielectric constant(ε_r) and dielectric loss(tan δ) for pure and proline added TGSP crystals at 10^3 Hz. The temperature dependence AC conductivity for pure and proline added TGSP single crystals are provided in Figure 4.The Curie point T_c for all the samples were not found to be the same. A small shift in temperature is found for doped TGSP crystals. The dielectric constant is small at lower temperature, which increases with temperature and rises sharply up to the Curie point for this frequency. Above T_c the dielectric constants decreases suddenly and obey Curie-Wess law. Variation of dielectric constant with temperature is generally attributed to the crystal expansion, the electronic and ionic polarizations and the presence of impurities and crystal defects. The variation of dielectric constant at lower temperature is mainly due to the expansion and electronic and ionic polarization. At higher temperatures, the increase is mainly attributed to the thermally generated charge carriers and impurity dipoles. The dopants shifted the phase transition temperature slightly, but the shift was not considerable compared to the deuteration effect [22]. The dielectric loss values and AC conductivity also gives similar results. From the figure 3 the value of the dielectric loss(tan δ) at room temperature is low and this indicates that the grown TGSP crystal is of high quality[23-26].

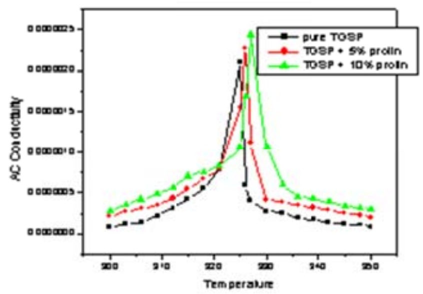

Figure 4 Variation of AC conductivity with temperature

The temperature dependence for DC conductivity is presented in Figure 5. The DC conductivity also increases with the increase in temperature. The defect concentration will increase exponentially with temperature and consequently the electrical conduction also increases. The addition of impurity further increases the electrical conduction in the temperature region considered[25.

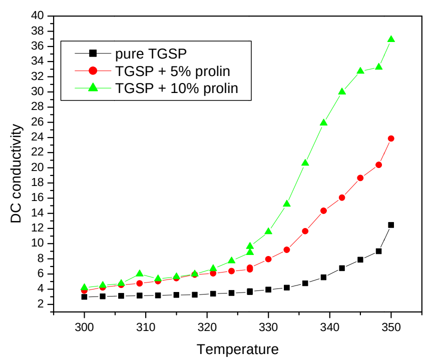

Figure 5. Variation of DC conductivity with temperature

Conclusion

Good quality single crystals of pure and doped, triglycine sulphophosphate ferro electric crystals were grown using solution growth method. The effect of dopant on the dielectric properties of the grown crystal has been studied. Shift in T_c shows that inhomogeneous dopant incorporation leads to micro regions in the crystal growth sectors with different transition temperatures.

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