CHAPTER - 9
SUMMARY, CONCLUSION AND FUTURE SCOPE

The summary of the research work, conclusion derived and future’s scope are provided in this chapter.

9.1. Summary

Today there is an increasing demand for crystals of complex composition containing multiple materials. Nowadays there would be no electronic industry, no photonic industry, no fiber optics communication, very little modern equipment without crystals. Everyday pure as well as mixed and doped crystals are grown in large quantities due to their wide applications. TGS is also one of the important crystal which are grown in everyday, due to its ferroelectric and pyroelectric properties. It shows second–order phase transition at curie point temperature \( T_c = 49^\circ C \)

In the present study, an attempt was made to grow pure as well as calcium and lanthanum doped TGS crystals by slow evaporation technique. The TGS was synthesized from glycine and concentrated sulphuric acid in the ratio 3:1, totally eleven crystals (1 pure, 5 calcium doped TGS and 5 lanthanum doped TGS) were grown under identical condition. Atomic Absorption Spectrum were taken to estimate the concentration of calcium contents present in the calcium doped TGS crystals whereas the concentration of lanthanum present in the lanthanum doped TGS crystals were determined using EDAX spectra taken.
An automated X-Ray diffractometer was used to collect the powder X-Ray diffraction data. The reflections were indexed using the conventional methods available. Lattice parameters were determined by available method. X-Ray powder diffraction intensity data were used to determine the thermal parameters like debye waller factor, mean square amplitude of vibration, debye temperature and debye frequency. Debye waller factors were determined by the Wilson plot method and the other above thermal parameter were determined from the debye waller factor using debye-waller theory. FTIR were recorded to confirm the presence of functional groups which were present in the raw materials.

The hardness and melting points of all the grown crystals were determined by Vicker’s microhardness measurement and TG/DTA curve respectively. Debye temperature have also been determined from the hardness value and the melting point temperature. Activation energy was also calculated from the thermogravimetric curve.

D.C and A.C for various frequencies viz. 50 Hz, 100 Hz, 1 KHz, 10 KHz, 50 KHz, 100 KHz and 200 KHz electrical measurements were done at various temperature ranging from 30°C to 120°C for all the eleven crystals by using megohm meter and parallel plate capacitor method respectively.

D.C electrical conductivity, dielectric constant ($\varepsilon_r$), dielectric loss factor ($\tan\delta$), A.C electrical conductivity ($\sigma_{ac}$), D.C and A.C activation energy and Debye temperature from A.C conductivity values were determined. Cole-Cole plot were also drawn from the complex dielectric constant value. SEM pictures were taken to exhibit the ferroelectric domain structure.
Impedance measurements were made for all the eleven samples in the frequency range 1 Hz to 1 MHz. UV-Visible spectra were taken for all the grown crystals to study the optical character of the grown crystals.

9.2. Conclusions derived

All the eleven crystals grown in the present study are stable and transparent and can be cut into required shape and polished. The dopant concentration (both Ca and La) determined from AAS and EDAX confirm the incorporation of dopant in to the doped crystals. FTIR spectra taken for all the eleven crystals confirm the presence of all the functional groups expected. The indexed X-Ray powder diffraction data show that all the grown crystals in the present study belong to monoclinic system.

Debye temperature determined from debye waller factor for pure TGS crystal agreed with the reported value. The dopant has non-linear influences on debye temperature for doped crystal. Debye frequency determined in the present study is in the infrared range.

The Vicker’s hardness value determined for the calcium doped crystals increases with calcium concentration and for lanthanum doped crystals it decrease with the increase in lanthanum concentration. Work hardening coefficient determined for all the eleven crystals implied that they belong to hard material category. Debye temperature calculated from the hardness value is greater than those calculated from the debye waller factor except for 0.010 LaTGS crystal.

The melting point temperature observed from the TG/DTA curve for the pure TGS crystal shows close agreement with the reported value. There was no significant change in the melting point of doped crystals. Debye temperature calculated from the
melting point temperature exactly match with the debye temperature calculated from the debye waller factor. Activation energy calculated from the mass change with respect to temperature in the TG/DTA curve for pure TGS agreed with literature value.

The D.C conductivity determined in the present study increases with increase in temperature but the dopant concentration has non-linear influence on the D.C conductivity for both calcium and lanthanum doped TGS crystals. It is also seen that the conductivity of doped crystals are less than pure TGS crystal. The dielectric constant determined for all the eleven crystals is higher at the curie point temperature irrespective of the applied frequencies and it decreases with increase in frequency. The dielectric loss factor also show the similar variation as in the case of dielectric constant.

The A.C conductivity determined for all the grown crystals are maximum at curie point temperature. Cole-Cole plot drawn in the present study for all the eleven crystals exactly fit into semicircle with different radius. The radius of the semicircles at curie point temperature is maximum compared to the other temperatures for all the eleven crystals. The radius of the semicircle at 70°C is greater than that for 30°C. The radius of the semicircle is increases with the dopant concentration.

The SEM picture shows rod-like structure parallel to the b-axis. The domain size of the grown crystals vary non-linearly with dopant concentration.

The Nyquist plot drawn for all the grown crystals have three relaxation frequencies and it also fit into a semicircle.
The $\lambda_{\text{max}}$ value decreases with increase in the calcium concentration for calcium doped TGS crystals whereas it increases with increase in concentration of lanthanum for lanthanum doped TGS crystals.

### 9.3. Future scope

Crystal growth and characterization is a thrust area of research in the field of crystalline materials science. This has got scientific as well as technological importance.

TGS is a order-disorder type ferroelectric and pyroelectric material. So our first future scope is to study the ferroelectric hysteresis nature of our crystal. Sawada et al showed that the formation of solid state battery by ferroelectric materials is interesting and has promising applications. So, our next future scope is to fabricate a solid state battery by using the crystal grown by us. Recently, Deepti et al shown that KDP doped TGS crystals is suitable for optoelectronic applications. In this view, we also want to fabricate a device for optoelectronic applications by using our grown crystals.

In the present study, only two rare earth elements viz. calcium and lanthanum were doped. In the future, study may extended to other rare earth elements.