Growth and Characterization of Deuterated L-Alanine Doped Triglycine Sulfate (DLATGS)

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Introduction

This is a presentation about the growth and characterization of L- Alanine doped triglycine sulfate(DLATGS). DLATGS is a ferroelectric material primarily used in applications of infrared detectors and imaging systems. Triglycine Sulfate (TGS) is the basis of the ferroelectric property, these crystals have the ability to spontaneously polarize under the application of an external electric field. This phenomena takes place under the Curie temperature of 49℃ [1]. In order to improve the performance of IR detectors at room temperature, the Curie temperature can be raised by deuterating the crystal to form (DTGS). DTGS has a Curie temperature of 57℃[1], this material however still needs an applied external electric field to polarize.

By doping the DTGS with the amino acid L-Alanine, the crystal properties are improved by contributing to effective internal bias which inhibits ferroelectric switching giving a permanently poled single domain crystal[2]. DLATGS retains a high pryoelectric coefficient while still having a low dielectric constant. This allows DLATGS to be widely used in detection equipment.

Synthesis

The crystals were grown using high grade glycine (CH_2NH_2COOH) and concentrated sulfuric acid (H_2SO_4). A glycine to sulfuric acid molar ratio of 3:1 was used from the chemical equation:

$$3(NH_2CH_2COOH)+H_2SO_4 \rightarrow (NH_2CH_2COOH)_3{*}H_2SO_4$$

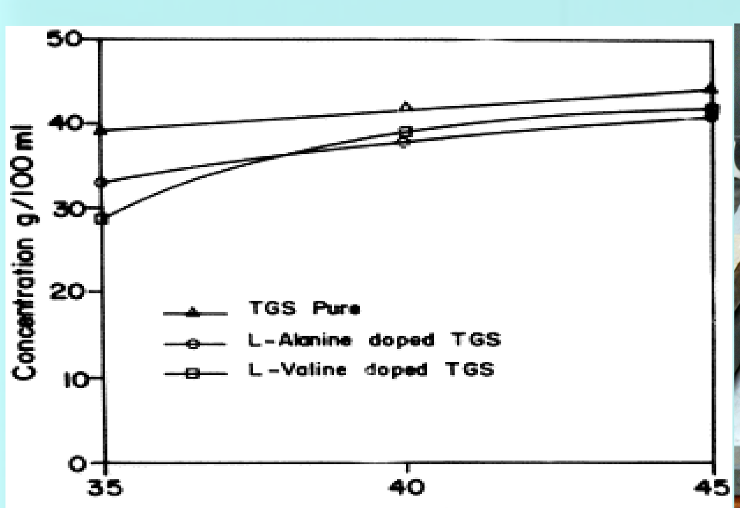


Fig.1[2]

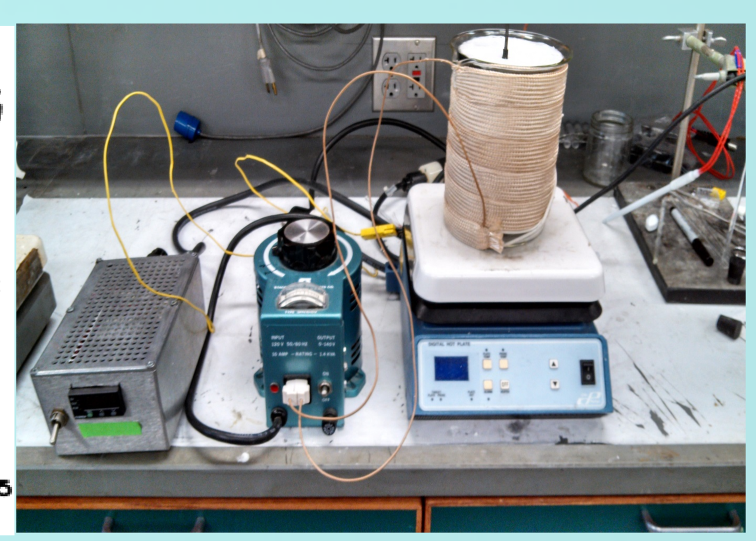


Fig.2

From Fig.1 the solubility of pure at 35℃ is 41.85g/100mL[2]. From this we calculated that 1 liter of water will yield≈ 420g of TGS. From this the calculated values for 1 liter of water 295g of glycine and 128.4g(68mL) of sulfuric acid was needed. Scaling down to a 400mL of deionized water, the amount of glycine added was 100g and 27.2 mL of sulfuric acid. The sulfuric acid was slowly added to the DI water followed by the glycine.

The solution was heated to 55℃, and stirred to dissolve all reactants. The hot plate was turned off and the solution was left overnight to cool and slowly evaporate. Good nucleation was observed and a seed crystal was chosen. The solution was re-dissolved to the saturation temperature in which the seed was re-introduced into the solution in the apparatus shown in Fig.2. A programmable controller was connected from a variable transformer to heating coils, the program allowed slow cooling of 0.5℃ for 10 days to grow a single bulk crystal. This TGS crystal is shown in Fig.3.

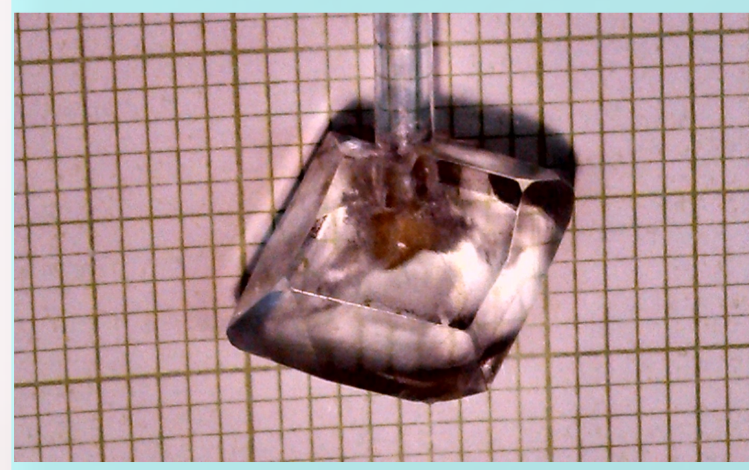


Fig.3

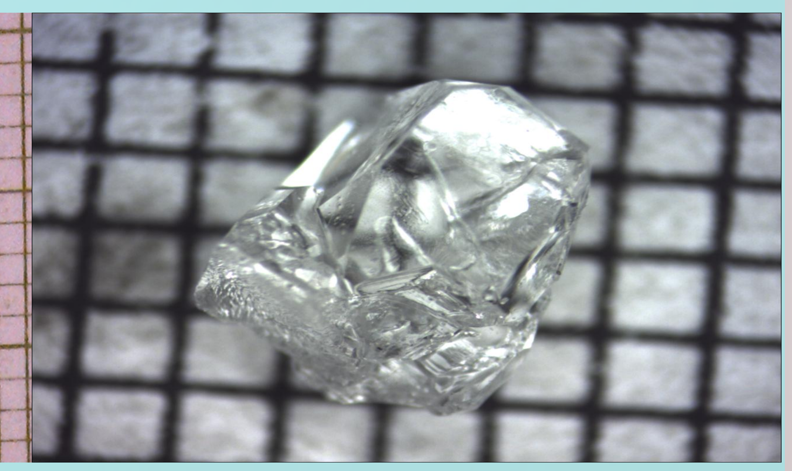


Fig.4

The same process was used to form DLATGS, the changes made were using deuterated water and adding 20 weight% L-Alanine to the solution. The yielded nucleation is shown in Fig.4. A single bulk DLATGS is in the process of being grown in the Fig.2 apparatus.

Characterization

The crystals were both ground into a fine powder and analyzed using a powder X-Ray diffraction machine. The resulting data was collected for TGS(Fig.5) and DLATGS(Fig.6).

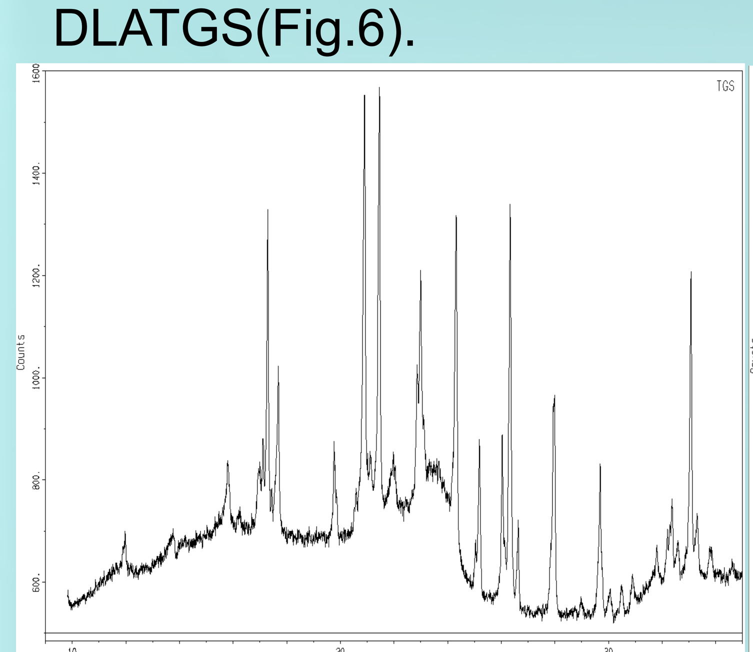


Fig.5

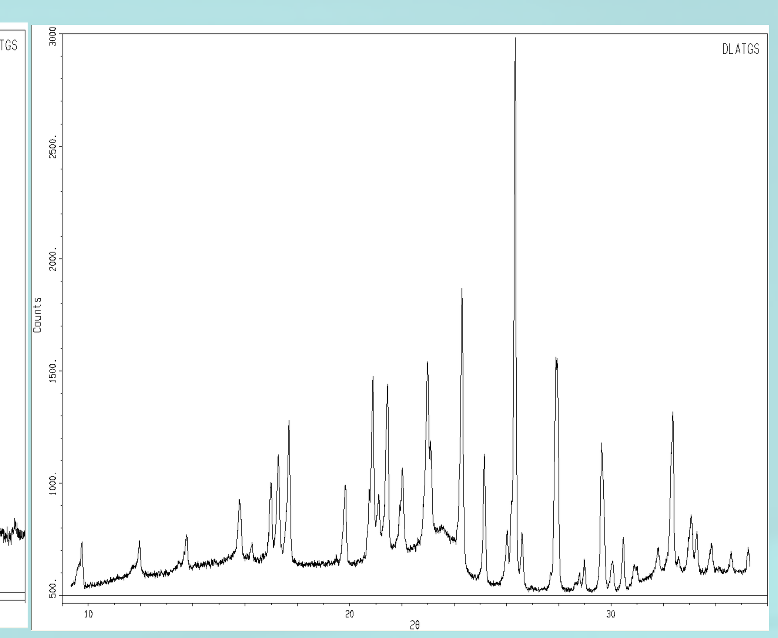


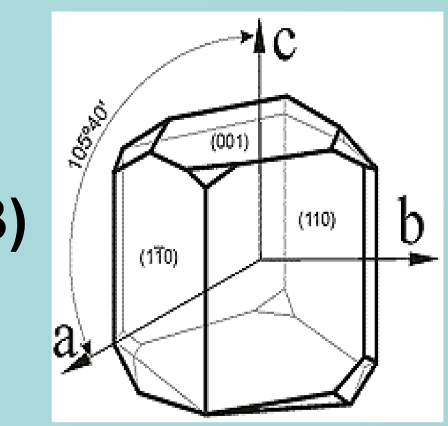
Fig.6

The data was compared to a peak fit model using the known unit cells of TGS and DLATGS shown in Fig.7. The comparison yielded very similar peak trends, not however exact. The next characterization test will be on a single crystal X-Ray diffraction machine, to yield a unit cell.

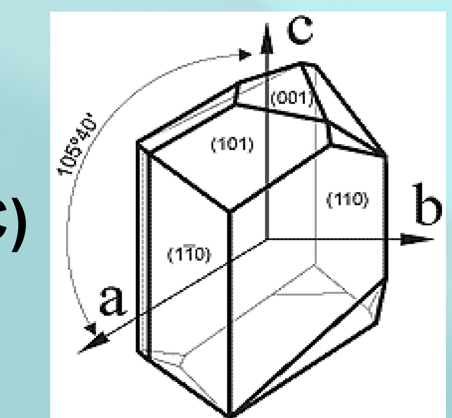
Samples	a (Å)	b (Å)	c (Å)	β °
Pure TGS	5.7254	12.6445	9.1583	105.5318
ATGS	5.7457	12.6093	9.0897	105.3251

Fig.7(A,B,C)

TGS(B) [2]



DLATGS(C) [2]



Conclusion

This purpose of this project was to synthesize pure tri-glycine sulfate and deuterated L-Alanine doped tri-glycine sulfate. Overall the experiment was executed successfully and yielded single crystals of TGS and DLATGS. The result of the single crystal XRD analysis will prove the accuracy of the unit cell.

Future Works

This project will continue will different doping materials and growth apparatus's, also optoelectronics properties and figures of merit will be measured to test the pyroelectric performance. A temperature controlled continuous stirred batch reactor will be used to grow a single bulk DLATGS crystal in the near future.

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References

[1] Batra, A. "Growth and Characterization of Doped DTGS Crystals for Infrared Sensing Devices." *Materials Letters* 57.24-25 (2003): 3943-948. Print.

[2] Aravazhi, S., R. Jayavel, and C. Subramanian. "Growth and Stability of Pure and Amino Doped TGS Crystals." *Materials Chemistry and Physics* 50.3 (1997): 233-37. Print.