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Growth and thermal, micro hardness and NLO studies of guanidinium L-monohydrogentartrate L-tartaric acid (GuHTT) single crystals

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Abstract: The guanidinium L-monohydrogentartrate L-tartaric acid (GuHTT) crystals were grown from aqueous solution by slow evaporation technique. The Good optical quality crystals of GuHTT (dimensions 28x7x5 mm3) were harvested after three weeks. The grown crystals of GuHTT have been subjected to various characterization studies such as UV-Vis spectrum, TGA/DTA, powder SHG test and Micro hardness studies. The above experimental result viz., bulk size, extremely good crystalline perfection, optical transparency, high SHG efficiency show that GuHTT have possible NLO applications.
Key Words: SHG; NLO applications; Hardness.

1. Introduction

Organic nonlinear materials make a drastic change in electronic and optical technologies in these days. These materials have been investigated widely due to their rapid response in optical devices because of their large optical nonlinearity compared to inorganic NLO materials [1]. Organic guanidine carbonate material has enhanced NLO efficiency compare to KDP. Guanidine is a strong base reacts with organic and inorganic acids resulting in the formation of guanidinium species useful for designing molecular complexes with exactly planned chemical and physical properties suitable for non linear optics [2]. Arumuganayagam et al., [3] recently reported the optical and dielectric behavior of GuHTT. In this paper we report the solubility, mechanical hardness and thermal properties of GuHTT.

2. Experimental procedure

An organic NLO crystal GuHTT was synthesized from Guanidine carbonate and Tartaric acid. The GuHTT crystal was grown by slow evaporation solution method. Guanidine Carbonate (AR) and Tartaric acid (AR) were taken in the molar ratio of 1:1 and dissolved in double distilled water. The solution is stirred well for 31/2 hrs with the magnetic stirrer. The solubility of GuHTT was measured by gravimetric method. Fig.1 shows that solubility of GuHTT increases with increasing temperature having positive temperature coefficient of solubility, highly suitable for growing bulk size crystals by slow cooling method.

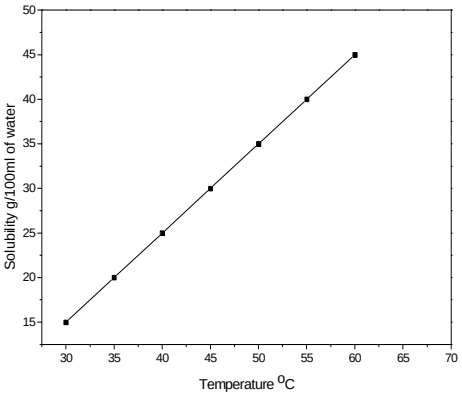

Fig.1 Solubility of GuHTT Crystal

3 Results and Discussion

3.1 Single crystal XRD analysis

The GuHTT crystal was subjected to single crystal X-ray diffraction studies using Enraf Nonius-CAD4 single crystal X-ray diffractometer with MoKα radiation (λ = 0.7107 Å) to estimate the lattice parameters values. The X-ray diffraction experiment shows that GuHTT crystal belongs to orthorhombic crystal system with non-centrosymmetric space group $P2_12_12_1$. The cell parameters of GuHTT crystal obtained are, a = 11.339 (3) Å, b = 11.146 (3) Å, c = 6.657 (3) Å and v = 841.33 Å. The obtained lattice parameter values are in good agreement with the reported literature values [4].

3.2 Optical analysis

The UV- Vis spectrum of GuHTT crystals was carried out using Shimadzu 1601UV-Vis spectrophotometer. To determine the transparency in the visible region and hence to know the optical properties of the GuHTT crystals the UV- Vis spectrum was recorded in the range of 300-900nm. From the Fig. 2 it is seen that UV transparency cutoff occurs around 235 nm. The transparency is around 98% in the visible region. The good transmission property of the crystal in the entire visible region suggests its suitability for a second harmonic generation.

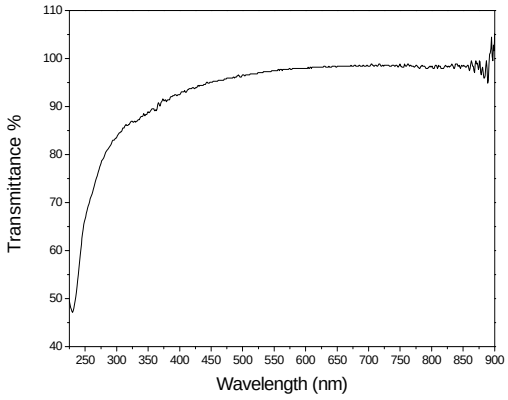

Fig. 2 UV- Vis Transmittance spectrum of GuHTT crystal

3.3 SHG measurements

The Nonlinear optical property of the crystal was carried out by kurtz and perry technique [5]. The fundamental beam of 1064nm from Q- switched mode locked, Nd: YAG Laser with a pulse width of 8ns. The powdered sample with average particle size of 100-150µm was used to test the second harmonic generation property of GuHTT. The second harmonic signal generated in the crystal was confirmed from the emission of green radiation by the crystal. The amplitude of the SHG output voltage was measured using a photomultiplier and a digitalizing oscilloscope assembly. The SHG efficiency of GuHTT was found to be 1.2 times of KDP.

3.4 TGA/DTA analysis

The TGA/DTA curve of GuHTT is shown in Fig.3. The exothermic peak at 98ºC indicates the evaporation of water molecule from the crystal lattice. The sample GuHTT was stable up to 284^0 C. And there is a weight loss between 288^0 C - 388^0 C which was assigned to decomposition of GuHTT crystal. From the observation it was concluded that the crystal decomposes only at 288°C. The sharp exothermic peak shows the good degree of crystallinity of the sample.

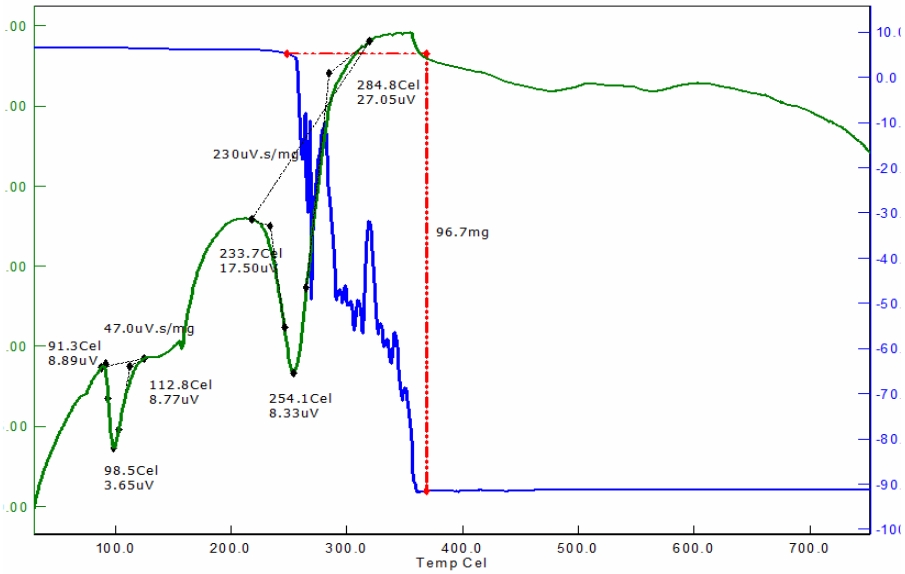

Fig. 3 TG/DTA Spectrum of GuHTT crystal

3.5 Hardness

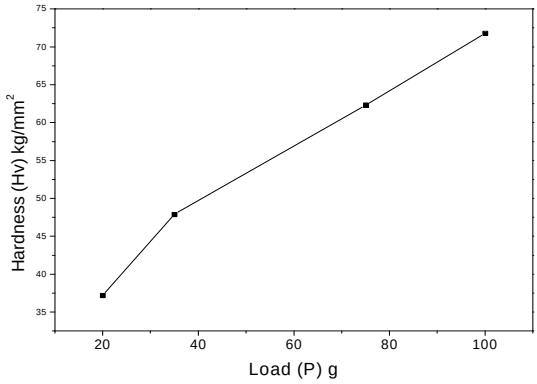

Fig. 4 Hardness number (Hv Vs Load P)

The micro hardness value for GuHTT was measured using Vickers Micro hardness tester. Selected smooth and flat surface in (100) orientation of GuHTT crystal was subjected to hardness study at room temperature with the load range from 20 to 100 g. Fig. 4 shows the Harness value (Hv) increases with increasing applied load which indicates the reverse ISE [6].

Conclusion

The good optical quality NLO single crystals of GuHTT were grown by slow evaporation technique. GuHTT possess positive temperature coefficient solubility. The second harmonic efficiency of the crystal was found to be 1.2 times greater than that of KDP. The sustainable mechanical stability is identified from Vickers micro hardness study. TGA/DTA curve show the good degree of crystallinity of GuHTT. The above properties confirm that the title compound has enhanced application towards device fabrication.

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