International Journal of ChemTech Research
CODEN (USA): IJCRGG ISSN: 0974-4290
Vol.7, No.01, pp 408-411, 2014-2015

Thermal, spectral and mechanical analysis of Glycine Potassium Penta Borate Octa Hydrate (GPPB) crystal

Kamatchi K[1]*, Gopinath S[2] and Radhakrishnan T[1]

[1]SRC, Department of Physics, SASTRA University, Kumbakonam,India.
[2]Arunai Engineering College, Thiruvannamalai, India

Abstract : In this present communication, we have grown Glycine potassim penta borate octa hydrate (GPPB) crystals by slow evaporation solution growth technique. To know its suitability for device fabrication, different characterization analyses have been performed. By powder X-ray diffraction (PXRD) method the lattice constants have been calculated. It is found that it crystallizes in monoclinic crystal system. The thermal stability of the crystal was examined by TG/DTA analysis. Its relative second harmonic generation efficiency was evaluated from Kurtz powder technique. The optical transmission property of the crystal was inspected by UV-vis-NIR spectral analysis. The mechanical property of the crystal was tested by Vicker's microhardness tester
Key words : Crystal growth, FTIR, UV-vis-NIR, TG/DTA,SHG.

Introduction

Nonlinear optical (NLO) crystals are widely used in harmonic generation, switching and other optical signal processing devices. So, the search for new materials possessing high optical nonlinearity is an important task. Some complexes of amino acids with inorganic salts are reported to be promising materials for optical second harmonic generation [1–3]. Amino acids with inorganic compounds are promising materials for nonlinear optical applications, as the high optical nonlinearity of the purely organic amino acid tends to combine with the favorable mechanical and thermal properties of the inorganic salt. Glycine is the simplest amino acid which has no asymmetric carbon and is optically active [4]. Glycine sodium nitrate crystals grown by Narayan Bhat and Dharmaprakash [1] had second harmonic generation efficiency (SHG) two times that of potassium dihydrogen orthophosphate (KDP). Nagaraja et al. [5] reported that the SHG efficiency of benzoyl glycine crystal was 1.5 times that of KDP. In this series, we have successfully grown glycine potassium penta borate octa hydrate crystals and the results of characterization of these crystals have been reported in this communication.

Experimental

Crystal Growth

Commercially available potassium penta borate octa hydrate (AR grade) and glycine were dissolved in the ratio 2:1 in deionized water until a saturated solution was obtained. The solution was stirred well using a magnetic stirre.Then the solution was filtered and crystallization was allowed to take place by slow evaporation under room temperature. Optically transparent crystal of average size 7x5x2mm^3 was harvested within 10 days.

Characterization

Single crystal X-ray diffraction analysis was carried out using ENRAF NONIUS CAD-4 X-ray diffractometer with MoK_ (_=0.1770 Å) to determine the lattice parameter values. To assess the SHG efficiency, Kurtz powder technique was performed on the grown crystals. The FTIR spectrum was recorded in the range 400-4000 cm^{-1} employing a Perkin-Elmer spectrometer by KBr pellet method to assign functional

groups present in GPPB. To study the linear optical properties, the optical absorption spectra was measured in the range 200 to 1100nm using the instrument Lambda-35 UV-Vis-NIR spectrophotometer. The microhardness measurements for the grown crystals were made using Leitz-Wetzlar microhardness tester fitted with a Vicker's diamond pyramidal indentor.

Results and discussions

Single crystal X-ray diffraction analysis

The unit cell parameters of GPPB crystal were obtained from the single crystal X-ray diffraction analysis. They were found to be a = c = 7.188Å, b = 11.058 Å and the cell volume is 560 $Å^3$ with $\alpha = \gamma = 90°$ $\beta = 101.83°$. The structure was solved by the direct method using the SHELXL program. It was found that the crystal belongs to monoclinic crystal system with the space group of P21/n.

FTIR spectral analysis

Recording FTIR spectrum is much useful to identify the functional groups present in a compound. This spectroscopy is used to examine the twisting , bending , rotating and vibrational modes of atoms present in a molecule. The FTIR spectrum recorded for our crystal is as shown in Figure.1. The bands observed at 506,606 and 693 cm^{-1} are assigned to carboxylate groups. The peaks at 1490 and 1125 cm^{-1} are due to NH_3 group [6]. The peaks due to NH_3^+ group of free glycine are found at 3173, 2616, 1605, 1515 and 1116 cm^{-1} [7]. The presence of C=O bond is confirmed by the peak at 1322 $cm^{-}1$ [8] .The band at 897 cm^{-1} indicates the CH_2 vibration [8].

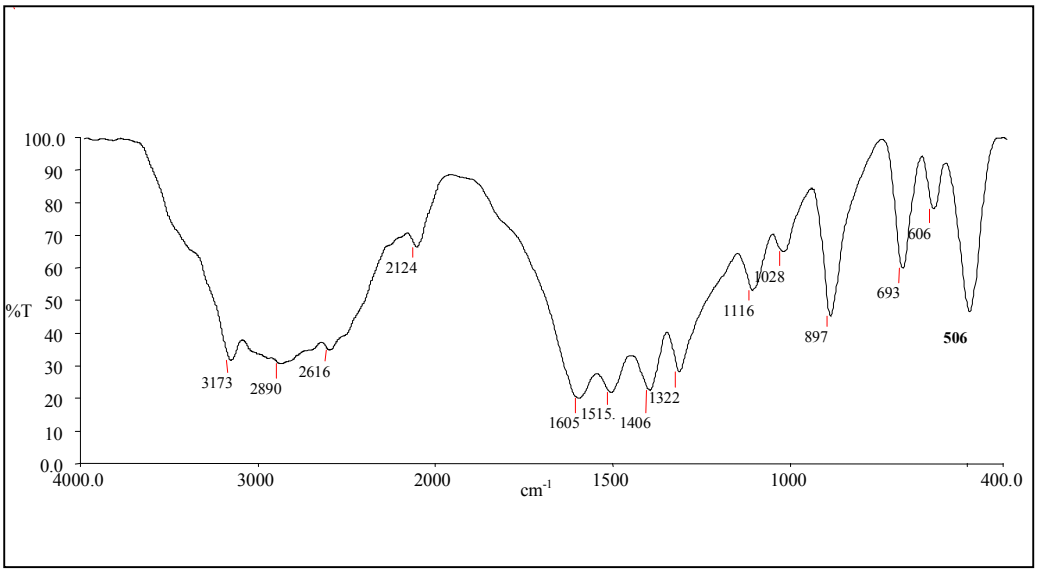

Figure 1 FTIR spectrum of grown GPPB crystal

Optical transmission studies

Analysis of optical transmission property of a crystal is useful for choosing the crystal to photonic applications. Figure 2 shows the UV–vis-NIR optical absorption spectra of GPPB recorded in the range 190–1100 nm. Optically polished single crystal of thickness about 2 mm was used for this study. The crystal possesses good transmission for UV, visible and IR regions. The transparency is comparable with other glycine compounds [9-11].

Nonlinear optical studies

In order to estimate the non linear optical property of the crystal, the grown crystal was subjected to second harmonic generation (SHG) efficiency test. An Nd: YAG laser beam was sent through the crystal and emission of green light was observed. This confirms the SHG efficiency of the crystal.

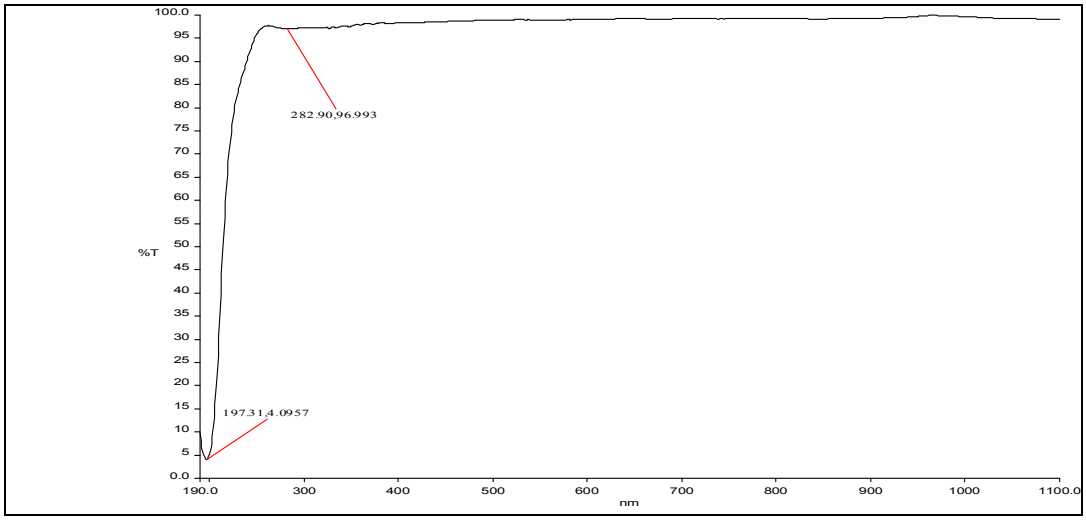

Figure 2 UV-vis-NIR spectrum of GPPB crystals

Thermal Analysis

For device fabrication, the information about the thermal stability is required. In order to find the thermal properties of the grown crystal, Thermo gravimetric analysis (TG), and Differential thermal analysis (DTA) were carried out for the crystal. The TG/DTA was carried out between 25° C and 1400° C in nitrogen atmosphere at a heating rate of 20° C/min. The TG/DTA curves were shown in Figure 3.

From the TG curve it is observed that there is no weight loss up to 172° C which indicates the good thermal stability of the crystal and also confirms the absence of water molecule. In the temperature range 172-210° C, only 26 % of the compound is melt. Above 210 ° C, there is no weight loss. In the DTA spectrum there is an endothermic peak at 172° C, which indicates the melting point of the sample which is well agreeing with the TG result. Hence the grown crystal is stable up to 172° C.

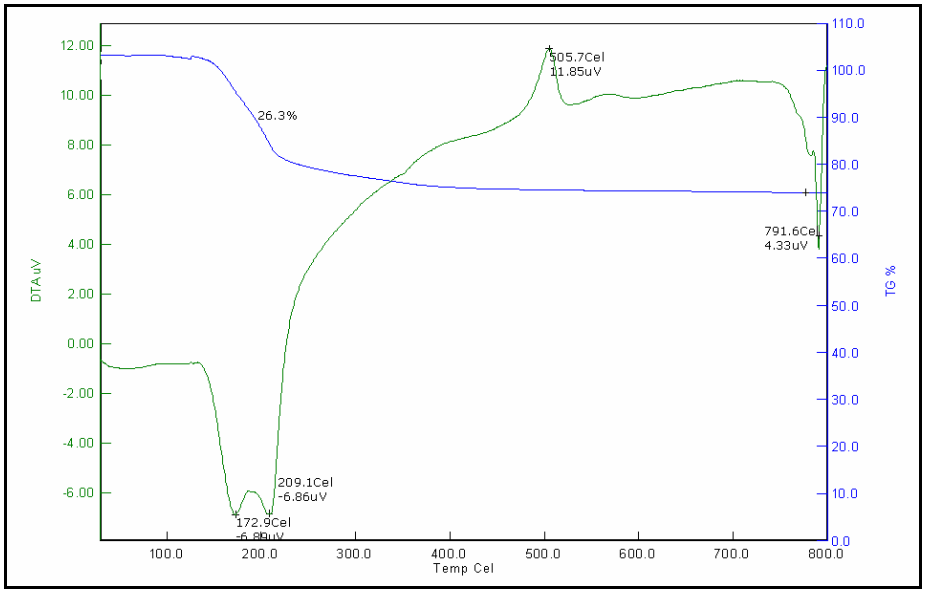

Figure 3 TG-DTA curves of grown crystals

Microhardness studies

Mechanical strength of GPPB crystals was studied by measuring microhardness and it plays an important role in the fabrication of opto-electronic devices. The Vicker's hardness number was estimated for various loads from 25 g to 100 g. The plot of load versus hardness number is shown in Figure 4. From the graph, it is observed that the hardness number increases with the increase in load. The hardness value indicates the good mechanical stability of the material.

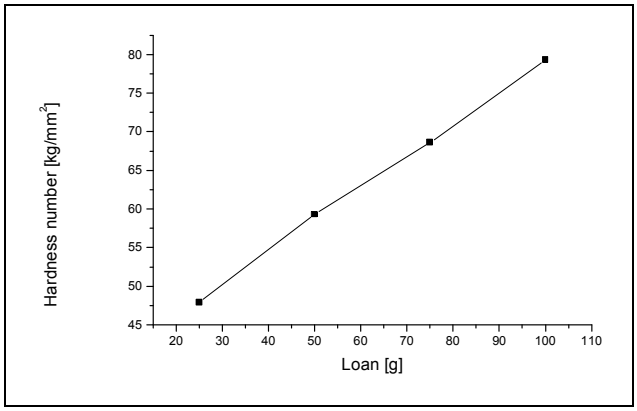

Figure 4 Load Vs Hardness number

Conclusion

By slow evaporation method, Glycine mixed potassium penta borate octa hydrate crystals were grown. The functional groups present in the material were identified using FTIR spectral analysis. The optical transparency of grown crystals was analyzed with the help of UV-vis-NIR spectrum and it was observed that the crystal possesses wide range of optical transparency which indicates the use of crystal in the photonic device applications. The emission of green light from the crystal for the input of Nd: YAG laser confirms the SHG efficiency of the crystal. From the TG-DTA analysis carried out for the crystal, the thermal stability of the crystal is found to be 172° C. The mechanical property of the crystal was examined by Vickers micro hardness test.

References

1. M. Narayan Bhat, S.M. Dharmaprakash, J. Cryst. Growth 235 (2002) 511.
2. A. Deepthy, H.L. Bhat, J. Cryst. Growth 226 (2001) 287.
3. R. Rajasekaran, P.M. Ushashree, R. Jayavel, P. Ramasamy, J. Cryst. Growth 229 (2001) 563.
4. T. Balakrishnan, K. Ramamurthi Spectrochimica Acta Part A 68 (2007) 360–363 Structural, thermal and optical properties of a semiorganic nonlinear optical single crystal: Glycine zinc sulphate
5. H.S. Nagaraja, V. Upadhyaya, P. Mohan Rao, P. Sreeramana Aithal, A.P.Bhat, J. Cryst. Growth 193 (1998) 674.
6. T. Balakrishnan, R. Ramesh Babu, K. Ramamurthi Spectrochimica Acta Part A 69 (2008) 1114–1118 Growth, structural, optical and thermal properties of γ-glycine crystal.
7. Senthil pandian M and Ramasamy P , Growth and characterization of solution-grown tetra glycine barium chloride single crystals , J Cryst. Growth 310 (2008) 2563-2568.
8. Palaniswamy S and Balasundaram O N, Effect of pH on the growth and characterization of gylcine sodium chloride single crystals, Rasayan J Chem. Vol.1, No.4 (2008),782-787.
9. B. Riscob , Mohd. Shakir ,V. Ganesh ,N. Vijayan , M. A. Wahab , G. Bhagavannarayana, Growth, optical, mechanical and thermal studies of diglycine cadmium chloride single crystal, J Therm Anal Calorim,DOI 10.1007/s10973-011-2110-8.
10. S. Sampthkrishnan, N. Balamurugan, R. Kumutha, Y. Vidyalakshmi, S. Muthu ,Journal of Minerals & Materials Characterization & Engineering, Vol. 11, No.6, pp.597-607, 2012.
11. Thilagavathy and Ambujam , Growth and Characterization of New Non Linear Optical Bis-Glycine Hydro Bromide (BGHB) Single Crystal Trans. IIM Vol. 64, Issues 1 & 2, February-April 2011.

*****