International Journal of Mathematics and Physical Sciences Research ISSN 2348-5736 (Online)
Vol. 3, Issue 1, pp: (162-165), Month: April 2015 - September 2015, Available at: www.researchpublish.com

Growth and Characteristics of Semi Organic Non-Linear Optical Material –Bismuth Thiourea Zinc Acetate (BTZA) Single Crystals

S.PRIYA

RESEARCH SCHOLAR –CHENNAI

Abstract: **Semi organic Nonlinear optical material bismuth thiourea zinc acetate (BTZA) single crystals were grown by slow evaporation technique. Solvent used here was water. The solubility range is about 25-45C whose PH value is 3.1.The structural parameters of grown crystal were estimated by XRD. Spectral bands have been compared with thiourea complex using FTIR in the range 400-4000cm^{-1}. FTIR studies confirm the functional groups present in the grown crystal. During the making of this crystal mechanical stress was applied on the crystal while cutting and polishing. Using Vickers's hardness test hardness of crystal was estimated.NLO property was confirmed using Nd: YAG laser of wavelength 1064nm and efficiency was estimated three times higher than that of KDP.**

Keywords: **Slow evaporation technique, XRD, FTIR, Thermal analysis and SHG.**

1. INTRODUCTION

Modern technology is based on non linear optical single crystals and much attention is paid on growth of non linear optical materials which is a part of material science. The field of non linear optics emerged nearly five decades ago with the development of first operating laser and demonstration of frequency doubling phenomenon. Recent studies reveal that non linear optical materials with remarkable optical non linearity find interesting applications in frequency doubling, telecommunication, optical information processing and high optical data storage. Organic materials have limitations due to volatility, low thermal stability; poor mechanical strength e.t.c. Inorganic materials have more mechanical, thermal and chemical stabilities.Semiorganic nonlinear optical material can combine with high optical nonlinearity physical and chemical stabilities. Linear optical properties crystalline materials based on Thiourea have attracted the researchers as legend Thiourea possess large dipole moment and an ability to form an extensive network of hydrogen bonds. The centrosymmetric thiourea molecule combined with inorganic salt yield noncentro symmetric complexes with high optical nonlinearity. In this paper characteristic of BTZA single crystals were studied.

2. SYNTHESIS AND CRYSTAL GROWTH

Aqueous solution of zinc acetate and thiourea were taken and mixed in stoichiometric ratio1:2. BTZA salt was synthesized .Solvent used here was water .It is non toxic and easily available in pure state. It provides reasonably wide range for selection of growth temperature.

$Zn\ (CH_3COO)_2 + (NH_2CSNH_2)_2 = ZN(NH_2CSNH_2)_2\ (CH_3COO)_2$

Using magnetic stirrer the required quantities of compound was stirred and dissolved in double distilled water .To improve the purity of synthesized salt successive recrystallisation was carried out. Recrystallisation followed by filtration of solution will increase the level of purity. The crystal was grown over a period of 25 days.

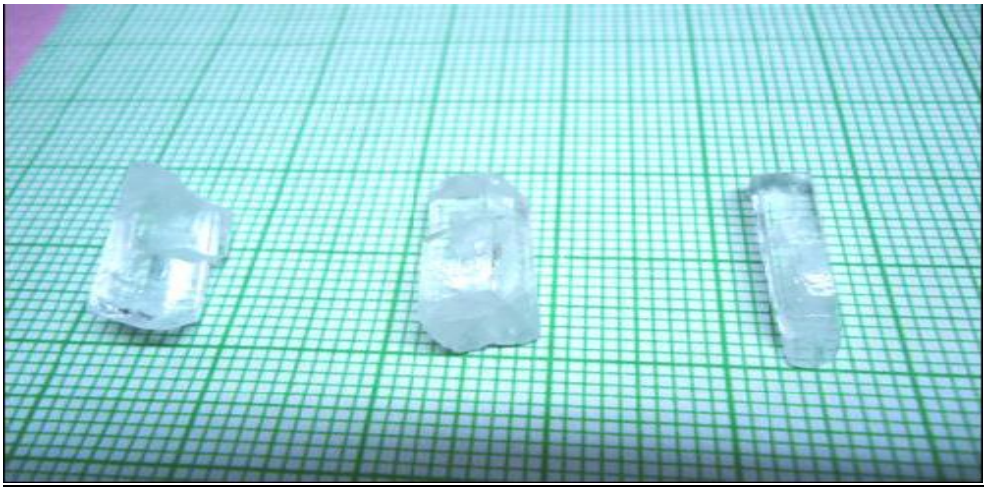

Figure: 1

3. EXPERIMENTAL PROCEDURE

3.1 Solubility:

The solubility of solute was determined by dissolving the solute in the solvent (water) with continuous stirring in an airtight container. This was done at constant temperature. An efficient stirring of solution prevents layering and spurious nucleation. Repeat this process at different temperature range from 25 to 45*C. The solubility curve can be plotted in this way by repeating the above for different temperature range .The growth depends on shape of solubility curve. The effects of PH value on the crystals have impact on solubility which ranges about 3.1. It has been found that solubility of BTZA increases with increase in temperature.

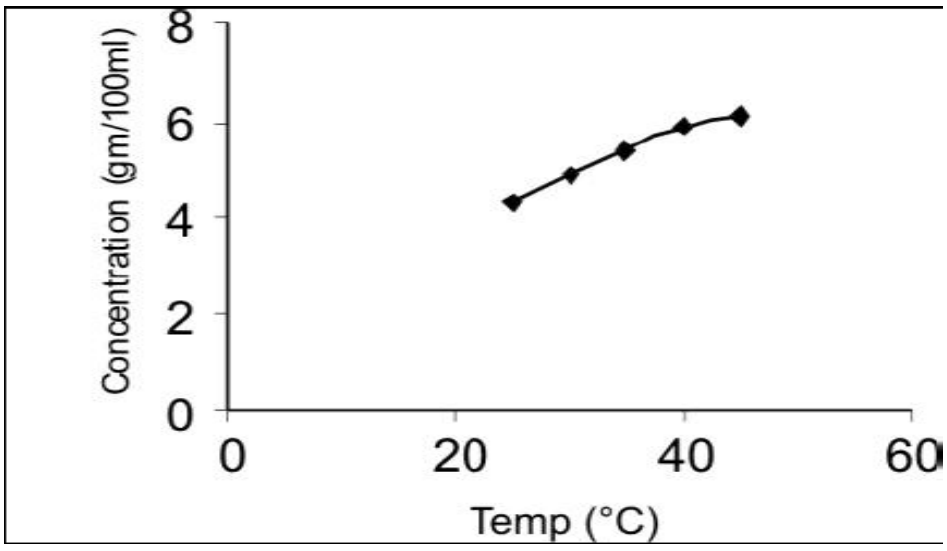

Figure: 2

4. RESULTS AND DISCUSSION

4.1 Single crystal XRD:

It is used to determine phase composition, grain size and internal stresses. To determine unit cell dimension and morphology of crystal XRD is used. X- ray beams are diffracted by crystal lattice of sample and produce certain level of intensity in the detector. The cell parameters are a=7.13 A, b=17.70 A, and C=11.16 A. This has been confirmed by Seifert Xray diffractometer with CuK_{α} (λ=1.541 A) radiation. The sample was scanned from 10-70*C at a scan rate of 1*/min.These angles were characteristic of lattice spacing of the sample. Measurements are carried out in vacuum and diffraction peaks were shown.

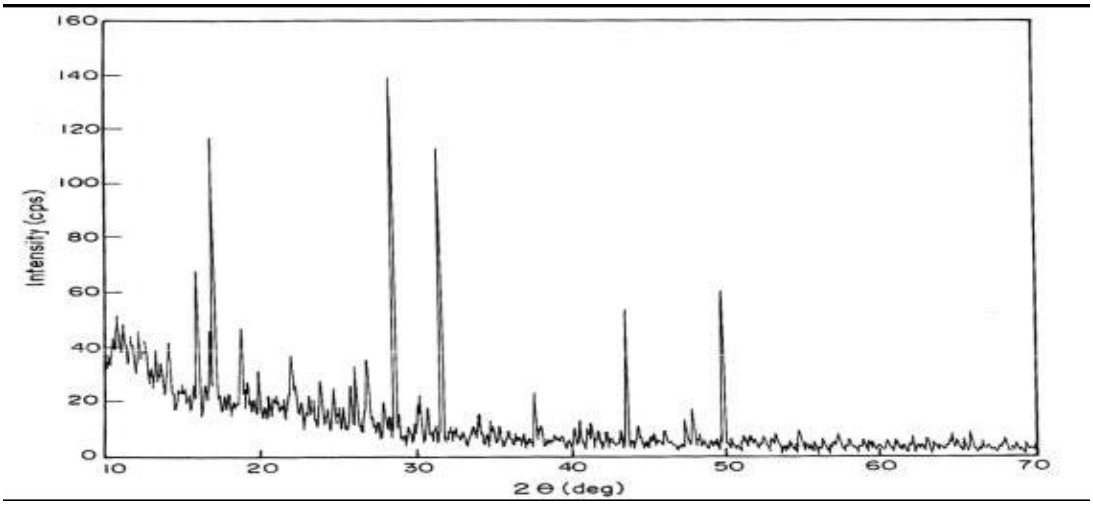

Figure: 3

4.2 FTIR:

FTIR is used to identify chemical composition of specimen based on IR radiation. The powdered specimen of BTZA crystal is subjected to FTIR using KBR pellet technique in the wavelength range 400 &4000cm^{-1}.The resulting pellet is transparent to IR radiation. The observed bands with vibrational assignments were tabulated. The size of peaks in a spectrum is a direct indication of amount of material present. The high frequency N-H absorption bands in the region 3100-3400cm^{-1} in the spectrum of thiourea have not been shifted to lower frequency on formation of metal thiourea complex which indicates that bonding is between sulphur and zinc and not of nitrogen and zinc. The symmetric and asymmetric C=S stretching vibration at 730 and 1417cm^{-1} of thiourea are shifted to low frequency at 677 and 1403cm^{-1} in BTZA.This confirms the formation of metal sulphur coordination bond. Stretching frequency is higher than bending frequency. All the frequencies were measured simultaneously in a matter of seconds.

Table: 1 ASSIGNMENT OF IR BAND FREQ CM^{-1} OF THIOUREA AND BTZA

Thiourea cm^{-1}	BTZA cm^{-1}	ASSIGNMENT
494	480	S-C-N symmetric
730	677	C=S stretching
1089	1130	C=S stretching
1417	1403	C=S asymmetric
1627	1644	NH_2 bending

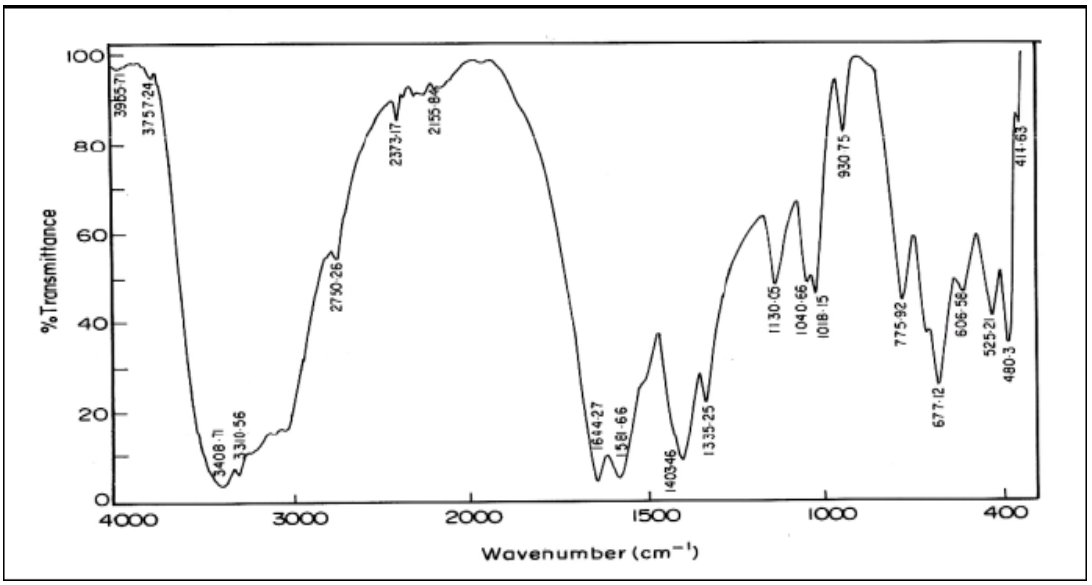

Figure: 4

4.3 Vickers hardness test:

The hardness property of crystals play key role in device fabrication.Vicker's micro hardness study has been used to analyse hardness property of grown crystals with micro hardness tester fitted with diamond pyramidal indenter attached to an incident light microscope. Loads of different magnitude was applied for fixed interval of time. Indentation time was 8 seconds for all the loads. At lower load hardness increases with load. It attains saturation at higher load. Higher the hardness number greater the stress to form dislocation. This confirms greater crystalline perfection. Meyer index number was calculated from Meyers law which relates load and indentation diagonal length as $P=Kd^n$ where n is called Meyers index no .

Meyer index no/ work hardening index=3.6

$H_v = 1.854 \times F/d^2$

Accurate readings can be taken and one type of indenter is used for all types of metals and surface treatments.

4.4 Second harmonic generation:

In this method powdered sample is irradiated with laser beam and scattered light is collected and analysed with the use of suitable filter. The second harmonic signal generated in the sample was confirmed from the emission of green radiation of λ=532nm.BTZA has better SHG efficiency than cadmium thiourea acetate (CTA).

5. CONCLUSION

BTZA crystals were grown by slow evaporation which is a very convenient method since it is possible to dissolve large amount of compound in a small volume of solvent. This technique is widely used to grow several types of crystals at room temperature. A good quality of semi organic Non linear optical crystals (NLO) OF BTZA were synthesized and grown by this technique. Solubility was estimated which dictates the growth procedure. Single crystal XRD study was carried out to confirm noncentro symmetric nature. The present work was aimed out at growing bulk sized single crystals and identifying crystal structure by XRD.The search and design of high efficient non linear optical crystals for visible and UV regions are extremely important for laser and material processing .The transparent crystals were free from cracks and hardness measurement was found out to be 108.38Kg/mm^2. SHG method was designed to find efficiency of NLO materials.

REFERENCES

[1] Fatan D.F (1991) Nonlinear optical material science vol.253 pp281-287

[2] Natamotak (1995) Raman and IR spectra of Inorganic and coordination compounds wiley inter science Newyork.

[3] W.S. Wang , K. Sutter , C.P.Z. Bosshard, H. Arend, P. Gunter, G. Chapius, F. Nicio, Jpn.J. Applied physics.., 1998,27,1138.

[4] J.D. Lee, Concise of Inorganic chemistry, Wellowish science, Germany,2004.

[5] Chiest, A; Gaetani; Nardelli, M.(1969) Abstract A.I.C.Congress,Torino,Italy,Pag.5

[6] M.D. Agarwal, J. Choi, W.S. Wang , K. Bhat, R.B.Lai, .D. Shields, B.G. Penn, D.O. Frazier, J. Crystal growth204(1999)179

[7] R. Rajasekaran, P.M. Usha Sree, R. Jayavel ,J. Crystal growth Vol-229,(2001)PP563-567.

[8] D. Rajan Babu, D. Jayaraman, R. Mohan kumar, R. Jayavel, J. Crystal growth Vol. 250(2003)

[9] Lansdise, R.A.1970" The growth of single crystal" prentice Hall, Eagelwood.

[10] S.K. Kurtz, T.T. Perry, J. Applied Physics.39 (1968)3798

[11] S. Kashino , M. Haisa ,Crystal Growth.

[12] G.P. Zhang Physics.Rev.Lett.86 (2001) 2086.