IOSR Journal of Applied Physics (IOSR-JAP)
e-ISSN: 2278-4861.Volume 7, Issue 1 Ver. I (Jan.-Feb. 2015), PP 16-20
www.iosrjournals.org

Growth and Characterisation of Bismuth Sulpho Chloride (Biscl) Single Crystals

S. Priya
(Physics, jawahar engineering college, chennai)

Abstract: *A semi organic non linear optical material Bismuth sulpho chloride was synthesised by slow evaporatio9n technique. The modes of vibration of different molecular groups were identified using FTIR analysis. Surface morphology was estimated using SEM. Micro hardness studies were carried to crystal. Kurtz powder test was taken for grown crystals to study the non linear optical effect. A powder x-ray diffraction study shows the crystalline structure of grown Biscl crystals.*

Keywords: *X-ray diffraction, low temperature solution growth, NLO material.*

I. Introduction

Non linear optical materials are material in which the intensity of light input, including its frequency is not related to intensity of light output by simple proportionality consultant. Because of this non linear behaviour an intense light beam propagating through a non linear optical material will produce new effects that cannot be seen with weak light beams. For example, an intense light beam propagating through a non linear material can generate, in addition, harmonics or overtones of original light frequency. This means that the red beam from a ruby laser can create an ultraviolet beam as it passes through the non linear optical material, while itself still propagating as a red beam. The interaction of intense light beams with non linear materials has opened a large field of potential applications in optical communication systems, where many well known radio-frequency techniques such as mixing, heterodyning and modulation can be transferred to domain of optical frequencies. There arises a need to develop semi organic non linear optical material which can combine high optical non linearity with more physical and chemical stabilities. The grown crystal was subjected to single crystal XRD study to confirm the non Centro symmetric nature for second harmonic generation. The intensity of second harmonic is monitored and displayed with a pen recorder as the temperature of the crystal is tuned through the phase matching temperature.

II. Experiment

2.1. Synthesis and growth of BISCL crystals.

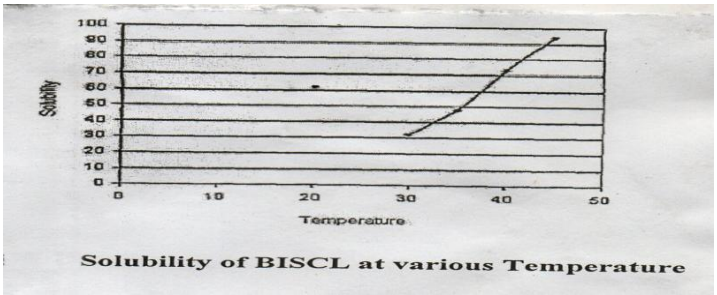
Sodium metasilicate and H_2S was taken in the stochiometric ratio 1:2 and this material was dissolved in 1 litre double distilled water and gently stirred for 5 hours using magnetic stirrer apparatus. After somedays the synthesised salt of Bismuth sulpho chloride was collected and preserved in a beaker. Thestochiometric equation is given by,

$Bi_2o_3 + 6\ Hcl = 2\ Bicl_3 + 3\ H_2O$

$2Bicl_3 + 2H_2S = 2Biscl + 4\ Hcl$.

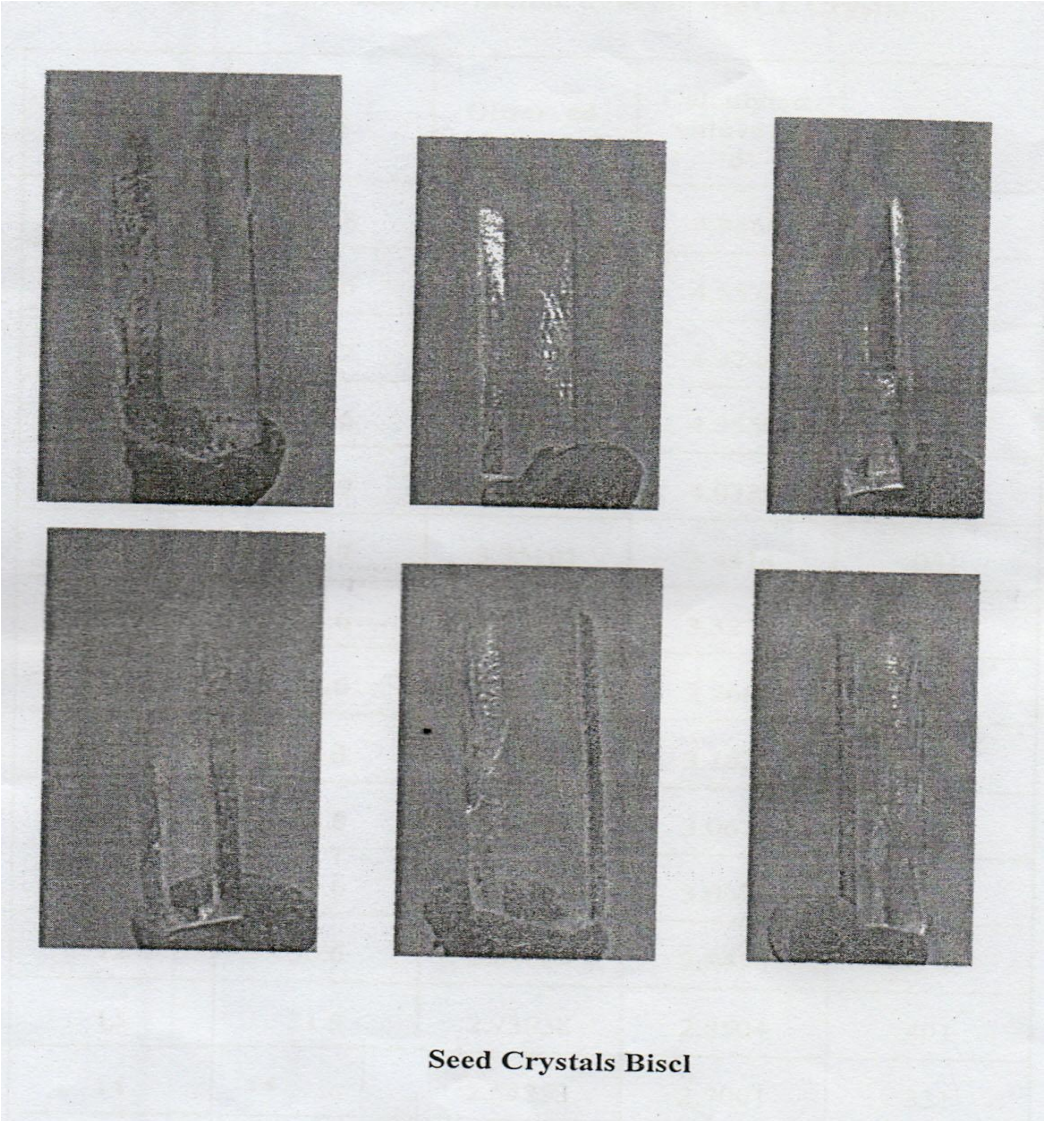
2.2. Solubility

The synthesised salt of Biscl was dissolved in 100 ml double distilled water, up to saturation level at 30˚c. Themass of the empty peltries dish is found out using digital weighing balance. Then 10ml of saturated solution is taken using pipette in peltridish and placed in an oven at around 60˚c. After complete evaporation of water in the peltridish the mass of dry salt was taken. Similarly the solubility of salt was found out using stochiometric method at different temperatures. A graph is drawn between the temperature and solubility and is shown



Solubility of BISCL at various Temperature

.



Seed Crystals Biscl

X-Ray Diffraction data for BISCL Crystal

S.No.	26	Observed values of 'd'	Calculated Values of 'd'	hkl
1	17.8	4.98285	4.9469	111
2	19.6	4.52912	4.5573	220
3	20.2	4.31146	4.3744	201
4	20.6	4.05838	4.3550	300
	21.9	3.46628	4.0335	130
6	25.7	3.35101	3.4414	031
7	26.6	3.23183	3.3279	131
8	27.6	3.15346	3.2662	400
9	28.3	3.09984	3.0676	410
10	28.8	3.01786	3.0676	321
11	29.6	2.92148	3.0382	330
12	30.6	2.84003	2.9450	002
13	31.5	2.73038	2.8504	401
14	32.8	2.54253	2.7001	331
15	35.3	2.50816	2.4734	222
16	35.8	2.39827	2.5876	430
17	37.5	2.36187	2.3959	312
18	38.1	2.16096	2.3474	511
19	41.8	2.11276	2.1146	332
20	42.8	2.04465	2.1555	412

Crystallographic information of BISCL

Formula		Biscl
Formula Weight		288.52
Crystal: Colour		uncoloured transparent
Dimension(mm)		0.17x0.22x0.29
Crystal system		orthorhombic
Space Group		Pnma
Cell Constants:	a (Å)	5.900(1)
	b (Å)	12.773(2)
	c (Å)	13.064(2)
Cell volume ($Å^3$)		984.5(4)
μ_{calc} (Cm^{-1})		34.60
d_{calc} (gcm^{-1})		1.94
Z		4
Temperature		room temperature
Diffractometer		Brucker A x S
Radiation, wavelength (Å)		MoKα , 0.71069
Reflections measure		10419
Independent fefL, R_{int}		1182, 3.034

III. Results And Discussion

3.1. X-ray diffraction.

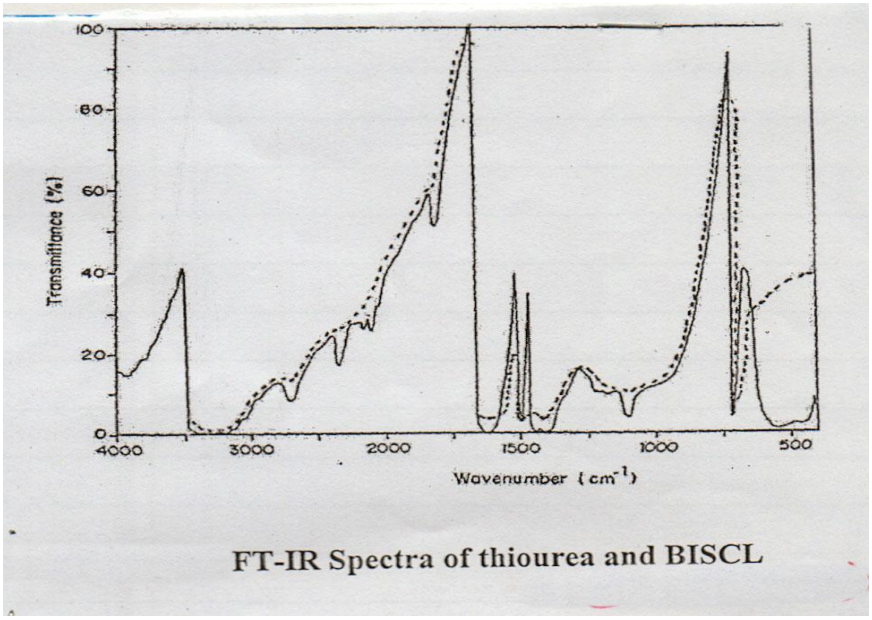
Powder x-ray diffraction has been carried out using rich seifert diffractometer. The sample was scanned over the range 10-70 degree at a rate of 1 degree per minute. Powder x-ray diffraction studies confirmedtheorthorhombic structure of grown crystals. The unit cell dimensions determined from single crystal x-ray diffraction analysis are a=13.012 A b=12.768 A c=5.890 A and V=978.64 A. Kurtz powder SHG test confirmed the non linear property of Biscl. In this technique, the sample was packed as a polycrystalline powder into a cell sandwiched between two glass slides. The sample is then subjected to Nd-YAG laser emitting 1.064m, ions laser pulses with spot radius 1mm to assess the SHG intensity.

3.2. FTIR analysis

The FTIR spectra of BISCL and thiourea are shown in which there are two possibilities. The co-ordination of Zinc may occur either through nitrogen or through sulphur of thiourea. The study of spectraof thiourea and BISCL shows a shift in low frequency region. The broad envelope positioned inbetween 2750 and 3500cm $^{-1}$ corresponds to symmetric and asymmetric stretching modes of NH_2 grouping of thiourea were not shifted to lower frequencies on the formation of Zinc thiourea complex. The absorption bands assigned to particular vibrations indicated the presence of sulphur to Bismuth bands in BISCL.

Assignment of IR band frequencies (cm^1) of thiourea and BISCL

Thiourea	BISCL	Assignments
1625	1612	NH bending
1470	1494	N-C-N stretching
1417	1404	C=S stretching
1083	1103	N-C-N stretching
730	713	C=S stretching



FT-IR Spectra of thiourea and BISCL

3.3. Kurtz Powder Test for Non linearity

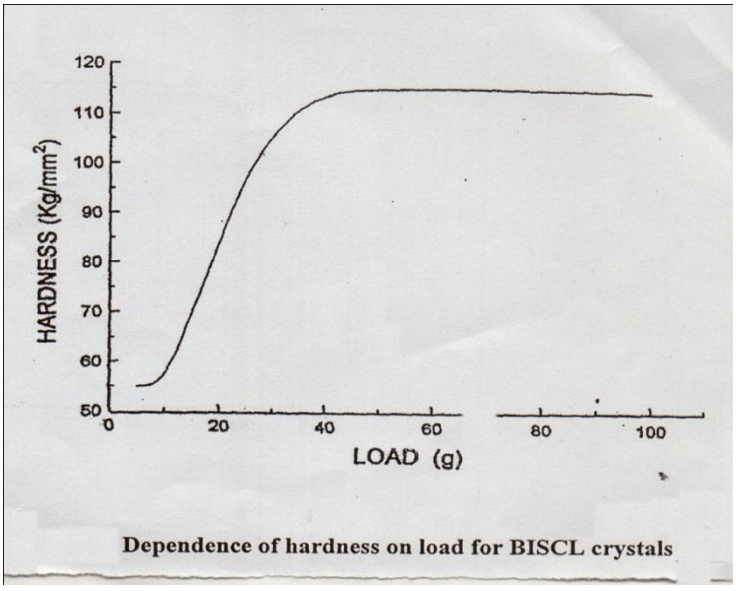
To confirm the non linearity property Kurtz powder SHG test was carried out for the sample. The crystal powder must be so finely ground that the average size of the particle is less than the coherencelength for the second harmonic generation, otherwise there would be no signal. Typical value ofcoherence length is 2µm.

3.4. Micro hardness studies

The polished (100) face of Biscl are subjected to static indention tests at room temperature using leitz Wetzlar hardness tester fitted with vicker's diamond pyramidal indenter. Vicker's micro hardness number was evaluated from the relation.

$Hv= 1.8544 (P/D^2) Kg/mm^2$.

Where Hv is Vicker's micro hardness number, P is applied load and d is diagonal length of indentation impression.



Dependence of hardness on load for BISCL crystals

IV. Conclusion

A powder x ray diffraction study shows the crystalline structure of the grown BISCL crystals. Since crystal x-ray diffraction study reveals that the BISCL crystal is orthorhombic structure/ FTIR spectroscopy confirms the Biscl crystal consists of all functional groups. Kurtz powder test shows the non linear optical property of the crystals. Vickers micro hardness study shows the mechanical strength used for device fabrication.

References

[1]. Fatan D.F.(1991) Non-linear optical material science vol.253 pp 281-287.
[2]. Nakamotok. (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. Nicoio, Jpn. J. Appl. Phys., 1998,27,1138.
[4]. J.D.Lee, Concise of organic chemistry, Wellowish Science, Germany, 2004.
[5]. Chiest,A.; Gaetani,A; Nardelli,M.(1969) Abstract A.I.C. Congress, Torino, Italy, Pag.5
[6]. M.D. Aggarwal, J. Choi, W.S. Wang, K. Bhat, R.B. Lai, A.D. Shields, B.G. Penn, D.O.Frazier,J. Crystal Growth 204 (1999) 179.
[7]. R. Rajasekaran, P.M. Usha Sree, R. Jayavel, J. Crystal Growth, Vol-229,(2001) PP 563-567.
[8]. D. Rajan Babu, D. Jayaraman, R. Mohan Kumar, G. Ra-:. R-Jayavel, J. Crystal Growth Vol.250 (2003) 157-161.
[9]. Landside, R.A.1970 "The growth of single crystal" Prentice, Hall, Eaglewood.
[10]. S.K. Kurtz, T.T. Perry, J. Appl. Phys. 39 (1968) 3798.