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MECHANICAL AND DIELECTRIC STUDIES ON BIS-THIOUREA CADMIUM CHLORIDE (BTCC) CRYSTAL BY SLOW EVAPORATION TECHNIQUE

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ABSTRACT- Bis-Thiourea Cadmium Chloride is abbreviated as (BTCC) were grown from aqueous solution by low temperature solution growth technique. Good optical quality single crystal of dimensions 12 × 7× 2.15 mm^3 was harvested in a period of two weeks. Single XRD analysis confirmed that the pure crystal is in orthorhombic structure with lattice parameters a=5.798(Å), b=6.456(Å), c=13.079(Å). FTIR spectroscopy study reveals the presence of functional groups present in the grown crystal. Microhardness studies were carried out using Vickers pyramidal indentation method at room temperature shows that the grown crystal belongs to soft material category. Dielectric studies are done on the grown BTCC crystal

Keywords- [Semi organic, Conventional slow evaporation growth technique, Dielectric Studies, NLO material]

1. INTRODUCTION

The rapid developments in the field of optoelectronics necessitates the search for new and efficient nonlinear optical (NLO) materials which can be utilized for optoelectronics applications such as optical computing, optical data storage, optical communications and electro-optic shutters. During the past decade, a number of organic NLO materials with high nonlinear susceptibilities have been synthesized .Recently number of researchers emphasize on the hunt of new semi-organic nonlinear optical materials which exhibit wide transparency region, high damage threshold and high nonlinear coefficient [1-3]. BTCC is a potential NLO candidate which has various applications in the field of laser and opto electronics. The second harmonic generation (SHG) efficiency of BTCC crystal is 0.73 times greater than urea. The intriguing feature of BTCC is that it has high laser damage threshold and it possess favourable mechanical properties which are comparable with KDP. The powder SHG efficiency of BTCC crystal is 110 times high than quartz [4-6]. In the present investigation Bisthiourea is combined with cadmium chloride to form a crystal and various characterization studies are done on the grown BTCC crystal.

2. MATERIAL SYNTHESIS

Bis-Thiourea Cadmium Chloride (BTCC) was synthesized by dissipating Thiourea and Cadmium Chloride taken in the ratio of 2:1. The reaction is given below

$$\mathbf{CdCl_2 + 2CS(NH_2)_2 \rightarrow Cd[CS(NH_2)_2]_2Cl_2} \quad (1)$$

Repeated crystallization helps to increase the purity of the synthesized salt. The supersaturated solution of BTCC was kept undisturbed at room temperature. A typical single crystal with size 12 × 7× 2.15mm^3 with in a period of three weeks. The grown crystal is shown in figure 1.

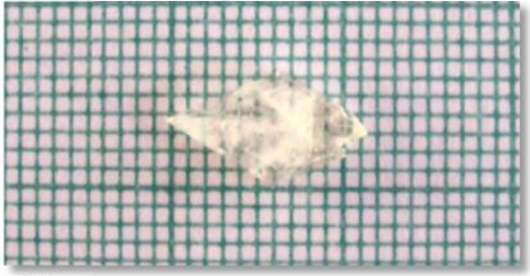

Figure 1- Photograph of Pure BTCC crystals

3. RESULTS AND DISCUSSION

3.1. Single Crystal X-ray analysis

The single crystal X-ray diffraction has been done by using ENRAF NONIUS CAD4 diffractometer with MoKα radiation (λ = 0.7170 Å) to identify the unit cell dimensions of the grown crystal. The single crystal X-ray diffraction study shows that the grown BTCC crystal is belong to orthorhombic system with space group $Pmn2_1$with lattice parameters a = 5.798Å, b = 6.456 Å, c = 13.079 Å and the Volume = 489.6 Å^3. These values agree well with the reported values [7].

3.2. FT-IR Analysis

FT-IR spectra was recorded using JAS-CO FT-IR 460 plus spectrometer using KBr pellet technique in the range 400-4000 cm^{-1} to identify the presence of functional group present in the grown crystal. The absorption bands at 3282 and 3389 cm^{-1} indicate the presence of N-H symmetric stretching and N-H asymmetric stretching respectively. The absorption band at 1614 cm^{-1} indicates the presence functional group NH_2 in the bending mode. The C=S stretching vibrations in the thiourea at 740 and 1417 cm^{-1} are shifted to 715 and 1438 cm^{-1} , respectively in BTCC which is due to the addition of metal (cadmium) ion in the complex. The C-N stretching vibrations at 1099 cm^{-1} in thiourea are shifted to the higher frequency of 1100 cm^{-1}. The band found in BTCC at 1494 cm^{-1} corresponds to the 1470 cm^{-1} band of thiourea assigned to the H-C-N vibrations.

3.3. DIELECTRIC STUDIES

The dielectric measurements were performed on bisthiourea cadmium chloride single crystals using Hioki Hitester model 3532-50 LCR meter. The dielectric constant and dielectric loss of grown BTCC crystal is shown in Fig 2 & 3 respectively. From the spectrums it is observed that the dielectric constant and dielectric loss decreases slowly with increasing frequency and attains saturation at higher frequencies. The dielectric constant of material is due to the electronic, ionic, dipolar and space charge polarization. The lower value of dielectric constant at higher frequencies is a suitable parameter for the enhancement of SHG. The characteristic of low dielectric loss with high frequency for the sample suggests that the crystal possesses enhanced optical quality with lesser defects and this parameter play a vital role for the construction of devices for non-linear optical materials. Dielectric constant of BTCC crystal is in good agreement with reported value [8]

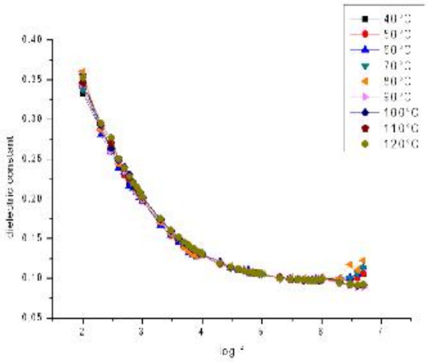

Figure 2-Dielectric Constant versus log f

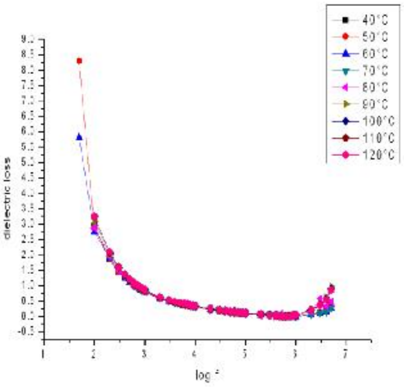

Figure 3- Dielectric Loss versus log f

3.4. Microhardness Measurements

Vicker'smicrohardnessstudy was made on the grown BTCC crystal with room temperature by varying the load range using Vicker'smicrohardness tester. The value of Hv is calculated using the following equation

$$H_v = 1.8544\ P/d^2 \qquad (2)$$

where P is the applied load in g, d is the diagonal length in mm and H_v in kg/mm^2 . The variation of Hv with the applied load of BTCC crystal is shown in Fig 5. A plot of log d vs log P is shown in Fig 6. The plot of log d Vs log P yields a straight line and the slope gives the work hardening coefficient 'n'. The value of n is 3.5 and it is more than 2 so it is soft material [9].

The Mayer's index number was calculated from Mayer's law, which relates load and indentation diagonal length. According to Meyer's relation

$$P=K_1 d^n \qquad (3)$$

Where K_1 is the standard hardness value which can be found out from the plot P versus d^n.

The material takes some time to revert to the elastic mode after every indentation a correction 'n' is applied to the d value.

Kick's law is related as $P=K_2 (d+x)^2$

$$(4)$$

From 3 and 4 equations

$$d^{n/2}=(K_2/K_1)^{1/2} d + (K_2/K_1)x \qquad (5)$$

The slope of $d^{n/2}$ versus d yield $(K_2/K_1)^{1/2}$ and the intercept is a measure of x. The fracture toughness K_c is given by

$$K_c=P/\beta c^{3/2} \qquad (6)$$

Where c is the crack length measured from the centre of the indentation mark to crack tip. P is the applied load and geometrical constant β=7 for Vickers indenter.

The yield strength (σ_v) of the material can be found out using the relation

$$\sigma_v = \frac{H_v}{2.9}\{1-(n-2)\}\left[\frac{12.5(n-2)}{1-(n-2)}\right]^{n-2} \qquad (7)$$

The elastic stiffness constant (C_{11}) was calculated using the formula [10]

Wooster emprical relation $C_{11} = (Hv)^{7/4}$ (8)

There are two factors important in determining the value of C_{11}. The first factor is tightness of bonding between the neighbouring atoms. The second factor is rate of variation with position of the atoms of the forces of attraction and repulsion between them. Stiffness constant increases with increase of load.

Parameters	**Values for BTCC**
N	3.5
K_1(kg/mm)	36.62 x 10^{-3}
K_2(kg/mm)	7.49 x 10^{-3}
H_v(kg/mm^2)	54.53
σ_v (Mρa)	1.723
C_{11} (10^{14} Pa)	19.460

Table 1- Various Parameters calculated using Microhardness study for BTCC crystal

CONCLUSION

BTCC has been grown by slow evaporation technique and have achieved good quality optical crystal. Single crystal X-ray Diffraction study reveals that the grown crystal comes under orthorhombic system with space group $Pmn2_1$. The presence of the functional group is revealed by FTIR spectroscopic study. From Dielectric study it is observed that the dielectric constant and dielectric loss decreases slowly with increasing frequency and attains saturation at higher frequencies. The load dependent hardness parameters are calculated using microhardness study

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