Available online at www.pelagiaresearchlibrary.com

Pelagia Research Library

Advances in Applied Science Research, 2013, 4(4):330-334

Pelagia Research Library

ISSN: 0976-8610
CODEN (USA): AASRFC

Structural and mechanical studies of sodium p-nitro phenolate para nitro phenol dihydrate (SPPD) single crystals

V. J. Priyadharshini[1,2] and G.Meenakshi[2]

[1]Karpagam University, Coimbatore, India
[2]Department of Physics, Manakula Vinayagar Institute of Technology, India
[3]Department of Physics, KMCPGS, Pondicherry, India

ABSTRACT

Sodium p-nitro phenolate para nitro phenol dihydrate (SPPD) is a semi organic nonlinear optical (NLO) material used for frequency doubling in IR region. It has been synthesized in room temperature. Lattice parameter and structural parameters have been determined by the single and powder X-ray diffraction method. Mechanical studies has also been studied which found that the material is a soft material and by photoconductivity studies it is confirmed that the material exhibit positive photoconductivity.

Key words: Solution growth, Single crystals, X-ray studies, Mechanical Studies.

INTRODUCTION

Nonlinear optical frequency conversion materials have significant impact on laser technology, optical communication and optical data storage. Sodium p-nitro phenolate para nitro phenol dihydrate is a semi organic NLO material possessing large values of hyperpolarizability due to the presence of an organic entity p-nitro phenol [1]. The crystal growth is found to be faster along *a* –axis. The fact is well correlated, since the cell dimension '*a*' is the shortest among others and in many examples [3-5] of well-known systems, the fastest growth is observed in the direction of the shortest axis. The crystal belongs to orthorhombic system with symmetry I*ma*2 a well – known non centrosymmetric space group, thus satisfying the requirements for second-order NLO activity.

From the microanalysis one can see that the percentage content of the anionic part of the compound is systematically higher in the case of crystals grown from aqueous solution as compared to those grown from methanol solution. This may be the reason for higher transparency of the aqueous grown crystals. The thermal stability of SPPD is more compared to the organic crystal like N-methyllutitone trihydrate[6]. Sodium paranitrophenolate dihydrate is one such semi-organic NLO crystal, characterized for its NLO property,where in the organic ligand nitrophenoxy ion is ionically bonded to the metal ion sodium [7-13].

1. Synthesis

Equimolar amount of Sodium hydroxide and p-nitro phenol were dissolved in double distilled water to prepare the aqueous solution of SPPD [14]. In deionized water, Sodium hydroxide and p-nitro phenol are reacted by the following chemical reaction to produce SPPD.

$$NO_2C_6H_4OH + NaOH + H_2O \rightarrow NO_2C_6H_4ONa + 2H_2O$$

Synthesized salt of SPPD was obtained from the solution by evaporating the solvent and collecting the precipitate formed at the bottom of the container with the solution. The supersaturated solution was prepared in accordance with the solubility. Single crystals of SPPD were grown from their aqueous solution using slow solvent evaporation technique. The solvent of the supersaturated solution was allowed to evaporate through the perforated lid of the container. Numerous tiny crystals were formed at the bottom of the container due to spontaneous nucleation.

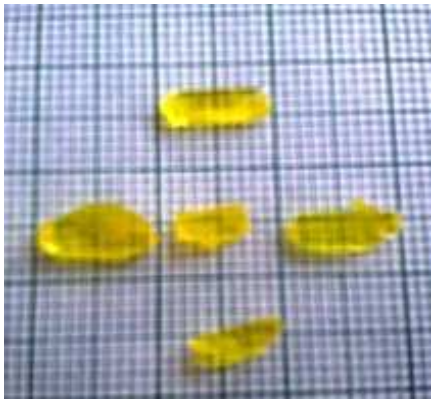

Fig:1 Grown crystal of SPPD crystals

The transparent and defect free ones among them were chosen as seeds for growing bulk crystals. By seeding the supersaturated solution and evaporating the solvent good optical quality crystals were harvested after a period of 30-40 days. The photographs of as grown crystals of SPPD are shown in Fig (1). The water grown crystals are found to possess less quality and are small in size compared to methanol grown crystals [2] .

MATERIALS AND METHODS

2. Characterization

2.1 Single Crystal XRD study

The unit cell parameters of SPPD single crystal were collected by subjecting the sample to XRD analysis using an automatic X-ray diffractometer Enraf Nonius CAD4-MV31. The X-Ray diffraction study reveals that the crystal belongs to the non-centrosymmetric monoclinic crystal system and the lattice parameters are a= 11.95 Å , b=6.95 Å, c=25.97 Å, and α=γ=90°, β= 101.47 ° which are in good agreement and belongs to the space group P21/C[15].this confirms the single crystal nature of SPPD.

2.2 Powder XRD analysis

Powder X-ray diffraction spectrum was recorded using a XPERT-PRO diffractometer with CuK α (1.5406 Å) radiation.

Table 1.1 Structural parameters

S.no	FWHM β (deg)	Particle size D (m)	Dislocation Density δ (Kg/m^3)	Strain Value ε (line $^{-2}$/m^{-4})
1	0.1828	8.313	14.644	0.0437
2	0.2306	5.8891	29.638	0.062
3	0.3337	4.535	48.98	0.0799
4	0.5424	2.8871	122.35	0.1261
5	0.9711	1.5984	391.407	0.2264

The sample was scanned over the range 2θ=70 ° at a rate of 10.13 min.The lattice parameter value has been calculated. The particle size, dislocation density and strain values were calculated in Table (1.1).It shows for corresponding FWHM value increases towards increase in dislocation density and strain value and decrease in particle size.

2.3 Vicker's hardness Studies on SPPD Single Crystal.

SPPD Single crystal was subjected to Vicker's Microhardness test with the applied load varying from 25 g to 200 g for an indentation period of 10 seconds. Vicker's Microhardness profile as a function of the applied test loads is illustrated by the Fig (2). It is Evident from the plot that the Microhardness of the crystal increases with increasing load. The increase in the Microhardness values of SPPD with increasing load is in agreement with the normal indentation size effect (ISE).

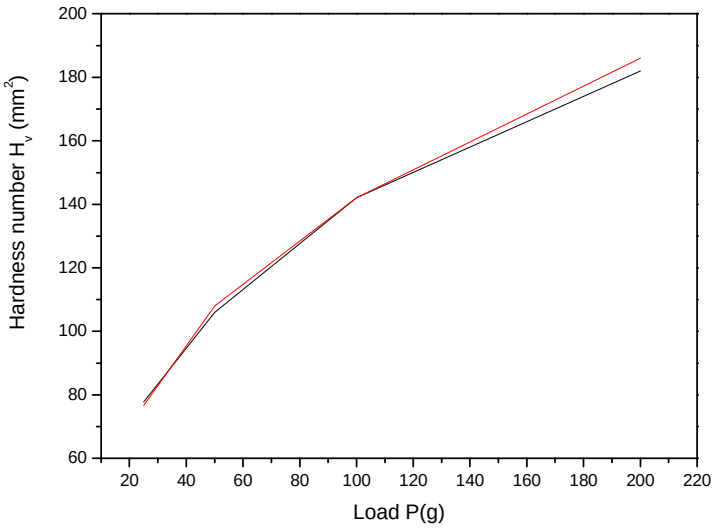

Fig: 2 Vicker's microhardness

The value of the work hardening co-efficient n was estimated from the plot of Log p versus log d drawn by the least square fit method. The hardness data of SPPD crystal is presented in the Fig (3). It is observed that the Vicker's hardness number and work hardening coefficients of the crystal increase with increasing load. According to Onistich, $1.0 \leq n \leq 1.6$ for hard materials and $n \geq 1.6$ for soft materials [16].n is found to3.60 which lie greater than 1.6 for the SPPD crystal. Hence it is concluded that SPPD belongs to soft materials.

2.4 Photoconductivity Study of SPPD Single crystals.

Experimental

The details of the experimental set-up used in the present study are reported elsewere[17-19].A polished sample of SPPD crystal was attached to a microscopic slide on which two electrodes of thin copper wire (0.14cm diameter) were fixed at a distance of 0.051 cm by using silver paint. The sample was connected in series to a d.c.power supply and a picoammeter (Keithley 480) as shown in Fig (4)

Dark current

After shielding the sample from all radiations, the applied field was increased from 0 to 3000 volt/cm and the corresponding current values shown by the Pico ammeter were noted. The current obtained in the present condition is known dark current.

Photo current

Keeping the sample in the same experimental setup, the sample was exposed to the radiation from a halogen lamp (100W) containing iodine vapour and tungsten filament by focusing a spot of light on it with the help of a convex lens. The photo current of SPPD was recorded for the range of 0-3000 applied field. Fig (4) shows the plot of dark and photo currents as a function of the applied electric field.

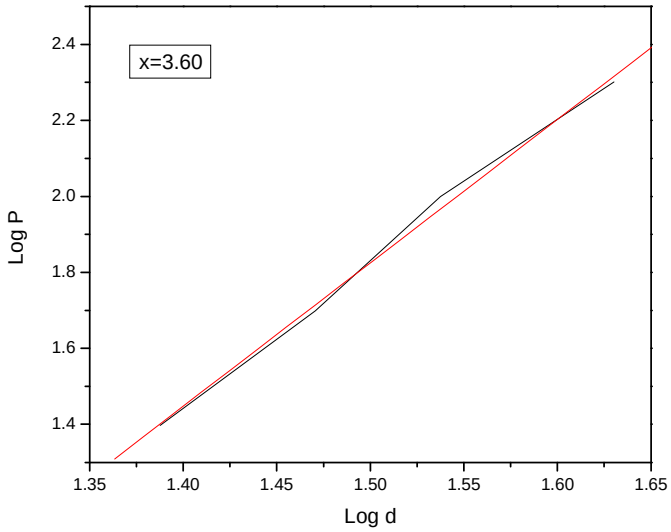

Fig: 3 Work Hardening co-efficient

It is observed from the Fig (4) that the dark current and photo current increase with increase in the applied field. The photo current is found to have greater values than the dark current. Hence, it can be concluded that SPPD single crystals exhibit s positive photoconductivity.

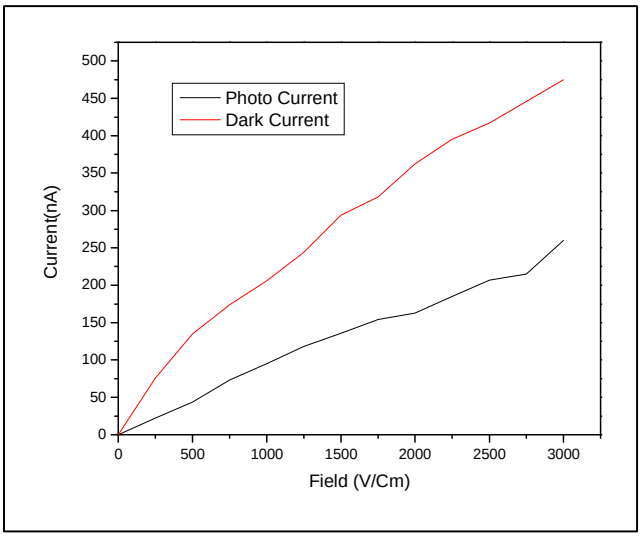

Fig:4 Photoconductivity Study

CONCLUSION

SPPD crystals have been grown by slow evaporation technique. The crystal belongs to the monoclinic system and Non Centro symmetric space group. Powder X-Ray diffraction shows for corresponding FWHM value increases towards increase in dislocation density and strain value and decrease in particle size. The work hardening co-efficient reveals that the material SPPD belongs to a soft material. The photoconductivity study states that the compound exhibit positive photoconductivity.

REFERENCES

[1] Dhanuskodi,A.PricillaJeyakumar,S.Manivannan,*SpectrochimicaActa* 66(**2007**)318-312

[2] S.Vanishri , S. Brahadeeswaran H.L.Bhat , *journal of crystal growth* 275 (**2005**) 141-146.
[3] A.S.H.Hameed,p.Anandan,R.Jayavel,P.Ramasamy,G.Ravi,*J.Cryst.Growth* 249 (**2003**) 316
[4] H.S.Nagaraja,V.Upadhaya,P.M.Rao,P.S.Aithal,A.P.Bhat,*J.Cryst.Growth* 193 (**1998**) 674
[5] P.A.Angeli Mary ,S.Dhanuskodi,*Cryst.Res.Technol*.36(**2001**) 123.
[6] S.Dhanuskodi,S.Manivannan,J.Phillip,*J.Cryst.Growth* 265 (**2004**)284-288
[7] Hisashi Minemoto, Yusuke Ozaki, Nobuo Sonoda, *J. Appl. Phys.* 63 (26) (**1993**) 3565.
[8] Hisashi Minemoto, Yusuke Ozaki, Nobuo Sonoda, *J. Appl. Phys*. 76 (7) (**1994**) 3975.
[9] S. Brahadeeswaran, V. Venkataramanan, J.N. Sherwood, H.L. Bhat, *J. Mater. Chem*. 8 (3) (**1998**) 613.
[10] M. Ben Salah, P. Becker, C. Carabatos Nedelec, *Vibr. Spectrosc.* 26 (**2001**) 23.
[11] S. Brahadeeswaran, V. Venkataramanan, H.L. Bhat, *J. Cryst. Growth* 205 (**1999**) 548.
[12] B. Milton Boaz, A. Leyo Rajesh, S. Xavier Jesu Raja, S. Jerome Das, *J. Cryst. Growth* 262 (**2004**) 531.
[13] B. Milton Boaz, A. Leyo Rajesh, S. Xavier Jesu Raja, S. Jerome Das,*J. Mater. Sci. Technol*. 20 (5) (**2004**) 505.
[14] V.J.Priyadharshini, G. Meenakshi, K.Thamizharasan, IOSR*Journal of Applied Physics* **(2012**), PP 13-16.
[15] S.Brahadeeswaran,H.L.Bhat, *Acta. Crystallogr.C* 46 (**1990**) 467.
[16] R.Uthrakumar, C. Vesta, M. Jose, K. Sugandhi, S. Krishnan, S. Jerome Das, *Physica B* 405 (**2010**) 3371-3375
[17] M.Jose,R.Uthrakumar,A.Jeya Rajendran, S.Jerome Das, *Spectrochimica Acta Part A* 86 (**2012**) 495-499.
[18] Mukerji s., and Kar T.(**1999**) *Cryst.Res.Technol.*, Vol.34,pp.1323-1328
[19] K.Vasantha, S.Dhanuskodi,*J.Crysat.Growth* 269 (**2004**) 333