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K Arunkumar, S Kalainathan. Growth and Dielectric Properties of 1,3-bis(4-methoxyphenyl)prop-2-en-1-one Organic Single Crystal . Mechanics, Materials Science & Engineering MMSE Journal. Open Access, 2017, 9, 10.2412/mmse.12.10.956 . hal-01503804

HAL Id: hal-01503804

<https://hal.science/hal-01503804>

Submitted on 7 Apr 2017

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Growth and Dielectric Properties of 1,3-bis(4-methoxyphenyl)prop-2-en-1-one Organic Single Crystal

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DOI 10.2412/mmse.12.10.956 provided by Seo4U.link

Keywords: crystal growth, organic material, dielectric properties, powder XRD.

ABSTRACT. High-quality single crystal of 1,3-bis(4-methoxyphenyl)prop-2-en-1-one (BMP) has been grown by the vertical Bridgman technique. Calculated the grain size of BMP single crystal from powder XRD. ¹H NMR spectrum was recorded to confirm the presence of protons in the synthesized compound. The dielectric constant and dielectric loss of the grown crystal was studied as a function of frequency with different temperatures and the results are discussed.

Introduction. In the past few years, optoelectronic materials have prompted researchers to grow large size of organic single crystals for advanced high technology devices. NLO has emerging as one of the most attractive field of research in view of vital applications such as high speed information processing, optical data storage, optical communications and frequency conversion [1, 2]. The high dipole moment generally leads to the molecules packing in centrosymmetric arrangements and SHG responses in these systems become zero. Therefore, one requires a highly hyperpolarizable molecule aligned in a head-to-tail fashion, connected through strong hydrogen bond interactions, crystallizing in noncentrosymmetric structures and produce high second harmonic generation efficiency. Chalcones are interesting organic NLO materials that show good crystallizability, high SHG conversion efficiency compared to urea [3, 4]. In recent years, researchers focused on the growth of high quality organic single crystals. The organic single crystals can be grown by different techniques. In certain organic materials, the solution growth techniques such as slow cooling and slow evaporation take more time to crystallize and difficult to grow large size crystals and sometimes the solvent inclusion can reduce the optical property of the crystal [5]. The melt growth technique is one of the techniques widely used to grow organic and inorganic single crystals. Bridgman technique is the simplest and best technique for the growth of large size good quality transparent crystals from melt in the stipulated period [6]. This paper, deals with the growth of 1,3-bis(4-methoxyphenyl)prop-2-en-1-one (BMP) by two zone vertical Bridgman technique and its rest of the characterizations ¹H NMR and Dielectric properties.

Experiment. In the present experiment, the synthesized and purified raw material of BMP was charged into the single wall conical shaped borosil glass ampoule [4]. In the ampoule cone length was increases the crystal quality increases [7], and then ampoule admitted to high vacuum (10⁻⁶ Torr) and carefully sealed off. The upper zone temperature was maintained at 3°C above the melting point [4]. The furnace temperature profile is shown in Fig. 1a. The ampoule was allowed to stand for 8 h in upper zone to maintain thermal equilibrium and to avoid bubble formation of during crystal growth. The translation of the melted substance from upper zone to the lower zone very slowly. Because the organic materials have low thermal conductivity takes more time to solidify from the melt [5]. In the present study, we tried three translations rate (1.0, 0.5 and 0.3mm). First two translation were occurred

multi- nucleation finally we optimized the third translation rate was 0.3mm/ hr got the good transparent BMP single crystal. After growth has been completed, the heating profile was reduced step by step to room temperature at a rate of 1 to 3°C/hr. In order to avoid cracks due to the difference in the thermal expansion coefficient between the borosil glass and crystal. The grown crystal was removed from the single wall ampoule carefully by using the standard diamond wheel cutter. The good transparent BMP single crystal with, cut and polished crystal is shown in Fig.1b.

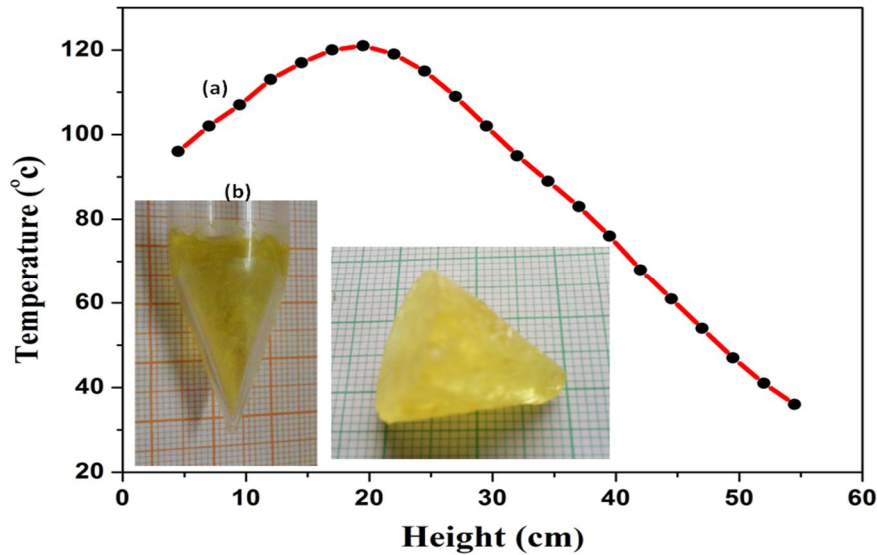


Fig. 1. (a) Temperature profile, (b) grown BMP single crystal.

Result and discussion. From single crystal X-ray diffraction analysis the lattice parameter were determined. The crystal belongs to be orthorhombic crystal system and the unit cell was determined as $a = 5.332\text{\AA}$, $b = 8.730\text{\AA}$, $c = 30.931\text{\AA}$ which is in good accordance with that of the reported value [4]. The indexed powder X- ray diffraction pattern of the BMP single crystal is shown in Fig. 2.

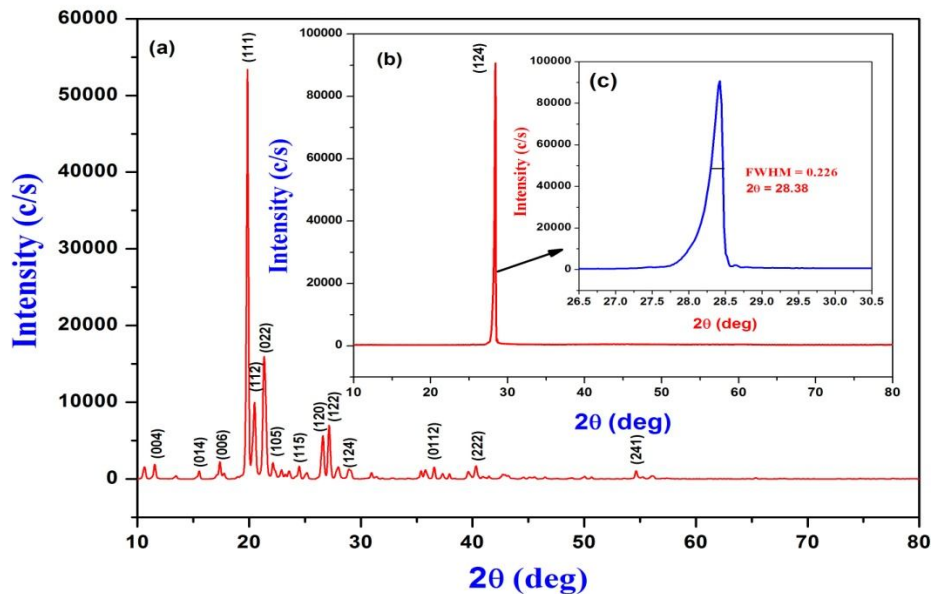


Fig. 2. (a) Powder X- ray diffraction (b) BMP single crystal and (C) Expand the single crystal peak.

3.1 Determination of Grain size from Powder XRD spectra. From the powder XRD pattern, it is observed that, the grain size is determined by measuring the width of the line with highest intensity peak in crystal using powder X software.

From the powder XRD pattern, it is observed that, the grain size is determined by measuring the width of the line with highest intensity peak in crystal using powder X software. The grain size can be calculated by using the formula,

$$D = \frac{0.9 \lambda}{\beta \cos\theta} \quad (1)$$

Where, β is full width of half maxima (FWHM) and D is grain size of the crystal.

$$D = \frac{0.9 \times 1.54056}{0.226 \times \cos(28.38)} = 0.6973 \text{ nm} \quad (2)$$

NMR spectra. The ^1H nuclear magnetic resonance spectrum is display in Fig. 3. The peaks absorbed at $\delta= 6.914, 6.934, 6.958$ and 6.978ppm are assigned to chalcone protons. The doublets at $\delta= 7.752, 8.016$ ppm ascribed to p-substituted phenyl ring next to the carbonyl group. The protons present in the methoxy substituted aromatic ring shows the doublets at 7.402 and 7.578ppm . The singlet peaks absorbed at 3.838ppm and 3.871ppm indicates the presence of hydrogen atoms in the $-\text{OCH}_3$ group. Thus the ^1H NMR spectral study confirm the functional groups present in the target compound BMP and hence the molecular structure.

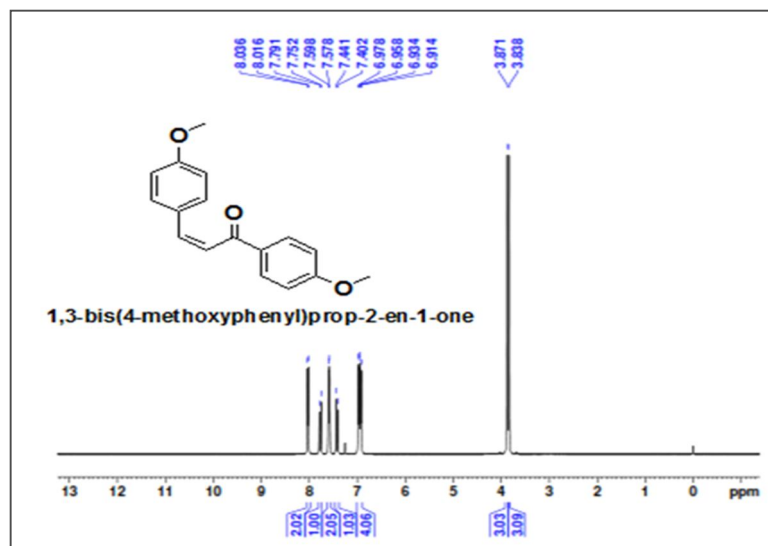


Fig. 3. ^1H NMR spectrum of BMP.

Dielectric study. The dielectric constant and dielectric loss of the grown crystal were measured at different temperatures using HIOKI 3532-50 LCR HITESTER. The crystals particularly when they are non conducting materials, therefore applied the high grade Silver paste over the sample to make uniform electrical contact with the surface of electrodes. The dielectric constant of BMP was calculated using the following relation.

$$\epsilon_r = \frac{Ct}{\epsilon_0 A}, \tag{3}$$

where ϵ_r is the dielectric constant, C is the capacitance of the medium, t is the thickness of the sample, ϵ_0 is the absolute permittivity in free space (8.854×10^{-12} F/M), and A is the area of the sample.

Fig.4 shows the plot of dielectric constant (ϵ_r) as a function of frequency. It is found that the dielectric constant decreases with increase in frequency for all temperatures. The high values of dielectric constant at low frequency enumerates the contribution from all four sources of polarizations are active namely electronic, ionic, atomic and space charge developed in the material due to the electric field variations. The high dielectric constant at low frequency observed for BMP crystal is due to the presence of space charge polarization arising at the grain boundary interfaces [8, 9]. The low values of dielectric constant at large frequencies revealed the good optical quality of the grown crystal with less defects, which is the desirable property of the materials to be used for various optical and communication devices. Fig. 2 shows that dielectric loss against log frequency. The low value of the dielectric loss reveals that the very high purity of the title compound. Suppose the dielectric loss value is large may be attributed to the space charge. The grown crystal low values of dielectric constant and dielectric loss with large frequencies, suggests that the sample possesses good optical quality with lesser defects [10]. Moreover this type of materials needs the microelectronics industry replacement of dielectric materials in multilevel interconnected structures with new low dielectric constant materials [11].

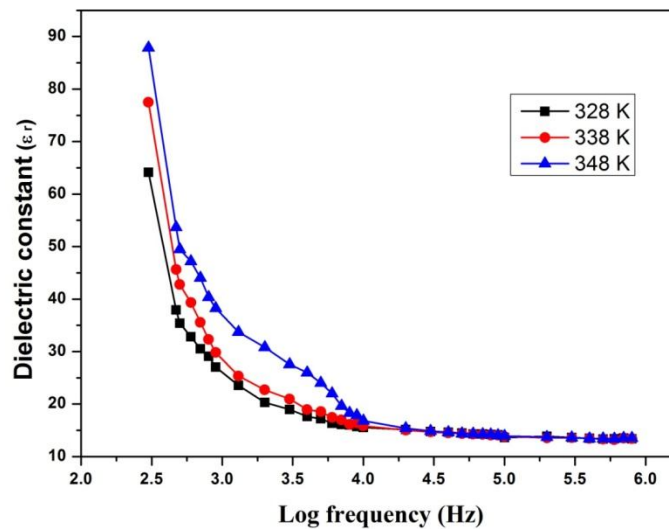


Fig. 4. Plot of dielectric constant versus applied frequency.

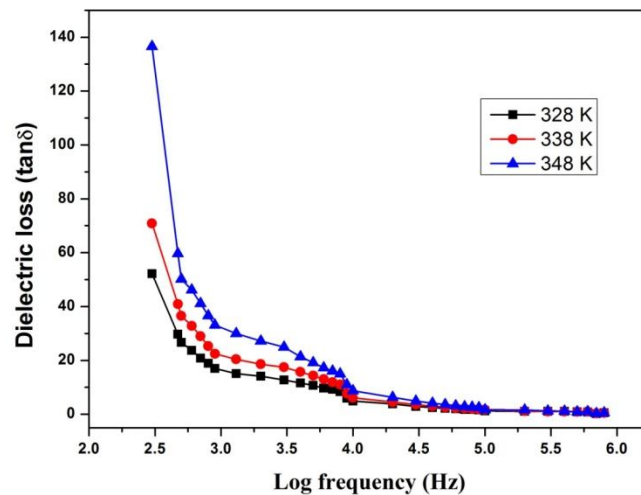


Fig. 5. Dielectric loss as a function of frequency.

Summary. It has been concluded that BMP single crystal can be grown by two zone vertical Bridgman technique. Calculated the grain size of maximum intensity peak value is 0.6973 nm. Low values of dielectric constant and dielectric loss with high frequencies, suggests that the sample possesses good optical quality and microelectronics industry replacement of low dielectric constant materials.

References

- [1] Nirmala, L. R., & Prakash, J. T. J. (2013). Investigation on the influence of foreign metal ions in crystal growth and characterization of L-Alaninium Maleate (LAM) single crystals. *Spectrochimica Acta Part A: Molecular and Biomolecular Spectroscopy*, 115, 778-782.10.1016/j.saa.2013.06.100
- [2] Lenin, M., & Ramasamy, P. (2008). Synthesis, growth and characterization of 3-nitroacetanilide—A new organic nonlinear optical crystal by Bridgman technique. *Journal of Crystal Growth*, 310(20), 4451-4455.10.1016/j.jcrysgr.2008.04.055
- [3] D'silva, E. D., Rao, D. N., Philip, R., Butcher, R. J., & Dharmaparakash, S. M. (2011). Synthesis, growth and characterization of novel second harmonic nonlinear chalcone crystal. *Journal of Physics and Chemistry of Solids*, 72(6), 824-830.10.1016/j.jpcs.2011.04.003.
- [4] Ravindra, H. J., Harrison, W. T. A., Kumar, M. S., & Dharmaparakash, S. M. (2009). Synthesis, crystal growth, characterization and structure–NLO property relationship in 1, 3-bis (4-methoxyphenyl) prop-2-en-1-one single crystal. *Journal of Crystal Growth*, 311(2), 310-315.10.1016/j.jcrysgr.2014.04.026 0022-0248/& 2014
- [5] Suthan, T., & Rajesh, N. P. (2010). Growth and characterization of organic material 4-nitrobenzaldehyde single crystal using modified vertical Bridgman technique. *Journal of Crystal Growth*, 312(21), 3156-3160.10.1016/j.jcrysgr.2010.08.002.
- [6] Babu, G. A., & Ramasamy, P. (2010). Growth and characterization of organic molecular single crystal ethyl p-amino benzoate by selective self seeding from vertical Bridgman technique. *Journal of Crystal Growth*, 312(16), 2423-2426.10.1016/j.jcrysgr.2010.05.012
- [7] Arivazhagan, T., & Rajesh, N. P. (2014). Investigation on the growth and characterization of nonlinear optical single crystal 4, 4'-dimethoxybenzoin by vertical Bridgman technique. *Optics & Laser Technology*, 64, 156-161.10.1016/j.optlastec.2014.05.014
- [8] Vijayan, N., Bhagavannarayana, G., Budakoti, G. C., Kumar, B., Upadhyaya, V., & Das, S. (2008). Optical, dielectric and surface studies on solution grown benzimidazole single crystals. *Materials Letters*, 62(8), 1252-1254.10.1016/j.matlet.2007.08.023

- [9] Sowmya, N. S., Sampathkrishnan, S., Sudhahar, S., Kumar, M. K., & Kumar, R. M. (2016). Synthesis, growth, structural, optical, thermal, dielectric and mechanical studies of piperidinium p-nitrophenolate single crystals. *Optik-International Journal for Light and Electron Optics*, 127(5), 3024-3029. 10.1016/j.ijleo.2015.12.065
- [10] Yadav, H., Sinha, N., & Kumar, B. (2015). Growth and characterization of new semiorganic nonlinear optical and piezoelectric lithium sulfate monohydrate oxalate single crystals. *Materials Research Bulletin*, 64, 194-199.10.1021/acs.cgd.5b00792
- [11] Suthan, T., Dhanaraj, P. V., Rajesh, N. P., Mahadevan, C. K., & Bhagavannarayana, G. (2011). Growth and characterization of benzil single crystals using nanotranslation by the modified vertical Bridgman technique. *CrystEngComm*, 13(12), 4018-4024.10.1039/c0ce00453g

Cite the paper

K. Arunkumar, S. Kalainathan, (2017). [Growth and Dielectric Properties of 1,3-bis\(4-methoxyphenyl\)prop-2-en-1-one Organic Single Crystal](#). *Mechanics, Materials Science & Engineering*, Vol 9. Doi [10.2412/mmse.12.10.956](#)