Optics and Laser Technology xxx (2017) xxx–xxx

Contents lists available at ScienceDirect

Optics and Laser Technology

journal homepage: www.elsevier.com/locate/optlastec

Full length article

Structural, mechanical, electrical and optical properties of a new lithium boro phthalate NLO crystal synthesized by a slow evaporation method

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ARTICLE INFO

Article history:
Received 1 January 2017
Accepted 5 June 2017
Available online xxxx

Keywords:
X-ray diffractions
Dielectric
Microhardness
Nonlinear optical materials

ABSTRACT

A new non-linear optical (NLO) single crystal of lithium boro phthalate (LiBP) was grown by slow solvent evaporation technique. The powder sample was subjected to powder X-ray diffraction (PXRD) to find its crystalline nature and the crystal structure of the grown crystal was determined using single crystal X-ray (SXRD) diffraction analysis. The Fourier Transform Infrared (FTIR) spectrum was recorded for grown crystal to identify the various functional groups present in the compound. The mechanical property of the LiBP single crystal was studied using Vickers microhardness tester. The dielectric constant and dielectric loss measurements were carried out for the grown crystal at various temperatures. The grown crystal was subjected to UV–Visible Spectral Studies to analyze the linear optical behavior of the grown crystal. The Kurtz-Perry Powder technique was employed to measure the Second Harmonic Generation efficiency of the grown crystal.

1. Introduction

In the resent years researcher's focusing on nonlinear optical (NLO) crystals. Because it's a potential material for laser oriented application [1], electro-optic switches, frequency conversion [2], color display and photonics included optical information processing [3–6]. Organic crystals have potential applications of semiconductors, superconductors and nonlinear optical devices [7]. Organic crystals have been high NLO response comparing the inorganic materials due to the presence of active π-bonds. Organic nonlinear crystals with large conversion efficiency for a second harmonic generation (SHG) and large transparency window in UV–vis region are required for device applications particularly in optical telecommunications and optical storage device [8]. In the presently researcher's searching in a novel nonlinear optical materials. At the same time semi-organic novel materials have interesting NLO properties [9–11]. In semi-organic crystals have polarizable organic molecules are bond with an in organic host. So semi-organic crystal have been large optical nonlinearity and a high degree of design chemical flexibility of NLO effects of organic with the physically strong and thermal properties of inorganic materials.

Phthalic acid is a potential material for NLO and electro-optic process. Potassium acid phthalate is an important NLO crystal in the phthalic acid family [12–14].

In the present studies synthesis, crystal growth and characterization of the semi-organic crystal lithium boro phthalate (LiBP) has been reported.

2. Experimental procedure

2.1. Material synthesis

Lithium carbonate, Phthalic acid and boric acid purchased in analytical grade reagents (AR) were taken in the 1:1:1equal molar ratio. The calculated amount of materials was taken too dissolved in deionized (18.2 MΩ/cm) water at constant stirring in room temperature.

Lithium boro phthalate (LiBP) was synthesized according to below chemical reaction. The calculated amount of salts added up to saturation stage in 250 ml deionized water at room temperature. The saturation solution was filtered using a filter paper; the filtered solution was transferred in Petri dish. The solution was left dry at room temperature by slow solvent evaporation technique. The synthesized compound purity was improved by repeated recrystallization process. The LiBP crystal was grown period 20–30 days. The optimized crystal growth conditions are shown in Table 1 and LiBP crystal shown in Fig 1.

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http://dx.doi.org/10.1016/j.optlastec.2017.06.001
0030-3992/

Please cite this article in press as: K. Mohanraj et al., Structural, mechanical, electrical and optical properties of a new lithium boro phthalate NLO crystal synthesized by a slow evaporation method, Opt. Laser Technol. (2017), http://dx.doi.org/10.1016/j.optlastec.2017.06.001

Table 1
Optimized growth conditions of LiBP crystal.

Technique	Slow solvent evaporation technique
Solvent	Double distilled water
Lithium carbonate + phthalic acid + Boric acid	1:1:1 Molar ratio
Temperature	Room temperature
Period of growth	20–30 days

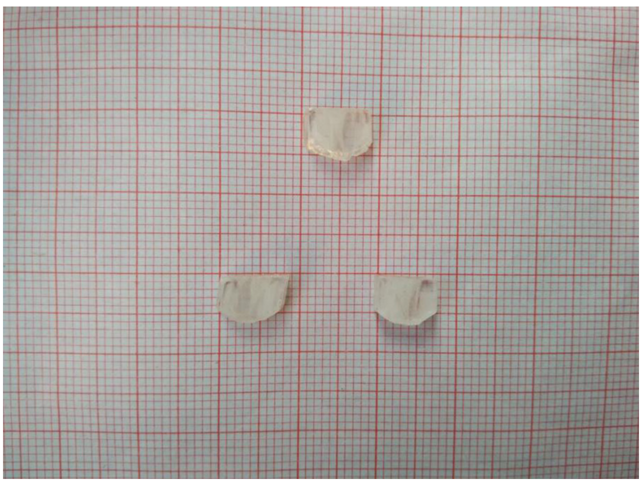


Fig. 1. As grown crystals of LiBP.

$$\text{Phthalic acid} + \text{Lithium carbonate} + \text{boric acid} \overset{\text{at room temp}}{\longrightarrow} \text{lithium boro phthalate}$$

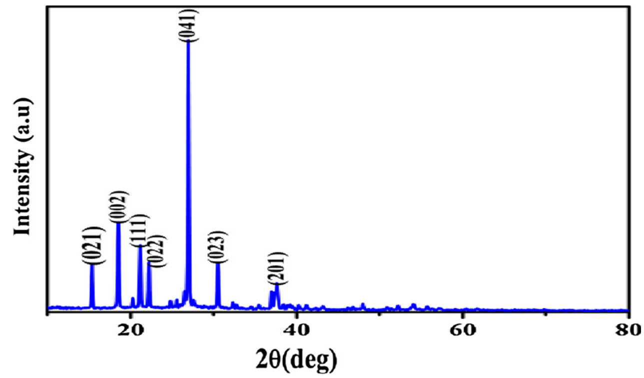


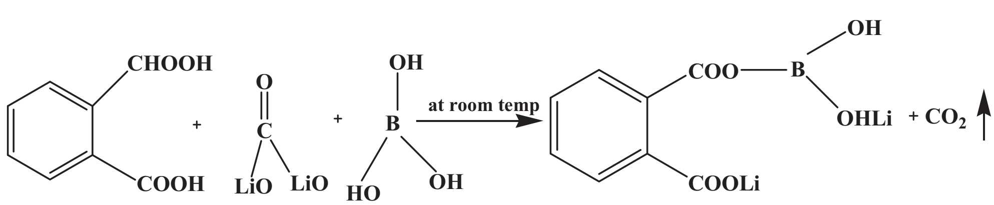
Fig. 2. Powder X-ray diffraction pattern of LiBP crystal.

3.1. Powder X-ray diffraction analysis (PXRD)

Rich Seifert diffractometer with Cu Kα (λ = 1.5418 Å) radiation technique was used to record the powder X-ray diffraction pattern of LiBP crystal. The LiBP powder sample was crushed to a fine powder and subjected to the scan over the range of 10–80° at a scan rate 2°/min. Fig. 2 shows that plot of Intensity (a.u) vs 2θ(deg). The sharp and intense peaks confirmed the good crystalline property of grown LiPB crystal. The peaks are indexed using the program DICVO91 used to index the *hkl* value.

3.2. Single crystal X-ray diffraction analysis (SXRD)

The grown LiBP single crystal was cut to suitable size and mounted on the X-ray goniometer of ENRAF NONIUS CAD-4 single crystal X-ray diffractometer with Cu Kα radiation (λ = 1.5418 Å). The crystal was optically centered at the sphere of confection and twenty-five reflections were collected from different zones of the reciprocal lattice using random search procedure. The parameters obtained are tabulated in Table 2 and LiBP crystal is found to be monoclinic structure with space group C2, which belongs to non- centro symmetric.



Scheme for the synthesis of LiBP

3. Characterization

Single crystal X-ray diffraction studies was reported crystal structure and lattice parameter using ENRAF NONIUS CAD-4 X-ray diffractometer with Mo Kα (λ = 0.1770 Å) radiation. The powder X-ray diffraction technique reported crystalline nature of material using SIEFRT X-ray diffractometer with Cu Kα (λ = 1.5406 Å) radiation. Kurtz powder technique was used to studied SHG efficiency. The FTIR spectrum was recorded in the range 400–4000 cm^{-1} using a Perkin-Elmer spectrometer by KBr pellet technique to analyzed the incorporated of boron into LiBP NLO crystal. The Lambda-35 UV–vis-NIR spectrometer used to record a linear optical property of grown LiBP NLO crystal in the range 200-1100 nm. The Leitz-Wetzlar micro hardness tester used to study the micro hardness property of grown LiBP NLO crystal. The HIOCKI 3532-50 LCR HITESTER used to investigated the dielectric property of grown LiBP crystal.

3.3. Linear optical analysis (UV–Visible)

The linear optical spectra are very important to study the optical character of any optical materials. The good quality single crystal of LiBP of 1.89 mm thickness was subjected linear optical analysis using LAMBDA-35 UV–vis spectrometer. The optical absorption spectrum was recorded in the range 200–900 nm and it is shown in Fig. 3. The spectrum gives two peaks, one intense peak at 275 nm corresponding to π–π^* transition and another peak at 224 nm corresponds to n–π^* transition (Fig. 5.3) [15]. Hence LiBP

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