Optik 127 (2016) 5613–5621

Contents lists available at ScienceDirect

Optik

journal homepage: www.elsevier.de/ijleo

Optical and electrical studies of non-linear optical crystal: Potassium boro-oxalate

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ARTICLE INFO

Article history:
Received 9 August 2014
Received in revised form
15 November 2015
Accepted 29 March 2016

Keywords:
Single X-ray diffraction
UV–vis-NIR spectrophotometer
NLO
SHG
Dielectric constant and dielectric loss

ABSTRACT

The optically transparent and bulk single crystal of potassium boro-oxalate (KBO) was grown by slow evaporation technique. Single crystal X-ray diffraction analysis revealed the crystal system and determined lattice parameters. The optical properties of the KBO crystal were determined using UV–vis spectroscopy. Optical constants such as the refractive index, the extinction coefficient, electric susceptibility and optical conductivity were determined from UV–vis spectroscopy. The refractive index of the grown crystal was determined using the Brewster angle method. The presence of various functional groups was verified from the FTIR spectrum. The dielectric constant and the dielectric loss were measured as a function of different frequencies and temperatures. The AC electrical conductivity study revealed that the conduction depended both on the frequency and the temperature. The electrical properties such as plasma energy, Penn gap, Fermi energy and polarizability were calculated to analyze second harmonic generation (SHG). Second harmonic generation (SHG) of KBO crystal was investigated by Kurtz powder technique.

1. Introduction

In recent years, owing to a number of practical applications in the field of micro-electronics and optoelectronics, a great deal of attention has been paid to the study of the properties of various materials. Recent years have witnessed a great demand for the organic materials for non-linear optical (NLO) applications because of their usage in devices such as second harmonic generators, electro-optic modulators, frequency conversion, etc. The NLO phenomena take place when the optical properties of molecules change in the presence of strong external electric fields, i.e., high energy laser beams. Most organic NLO crystals have usually poor mechanical and thermal properties and are susceptible for damage during processing even though they have large non-linear optical efficiency. Also it is difficult to grow larger size optical-quality crystals of these materials for device applications. Purely inorganic NLO materials have excellent mechanical and thermal properties, but possess relatively modest optical non-linearity because of the lack of extended π-electron delocalization. Hence it may be useful to prepare semiorganic crystals which combine the positive aspects of organic and inorganic materials, resulting in useful non-linear optical properties. Photonic crystals are expected to play an important role in the development of new optical devices [1,2]. The device possibilities for photonic crystals will be greatly enhanced by the addition of optical non-linearity; such possibilities include ultra fast optical switching for communication, and potentially even optical computing [3–9]. Non-linear optics (NLO) have been an active field of research since the late 1960's with the advent of lasers followed by the demonstration of harmonic generation in quartz [10]. Recent

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http://dx.doi.org/10.1016/j.ijleo.2016.03.067
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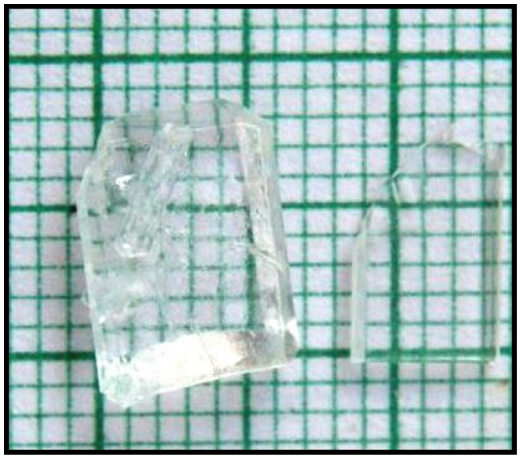

Fig. 1. Photograph of KBO crystal.

researchers have focused their attention on developing new semi organic non-linear optical (NLO) materials, as they have the advantages of both organic and inorganic materials like high resistance to laser-induced damage, wide transparency range, low angular sensitivity, high non-linearity and good mechanical hardness which make them suitable for device fabrication [11–14]. The extensive search for new types of borate crystals has led to the discovery of many excellent materials such as lithium bis-L malato borate, potassium boromalate, and potassium borosuccinate. In this sequence, a new semiorganic borate family crystal doped with inorganic salt, namely potassium boro-oxalate has been developed [15]. In the present investigation, the growth aspects of the semi-organic potassium boro-oxalate (KBO) crystals were developed by slow evaporation technique. The lattice parameters were found by single crystal XRD studies. UV–vis studies and FTIR studies revealed the absorption range and functional groups for the given material. The refractive index was determined using Brewster's angle method. Second harmonic generation (SHG) of KBO crystal was investigated by Kurtz powder technique. The dielectric constant and the dielectric loss studies were carried out for different temperatures and frequencies.

2. Experimental procedure

The bulk crystal of potassium boro-oxalate (KBO) was grown by slow evaporation technique. The evaporation method has the advantage that the crystals grow at a fixed temperature. This method can be effectively used for materials having moderate temperature coefficient of solubility. The evaporation of solvent from the surface of the solution produces high local supersaturation and formation of unwanted nuclei. Small crystals are also formed on the walls of the vessel near the surface of the liquid from the material left after evaporation. These tiny crystals fall into the solution and hinder the growth of the crystal. Potassium boro-oxalate (KBO) was synthesized by dissolving potassium hydroxide, boric acid, and oxalic acid in equimolar ratio (1:1:1) in double distilled water. The solution was stirred continuously using a magnetic stirrer. The obtained saturated solution was further purified and allowed to evaporate at higher temperature. The synthesized material was purified by repeated recrystallization process. Tiny seed crystals with good transparency were obtained due to spontaneous nucleation. Among them, a defect free seed crystal was selected and suspended in the mother solution, which was allowed to evaporate at room temperature. Large size single crystals were obtained due to the collection of monomers at the seed crystal sites from the mother solution, after the nucleation and growth processes were completed. KBO crystal of dimension about $11 \times 8 \times 5\,mm^3$ was harvested in a growth period of twenty four days by slow evaporation of the solvent. The photograph of the grown KBO crystal is shown in Fig. 1.

3. Results and discussion

3.1. Single-crystal X-ray diffraction

Single crystal X-ray diffraction analysis for the grown crystals was carried out to identify the cell parameters using an ENRAF NONIUS CAD 4 automatic X-ray diffractometer. Single crystal X-ray diffraction analysis was carried out to determine the lattice parameters. The results of the XRD analysis revealed that the grown crystals had orthorhombic structure with $P2_12_12_1$ space group and the lattice parameter values of the KBO crystals being $a = 3.726$ Å, $b = 9.503$ Å, $c = 17.772$ Å and $\alpha = \beta = \gamma = 90^\circ$. These values were found to be in close agreement with the reported values [15].