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A study on the properties of potassium pentaborate crystals

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ABSTRACT

Potassium pentaborate (KB5) single crystals were grown by slow evaporation solution growth method and the properties of the grown crystals were investigated. Single crystal and powder X-ray diffraction studies were carried out to determine the crystal structure and lattice parameters of the grown crystals. The optical transmittance of the crystal was ascertained by recording UV–vis–NIR spectrum. Vibrational modes were assigned using FT-IR and FT-Raman spectra. Vickers hardness test estimates the mechanical properties for various loads. Thermal properties were investigated using TGA/DTA and DSC analysis. Second Harmonic Generation (SHG) was confirmed by Kurtz–Perry technique. Third order nonlinear optical properties nonlinear absorption coefficient, nonlinear refractive index and third order nonlinear susceptibility was calculated by *Z*-scan method using 632.8 nm He–Ne laser.

1. Introduction

In the past few decades, there has been increasing demand for materials for technological applications. Nonlinear optical (NLO) materials are having a significant impact on laser technology. The application of NLO crystals is required in all frequency ranges but their demand in producing laser beams in UV and visible regions is growing enormously. Borate crystals are reliable materials for effective nonlinear process [1]. They are employed for frequency conversion and used as self-frequency doubling active laser sources.

KB5 is an important inorganic NLO crystal and it is successfully used for frequency conversion of laser radiation to the UV region [2]. In this paper we report the crystal growth, structural properties, optical transmittance, FT-IR and FT-Raman, mechanical, thermal, SHG and third-order nonlinear optical properties of the KB5 crystals.

2. Experimental

KB5 single crystals were synthesized by following stoichiometric incorporation of potassium carbonate and boric acid in 1:10 ratio [3,4].

$$K_2CO_3 + 10H_3BO_3 \rightarrow 2[K(H_4B_5O_{10})\cdot 2H_2O] + 7H_2O + CO_{2\uparrow}$$

Single crystals of KB5 were grown by slow evaporation solution growth method using distilled water as solvent. The solution was housed in a constant temperature bath at 32 °C. Good quality single crystals were obtained in 50–60 days.

3. Results and discussion

3.1. Single crystal X-ray diffraction

The single crystal X-ray diffraction analysis of KB5 was carried out using BRUKER NONIUS CAD4 single crystal X-ray diffractometer which confirms that crystal belongs to orthorhombic crystal system and the cell parameters are $a = 9.09$ Å, $b = 11.20$ Å, $c = 11.10$ Å and Volume, $V = 1130$ Å^3. The lattice parameter values of the KB5 crystals are in agreement with the reported values [5].

3.2. Powder X-ray diffraction

Powder X-ray diffraction studies of pure KB5 crystals were carried out using pan analytical XPERT-PRO X-ray diffractometer with Cu $K\alpha$ ($\lambda = 1.54060$ Å) radiation. The samples were scanned for 2θ values from 10.0231° to 80.9231° with step size 0.05°/s for10.1600 s. Powder XRD pattern of the KB5 crystals is shown in Fig. 1. By using the lattice parameters values taken from single crystal XRD, powder XRD diffraction patterns of the crystals have been indexed by the software package Indx. And using the simulated hkl values and d spacing values lattice parameters are calculated with

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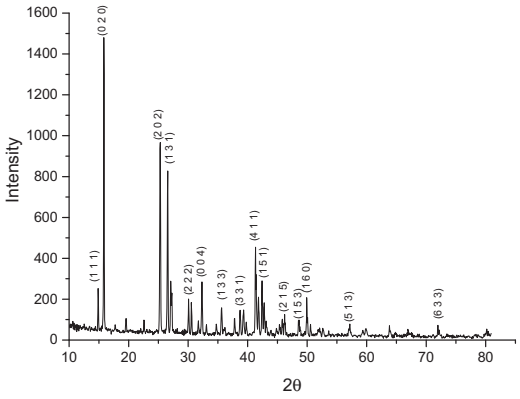

Fig. 1. Powder XRD pattern of KB5.

Table 1
Comparison of crystal data of single crystal XRD and powder XRD.

	a (Å)	*b* (Å)	*c* (Å)	Volume (Å^3)
Single crystal XRD	9.09	11.20	11.10	1130
Powder XRD	9.02	11.19	11.13	1126

the help of programme, Unit Cell. The calculated values of lattice parameters are compared with values obtained from single crystal XRD in Table 1. It shows that the lattice parameters calculated from powder XRD is closely matched with results obtained from Single crystal XRD.

3.3. UV–vis–NIR spectral study of KB5

UV–vis–NIR characterization is an important study for NLO crystals to know the optical transmission range of the crystals. The grown crystals of KB5 were subjected to absorption-transmission measurements in the spectral region of 190–1100 nm using the Perkin-Elmer, UV–vis–NIR spectrophotometer. The transmission spectrum for KB5 crystal is shown in Fig. 2. There is no absorption between 200 to1100 nm. The good transmission property of the crystal in the entire visible region suggests its suitability for NLO applications.

3.4. FT-IR and FT-Raman spectral study of KB5

The FT-IR spectrum is recorded in the range 400–4000 cm^{-1}, by Perkin-Elmer spectrometer. FT-IR spectrum of KB5 is given in Fig. 3. OBO ring bending is assigned to 456.42 cm^{-1} and 509.63 cm^{-1}. At 629.59 cm^{-1} OBO ring asymmetric bending is assigned. B–O ring stretching is assigned to 777.12 cm^{-1} and 921.12 cm^{-1}. At

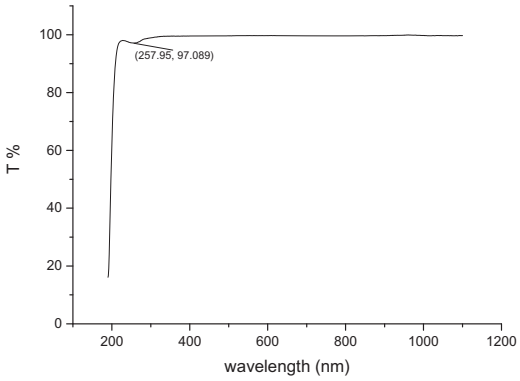

Fig. 2. Transmission spectrum of KB5.

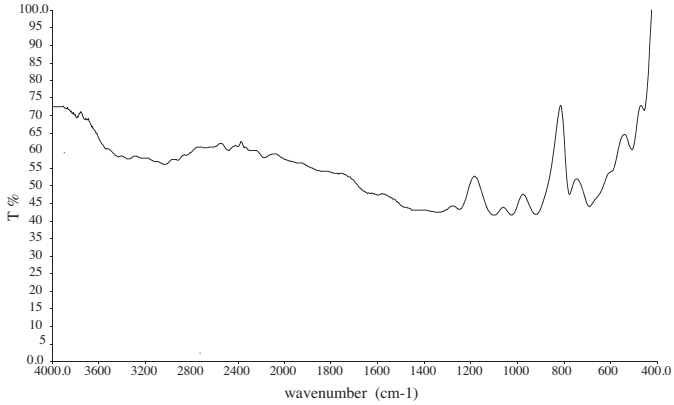

Fig. 3. FT-IR spectrum of KB5.

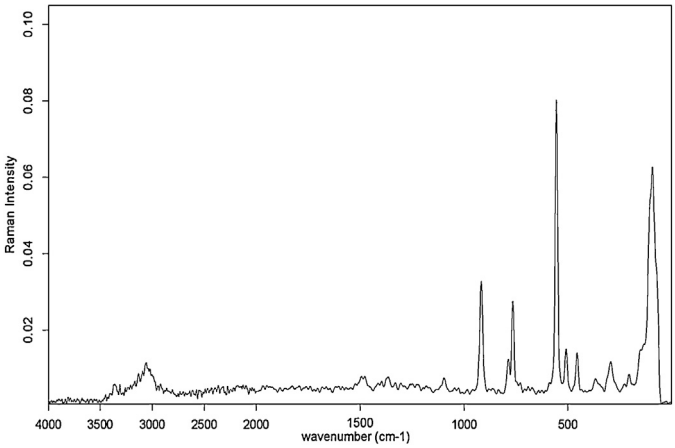

Fig. 4. FT-Raman spectrum of KB5.

Table 2
Assignments of FTIR and FT-Raman spectra.

FT-IR (cm^{-1})	FT-Raman (cm^{-1})	Assignments
–	367.39	OBO deformation
455.42	455.69	OBO ring bending
509.63	508.61	OBO ring bending
–	555.45	OBO terminal bending
629.59	–	OBO ring asymmetric bending
–	765.29	B–O ring stretching
777.12	786.65	B–O ring stretching
921.12	917.31	B–O ring stretching
1026	–	B–O terminal stretching
1102.20	1095.71	B–O asymmetric stretching
1250.27	–	B–O vibrations
1337.60	–	B–O asymmetric stretching

1102.20 cm^{-1} and 1337.60 cm^{-1} B–O asymmetric stretching is assigned.

FT-Raman spectrum of KB5 crystals are recorded using BRUKER RFS 27 FT-Raman spectrometer with Nd: YAG laser (1064 nm). Sample was scanned over the range of 50–4000 cm^{-1}. The recorded FT-Raman spectrum is shown in Fig. 4. Peak observed at 367.39 cm^{-1} is due to OBO deformation. The observed peaks at 455.69 cm^{-1} and 508.6 cm^{-1} are due to the OBO ring bending. 786 cm^{-1} and 917.31 cm^{-1} peaks are attributed to B–O ring stretching. OBO terminal bending is indicated by 555.45 cm^{-1} peak. Comparison of FT-IR and FT-Raman assignments is shown in Table 2 [6].