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Structural, Micro hardness and Etching studies of Barium- Strontium Borate (BSB) Single crystals

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ABSTRACT

Inorganic borates exist in numerous structural types and some crystals such as KB5 and BBO are excellent non-linear optical (NLO) materials, particularly in the UV region. These borate crystals generally possess chemical stability, high damage threshold and high optical quality, as well as wide range of transparency far into the ultraviolet on account of the rather larger difference in the electro negativities of B and O atoms. A single crystal of Barium- Strontium Borate (BSB) has been synthesized from slow solvent evaporation technique. The grown crystals of size up to 2.5x 2 x 6 mm^3 are obtained in a period of 50-90 days. The crystal structure was confirmed by Single crystal XRD. The mechanical stability of the sample was determined by Vicker hardness study. Etch pattern was observed by optical microscope.

KEY WORDS: NLO materials, Crystal Growth, Borate crystal, Microhardness, Chemical etching.

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1. INTRODUCTION

Borate family complexes are excellent nonlinear optical (NLO) materials, as they possess high chemical stability, damage threshold, optical quality and wide range of transparency [1]. Since the discovery of BaB_2O_4 (BBO)[2],various non-centrosymmetric borate crystals, e.g.LiB_3O_5 (LBO)[3], CsB_3O_5(CBO)[4], $CsLiB_6O_{10}$ (CLBO)[5] and $K_2Al_2B_2O_7$ (KABO) [6], have been synthesized and intensively studied because of their excellent NLO properties. Hence the present investigations deals with structural, Microhardness and Etching studies of BSB single crystals.

2. EXPERIMENTAL PROCEDURE

Barium strontium borate (BSB) was synthesized by thoroughly mixing barium chloride ($BaCl_2.2H_2O$) and strontium chloride ($SrCl_2.2H_2O$) in 10:1 molar ratio with excess of boric acid in millipore water. The content was stirred continuously for 3 days at a constant temperature 60 ºC.

The resulting solution was filtered and kept undisturbed in a beaker to initiate nucleation. Seed crystals were formed within a period of 7 to 10 days due to spontaneous nucleation. The seed crystals were optically transparent with clear morphology. After careful selection of the seed crystals, they were immersed in to the mother solution using nylon thread to facilitate the growth of bulk size crystals. Well defined and transparent crystals of dimension up to 2.5 x 2 x 6 mm^3 were grown within 50-90 days by slow solvent evaporation technique at room temperature. The crystals (Figure. 1) are optically transparent and non hygroscopic in nature.

3. CHARACTERIZATION

3.1 Single crystal X-ray diffraction

Before performing the experiment with X-ray diffract meter, the crystal quality of the sample was verified with Leica polarizing microscope. Single crystal of suitable size was cut and mounted on the X-ray goniometer. The crystal was optically centered at the sphere of confusion using the built in tele-microscope. Twenty five reflections were collected from different zones of the reciprocal lattice using random search procedure. The reflections were indexed using method of short vectors followed by least square refinements. The unit cell parameters thus obtained were transformed to correct Bravais cell.

Single crystal XRD data of $Ba_{11}Sr(BO_3)_8$ crystal indicates its monoclinic crystal system, with a non- centrosymmetric space group 2mB. The cell parameters are a= 7.1308Å, b= 10.9075 Å, c= 40.3358 Å, and V= 3136.667$Å^3$.

3.2 Micro hardness test

Hardness study on as grown crystals of BSB was carried out by static indentation test at room temperature using HMV SHIMADZU micro hardness tester, fitted with Vickers diamond pyramidal indenter. Several indentations were made on the most prominent face (0 1 0) of the crystal by varying the loads from 10-30 g and the hardness number (H_v) was determined. The indentation time was kept as 15 s for all the loads. Test loads above 30 g developed multiple cracks on the crystal surface due to the release of internal stresses generated locally by indentation. Variation of hardness number (H_v) with applied load (p) is shown in Figure 2. It is significant to note that the value of hardness increases and then attains almost saturation with the increase of applied load. The maximum value of hardness was found to be 30.5 kg/mm^2 at a load of 30 g. The value of the work hardening coefficient 'n' was estimated from the plot of log p versus log d (Figure 3) by the least square fit method. The value of 'n' is found to be 3.306 for (0 1 0) plane. For specimens showing increase in H_v with increasing p, the Meyer index n > 2, which confirms the reverse ISE behaviour sangwal[7]. Since the value of n for BSB is greater than 2, the hardness of the material is found to increase with applied load further confirming the prediction of Onitsch[8].

3.3 Etching studies

The etching technique is the simplest characterization technique that can be best employed to study the defect structure of a single crystal [9]. However, the success of this technique lies in the efficiency of the chemical etchant to sense the dislocation sites selectively. Subsequently, etch pits are formed at the dislocation centers on those faces at which the additives are bound. Water is a superior etching solution for revealing dislocation etchpits and it is in sensitive to surface orientation as it produced pits almost on all surfaces. BSB crystal was etched in water with 2s. The etched surface was soaked with a filter paper and examined under an optical microscope, it exhibit square shaped etch pattern shown in Fig. 4

4.0. CONCLUSION

The Barium- Strontium Borate (BSB) single crystals were harvested by slow solvent evaporation method. The crystal structure of the BSB was confirmed by Single crystal XRD. The maximum value of hardness was found to be 30.5 kg/mm^2 at a load of 30 g from the microhardness testing method. The etch pattern of the BSB single crystal was checked by chemical etching method.

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Fig. 4: Etch pattern produced on the plane of BSB crystal by acetone after etching for 2 s

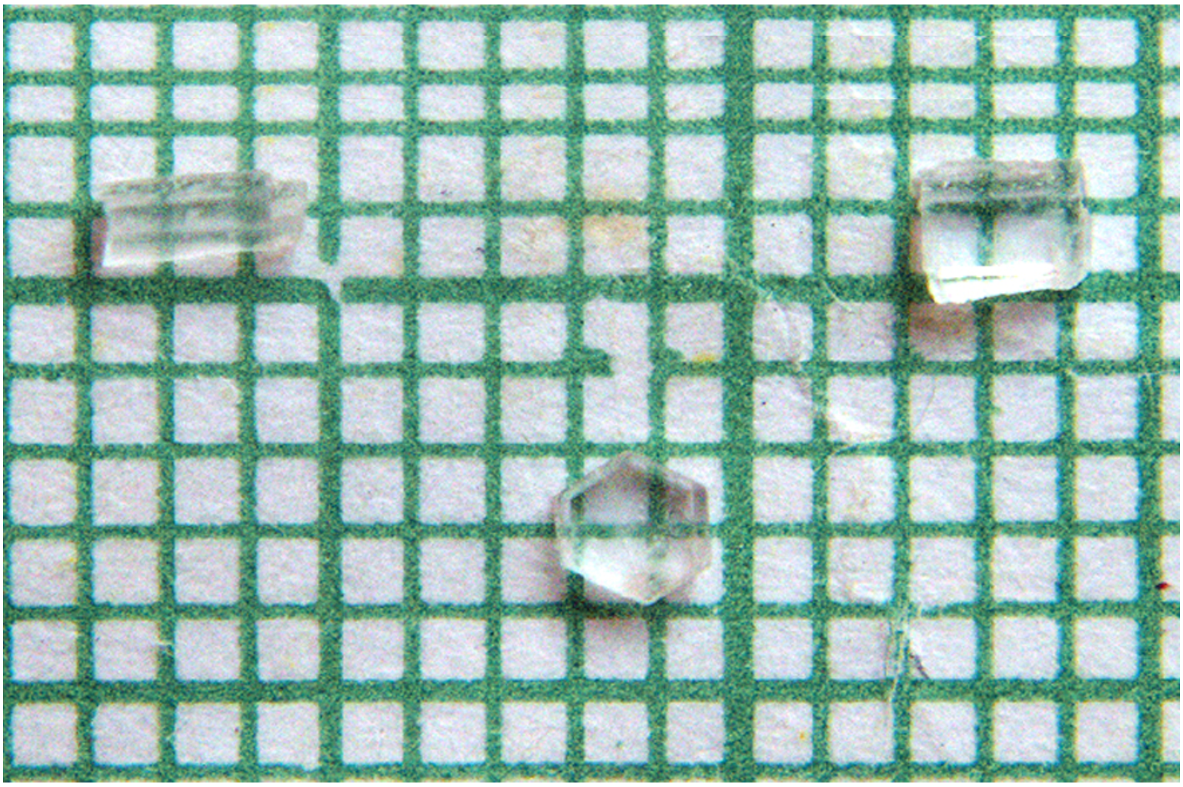


Fig.1: As grown crystals of Barium- Strontium Borate (BSB) single crystals

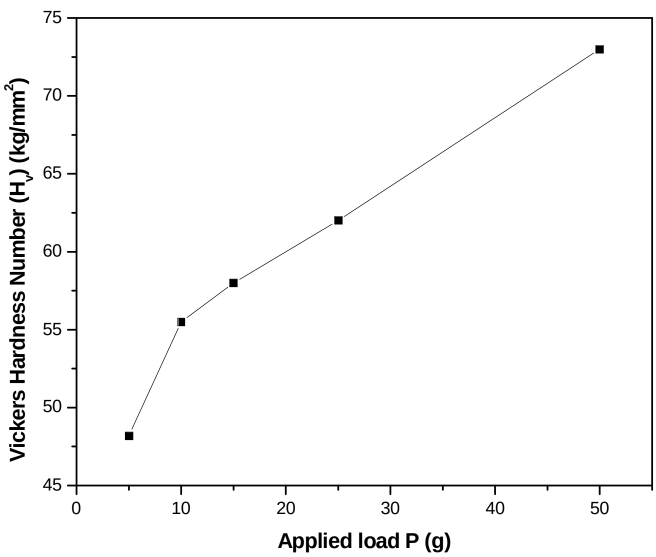


Fig. 2: Vickers hardness Vs Applied load of single crystal of BSB

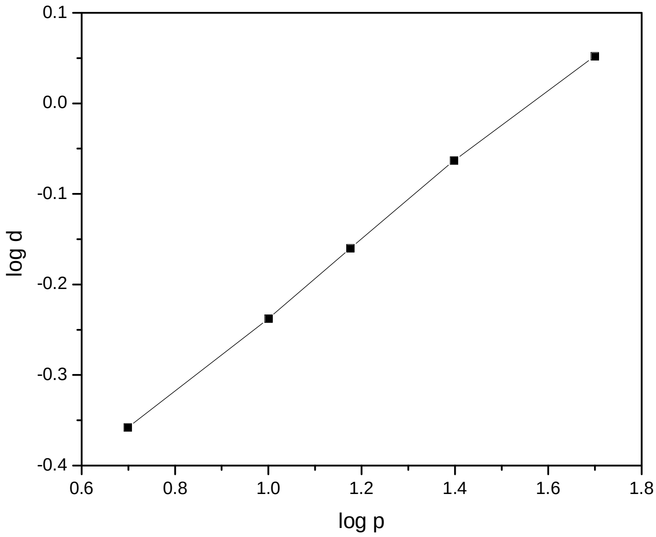
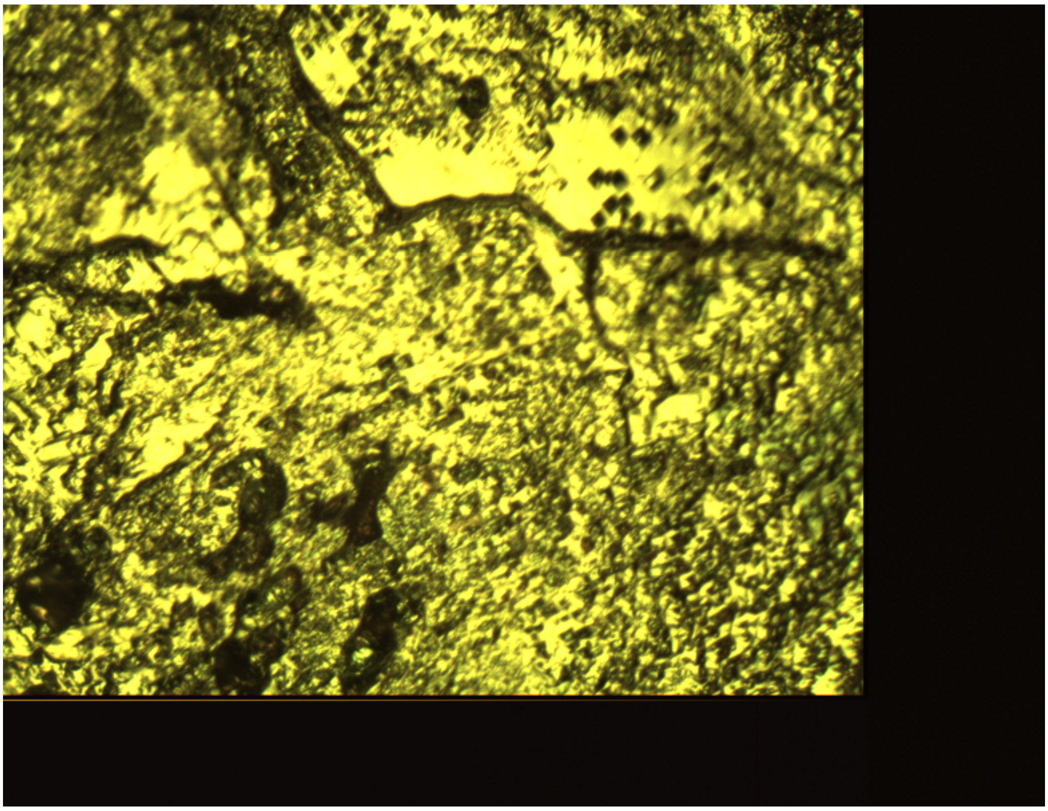


Fig. 3: Plot of Log d vs Log P for BSB single crystal



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