

Growth and Microhardness Studies on Bismuth Sulpho Fluoride Single Crystals

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Bismuth sulpho fluoride is one of the ternary compounds belonging to V-VI-VII group compounds. Being a ferro electric crystal, it exhibits various interesting properties. Thiourea solution of concentration 10% by weight was prepared. It was mixed with bismuth oxide in hydrogen fluoride and crystals were harvested. For the indentation test Vicker's hardness tester was used. The mechanical properties were discussed.

INTRODUCTION

From the wide variety of current techniques for the growth of single crystals, the gel technique has gained considerable importance due to its simplicity and the effective growth of single crystals. $A^vB^viC^{vii}$ group compounds (A^v being antimony or bismuth, B^vi being sulphur, selenium or tellurium and C^{vii} being chlorine, bromine or iodine) are semiconduction ferroelectric materials. They exhibit certain other inherent characteristics like electro-optic, photo-electric, piezo-electric, electro-chemical and electro-mechanical properties. These properties have been interlinked with each other ensuring other peculiar properties. Among the $A^vB^viC^{vii}$ compounds BiSF has been reported as useful material for device applications¹. This material has been used in light modulation time controlling devices and IR radiation convertors².

The hardness of a material is the resistance it offers to indentation by a much harder body. An important use of microhardness studies is the possibility of making an indirect estimate of other mechanical characteristics of materials having a specific correlation with their hardness. The hardness depends not only on the properties of material under test but also largely on the conditions of measurement³. Microhardness method is widely used for studying the individual structural constituent elements of minerals, glasses, enamels and artificial abrasives.

EXPERIMENTAL

A known quantity of thiourea solution of concentration ranging from 8 to 10 per cent by weight was mixed with 100 mL of sodium silicate gel solution of density 1.04 g/cm³. The gel solution impregnated with the inner reactant was acidified with 1 N acetic acid and taken in straight tubes of different diameters (1 to 3 cm) and length (10 to 25 cm) and allowed to set. The period of gelation was varied from 1 to 24 h. by changing the pH value of the solution⁴. After gelation, solution of Bi₂O₃ (5 to 10 g) dissolved in 100 mL of 10 N HF was taken over the set gel. The outer reactant diffuses into the gel medium and reacts with the inner reactant giving rise to single crystal of BiSF.

The basic test of indentation is named after Brinell⁵ and Vickers. In this type of test an indenter is forced by a known load into the surface of the material and the hardness is calculated from the size of indentation mark, typically 0.5 to 5 mm across. The Knoop test, which is used on very hard materials, is a miniature form of the Vickers test. Smith and Sandland proposed a pyramid substituting a ball in order to provide geometrical similitude under different values of load. The apex of the indenter was 136 degrees since this is the angle subtended by the tangents of a sphere. The Vickers hardness may be calculated by this method also.

Vickers pyramidal indenter has been used to study the microhardness of the gel grown crystals of bismuth sulpho halides and its type⁶. Selected smooth surfaces of the crystals were subjected to static indentation test at room temperature. A Leitz-Wetzlar hardness tester fitted with the diamond pyramidal indenter attached to an incident light microscope was used in the present work. The load (P) was varied from 2 to 100 g and the time of indentation was kept constant at 10 seconds for all the trials. The diagonal length (d) of the indented impressions obtained at various loads was measured using a micrometer eye piece. Several indentation trials at each load were carried out and the average value of the diagonal length of the indentation mark in each trial was calculated. The microhardness value H_v was calculated using the relation

$$H_v = 1.8544 p/d^2 \text{ kg mm}^{-2}$$

where

H_v is the Vickers microhardness number,

p is the applied load in kg,

d is the average diagonal length of the indentation impression.

RESULTS AND DISCUSSION

Table-1 shows the hardness value obtained for different loads on the BiSF crystal. When the load increases microhardness number (H_v) decreases and attains a constant value after a particular load. Figure 1 (a) shows the indentation marks on the (100) face of BiSF crystal at 10 g. Fig. 1 (b) shows the indentation marks on the (100) faces of BiSF crystal at 20 g.

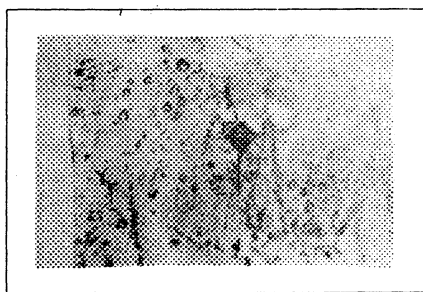


Fig. 1 (a) Indentation marks on the (100) faces of BiSF crystals (weight 10 g).

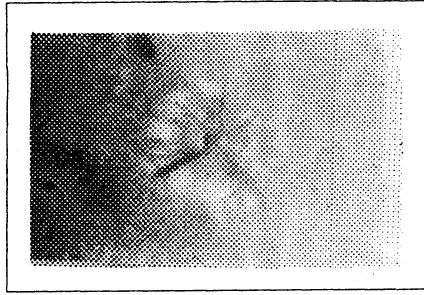


Fig. 1 (b) Indentation marks on the (100) faces of BiSF crystals producing cracks.

TABLE-1
HARDNESS VALUE OBTAINED FOR DIFFERENT LOADS OF BiSF CRYSTALS

Load (P) gms	Diagonal length (d) μm	H_v kg mm^{-2}
5	10.98	76.940
10	16.12	71.360
20	23.35	68.020
30	29.91	62.185
40	34.48	62.390
50	38.65	62.036

Slip lines were observed at each corner of the impression at 20 g load. Maximum indenter load applied for BiSF was 50 g, because microcracks were observed around the impression; cracks initiate at 20 g load and tend to propagate with the increased load of 10 g as may be evident from Fig. 1 (b). This implies that the increased hardness of BiSF crystals as also reflected from the decreased value of hardness number⁷, shows the variation of hardness with indenter load. With the gradual increase of the applied stress, the elastic limit of the material can be exceeded and the specimen will not restore its original shape on the removal of the stress. In the present case, microhardness number H_v was 62.036.

Conclusion

The hardness properties of the gel grown crystals of bismuth sulpho fluoride have been studied by micro-indentation technique. The microhardness numbers have been calculated for gel grown BiSF crystal and the H_v value decreases when load increases. The H_v becomes almost constant after a load of 30 g.

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