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Study on Indentation Size Effect and Microhardness of Borate Doped Glasses

***S. Thirumaran and A. Priyadharsini[1]**

**Department of Physics (DDE), Annamalai University, Annamalai Nagar, India.*
[1] Department of Physics, Annamalai University, Annamalai Nagar, India.

Abstract

The present study divulging about the room temperature measurement of microhardness of glass samples doped with borate content namely, (i) B_2O_3-WO_3-Bi_2O_3 (BTB-glass systems) and (ii) B_2O_3-SiO_2-Bi_2O_3 (BSB-glass systems). The Microhardness probing was carried out by using HMV-2T SHIMADZU hardness tester with a Vicker's diamond pyramidal indenter. It is observed that the hardness of the glass materials are decreasing with increase of applied load in all the two glass systems, exhibiting the direct Indentation Size Effect (ISE). XRD spectral investigation has been carried out with a view to ascertain the amorphous nature of the glass specimen. Meyer's work hardening index number has also been evaluated to probe the strength of the glass specimen. The present investigation clearly advocates that BSB glass systems possess more softness over the BTB glass systems.

Keywords: Indentation Size Effect, Pyramidal Indenter, Amorphous, Work Hardening Index Number.

INTRODUCTION

Microhardness method is considered to be very useful for testing on a wide type of materials, including metals, composites, ceramics or applications such as testing foils, measuring surface of a part , testing individual micro structures or measuring the depth of case hardening by sectioning a part and making a series of indentations. The hardness of a material is defined as its resistance to another material penetrating its surface and is related to its wear resistance and strength. It is otherwise may be treated as the resistance of a material to permanent deformation such as indentation, wear, abrassion, scratch principally, the importance of hardness testing with the relationship between hardness and other properties of material. The Vickers hardness test method used to measure the hardness of materials [1]. The Vickers test is easier to use comparing other hardness test since the required calculations are independent of the size of the indenter, and the indenter can be used for all materials irrespective of hardness. Indentation hardness test compose the majority processes used to determine material hardness. The Vickers hardness test method is based on an optical measurement system. A square base pyramid shaped diamond is used for testing in the Vickers scale. Generally, the apparent hardness of the materials varies with applied load, known as the indentation size effect (ISE), usually involves a decrease in the microhardness with increasing applied load[2]. In contrast to the ISE, a reverse type of indentation size effect (reverse ISE), is also known[3], where the micro hardness increases with increasing applied load.

The present work has been initiated to investigate the effect of borate ions doped with other Bi_2O_3, WO_3 and SiO_2 ions with various studies like x- ray diffraction (XRD) and microhardness were carried out in order to explore its amorphous nature and the nature of the glass specimen.

MATERIALS AND METHODS

The chemicals utilized in the present study for the preparation of glass specimen were obtained from the standard and reputed chemical companies. The glass composition was prepared in mole percentage (mol %) with increasing content of borate with decreasing content of Bi_2O_3 in BTB glass systems and increasing content of borate with decreasing content of Bi_2O_3 in BSB glass systems where the mole percentage increased in steps of 2 % have been prepared by using the conventional melt-quenching technique. The proper compositions were mixed together by grinding the mixture repeatedly to obtain a fine powder. The mixture is melted in silica crucible and melting was carried out under controlled muffle furnace with occasional stirring. The temperature controlled furnace was gradually raised to a higher temperature at the rate of 373 K per hour and a glassy structure was noticed at 1303K for BTB glass systems and 1353 K for BSB glass systems respectively. Eventually the molten glass melt was immediately poured on a heavy copper molding block having the dimensions 12mm diameter and 4mm thickness kept at a room temperature. Then the glass samples were annealed at 673 K for two hours to avoid the mechanical strains developed during the quenching process. The muffle furnace was switched off and glass was allowed to cool gradually at room temperature. The prepared glass samples were polished and the surface is made perfectly plane and smoothened. Nomenclature and glass composition in mol % are given in Table 1 and the photographs of BTB and BSB glass systems are displayed in figure 1.

Table 1: Nomenclature and Composition of Glass Specimen

S. No.	Nomenclature	Composition in mol %	Remark
System I (B_2O_3 – WO_3- Bi_2O_3)			
1	BTB-1	65-05-30	Mol% of WO_3 is kept constant
2	BTB-2	67-05-28	
3	BTB-3	69-05-26	
4	BTB-4	71-05-24	
5	BTB-5	73-05-22	
6	BTB-6	75-05-20	
System II (B_2O_3- SiO_2-Bi_2O_3)			Mol% of SiO_2 is kept constant
1	BSB-1	65-07-28	
2	BSB-2	67-07-26	
3	BSB-3	69-07-24	
4	BSB-4	71-07-22	
5	BSB-5	73-07-20	
6	BSB-6	75-07-18	

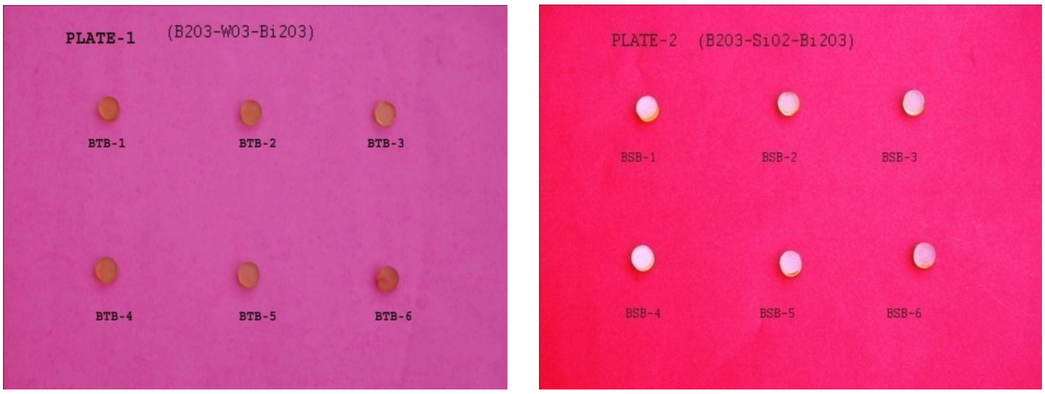
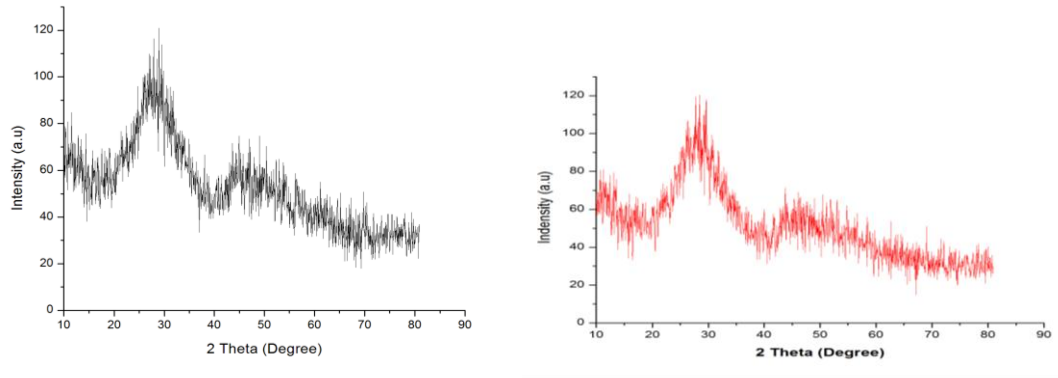


Figure 1. Plate – 1. BTB glass system and plate – 2. BSB glass system

The glassy nature of the prepared glass specimen was confirmed by crystallographic analysis. The crystallization of the glass was determined in powder form through x-ray diffraction (XRD) analysis on a model : XPERT –PRO X-ray diffract meter at a range of 2ϴ = (10-80˚) utilizing cu-kα (1.5406 A˚) radiation with an applied voltage of 40 kV and 30 mA anode current at a rate of 2 ˚/ min^{-1}, with a resolution of 0.001˚. The XRD spectra (figure 2a &2b) taken for two glass systems BTB and BSB find no continuous or discrete sharp peak revealing their amorphous nature [4]. Microhardness measurement was carried out using HMV – 2T hardness tester fitted with a vicker's diamond pyramidal indenter.



2(a).BTB Glass system 2(b).BSB Glass system

Figure 2a & 2b. XRD pattern of BTB and BSB glass samples

THEORY

Microhardness testing is one of the best methods of understanding the mechanical properties of materials such as fracture behavior, yield strength, brittleness index and temperature of cracking [5]. All the indentation measurements were carried out on the freshly polished glass at room temperature. The indentation was made by varying the load from 50 gm, 100 gm up to 200 gm with the time of indentation was 10 sec each. The indented impressions were approximately square. Diagonal length of the indented impression was measured using calibrated micrometer attached to the eye piece of the microscope.

Vickers microhardness value H_V [6] has been calculated using

$$H_V = 1.8544\ P/d^2 \text{ -------------------- } (1)$$

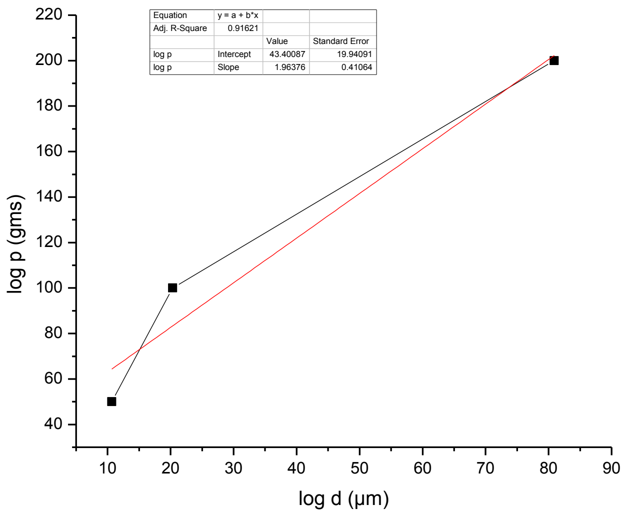
Where p is the applied load, d is the mean diagonal length of the indentation impression and 1.8544 is a constant.

The Meyer's index number (n) was calculated from the Meyer's law, which relates the load and indentation diagonal length

$$P = Kd^{\,n} \text{ ------------------------------ } (2)$$

$$\log P = \log k + n \log d \text{ -------------- } (3)$$

Where k is the material constant and n is the Meyer's index. Figure 3 shows Plot of log P *versus* log d gives a straight line and slope of the line yields the value of 'n'which is useful to determine whether the material is hard or soft.



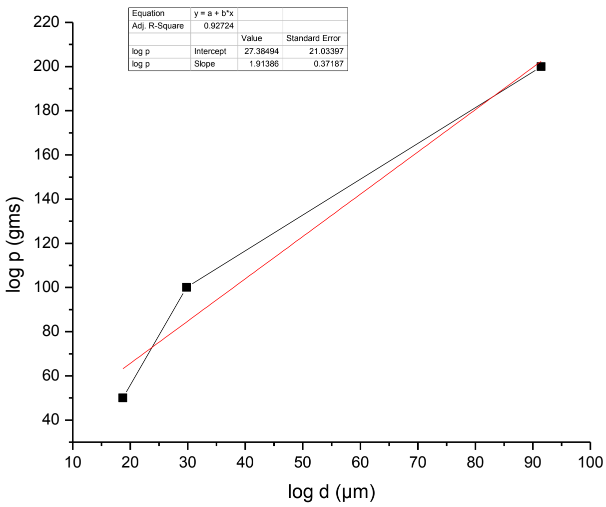


Figure 3. Plot of log p versus log d for BTB- 6 and BSB- 6 glass system

RESULTS AND DISCUSSION

The experimental values of microhardness (Hv) and Meyer's Index Number (n) with various applied load for the two glass systems BTB & BSB are reported in table 2 . It is seen that as the value of applied load increases, the microhardness (H_v) decreases for higher loads up to 200gm revealing the direct indentation size effect (ISE). Above 200gm cracks were initiated on the glass specimen and the decrease in hardness is attributed to the high stress required for homogeneous nucleation of dislocation in the small dislocation free region indented[7, 8] .

Table 2 : Values of microhardness (Hv) and Meyer's index number / work hardening exponent (n) for various glass compositions with different applied load at room temperature

Glass samples label	Glass composition in mol %	Microhardness (Hv)Mpa			Meyer's index number / work hardening (n)
		Load (gms)			
		50	100	200	
BTB – 1	65-05-30	634	605	516	1.6163
BTB – 2	67-05-28	581	546	501	1.6225
BTB – 3	69-05-26	563	530	493	1.6717
BTB – 4	71-05-24	550	528	488	1.7694
BTB – 5	73-05-22	474	471	460	1.7940
BTB – 6	75-05-20	468	462	455	1.9636
BSB – 1	65-07-28	459	440	432	1.6434
BSB – 2	67-07-26	466	462	457	1.6523
BSB – 3	69-07-24	497	489	473	1.6820
BSB – 4	71-07-22	525	504	494	1.7500
BSB – 5	73-07-20	576	554	498	1.8602
BSB – 6	75-07-18	631	619	597	1.9138

When the applied load is small, the indenter penetrates only the surface layers, and hence consequently dislocations are nucleated along particular slip plane near the surface. When the penetration depth increases applied load, nucleation of dislocation involves in another set of slip planes just below the indentor. After a certain penetration, the effect of inner slip planes along the surface layers and hence the microhardness is independent on load[9]. It can also be interpreted, as the depth of the indentor increases with load, the effect of the distortion zone decreases and hence the dependence of microhardness is less. For large values (above 200gm), the indenter reaches the undistorted zone, hence the microhardness is independent on the load[10, 11].

It is well known that the apparent microhardness of solids depends on the applied indentation test load, known as Indentation Size Effect (ISE), which usually involves a decrease in the apparent microhardness with increasing of applied test load[12]. In all the studied BTB glass systems and BSB glass systems, the microhardness number

(V_H) decrease with increasing load, which is well in agreement with direct indentation size effect (ISE).

The variation of microhardness number (V_H) with the applied load for the sample is shown in the figure 4. The results show that the hardness number decreases gradually up to 200gm with the increase of applied load, indicating that the sample could withstand the weight up to 200 gm. The decreasing part of the curve is due to direct indentation size effect.

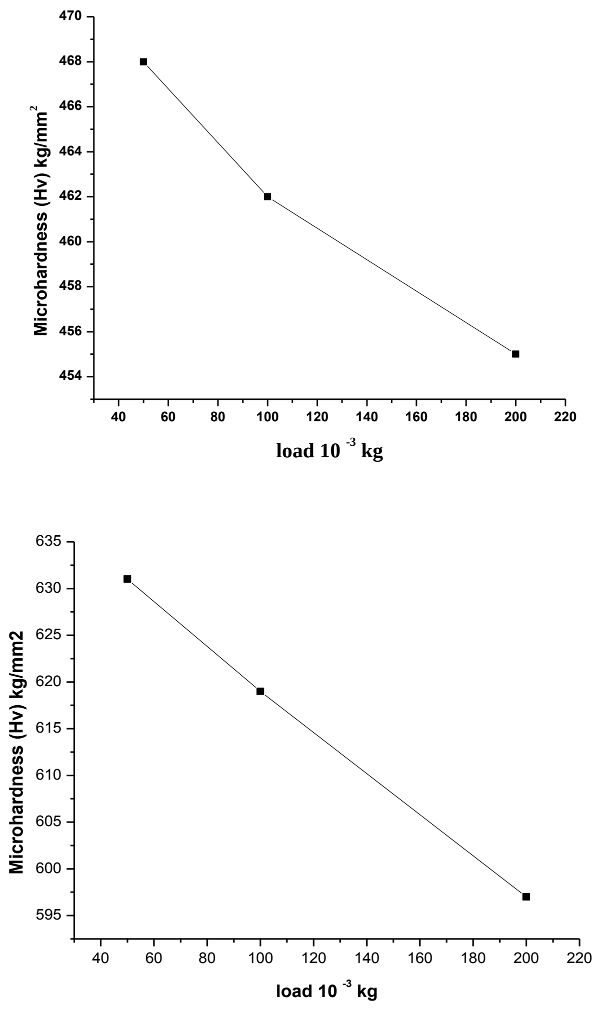


Figure 4. Microhardness vs. Load for BTB-6 and BSB-6 Glass

When a sharp indenter such as Vickers indenter is loaded onto a glass, a residual surface impression is observed after unloading, and the material hardness is conveniently estimated from the projected area of the impression. It is known that some extent of deformation during loading is recovered during unloading, i.e., elastic recovery. Since it is expected that the degree of elastic recovery depends on glass systems and glass compositions, the hardness evaluated from a residual surface impression after unloading is insufficient for an in-depth understanding of deformation behaviors in glass.

The evaluated work hardening exponent (or) Meyers Index Range (n) [13] in this study is found to be less than 2. From the earlier works, it is learnt that the value of 'n' lies between 1 and 1.6 for hard materials and it is more than 1.6 for soft materials[14]. From the Table -2, the present evaluated value of Meyer's Index (n) is found around 1.9 clearly suggesting that the present prepared glass specimen is belonging to the soft material category.

CONCLUSION

The XRD spectral investigation confirms the amorphous nature of glass specimen. The microhardness measurement reveals that the prepared glass specimen exhibiting Direct Indentation Size Effect (ISE). From the trend of Meyer's Index, it is obvious that the present two glass systems BTB & BSB belonging to the soft material category. Among the two glass systems taken for investigation, it is quite obvious that BSB glass system possess more softness and less rigidity over the BTB glass system.

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