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Vickers Micro hardness Measurement of Glass-Nanocomposites

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Abstract: *Our results open the way to the preparation, application and investigation of significant mechanical properties of new type of copper-molybdate glass-nanocomposites and their heat-treated counterparts. We have observed giant hardness, shear modulus and yield stress, even greater than that of steel. This surprising result shed some light on the materials for their important potential applications in which a material of high strength and hardness is needed. It deserves further intensive exploration by advanced methods such as the one presented here.*

1. INTRODUCTION

The term hardness refers to stiffness or temper or to resistance to bending, scratching, abrasion or cutting. It also gives the ability to resist bending permanently and resist deformation, when a load is applied. The greater the hardness of the metal, the greater resistance to deformation. Since the hardness of a material correlates directly with its strength, wear resistance and other mechanical properties. As a result, hardness testing is widely used for material evaluation, because of its simplicity and low cost relative to direct other measurements. Hardness is one of the most basic mechanical properties of engineering materials.

This test is also employed for quality assurance in parts which require high wear resistance such as gears [1]. It plays a key role in the progression of civilization because the sophistication and reliability of the component or a machine depend on it [2,3]. Tensile strength for many metals are an indicator of wear resistance and ductility and show the relationship with depth of indention and other mechanical properties like ductility, engineering strength, Composition of metals and ceramics, Anisotropy of materials surface, Cutting ability ,Abrasive strength, Fracture strength, Surface coating quality, depth and structure, hardness of different components of alloys, Degree of crystallinity in thermoplastic polymers, Degree of cross-linking (curing) in thermosetting polymers, Glass transition temperature(Tg), Time-dependent visco-elastic properties [4,5]. The relationship among Brinell, Vickers, or Rockwell hardness values is possible with the help of conversion tables. The conversion values are solely empirical in nature [6].

Hardness is measured by the applied force divided by the indentation surface area. Pelleg [7] pointed out that the mean pressure under the indenter is ~30. It is also a measure of forge ability of a material undergoing a cold forming process [8]. There are different types of hardness testing methods. Commonly used tests for hardness are: (a) Brinell hardness test, (b) Vickers hardness test, (c) Rockwell hardness test, (d) Rebound or Dynamic hardness test, (e) Scratch test (f) Meyer Hardness test. Among of them the Vickers hardness test method is a versatile hardness test, since it may be adapted for micro hardness testing, as well as for a variety of materials, such as metals, ceramics Glasses, composites etc. and also used for the determination of hardness of very thin and very hard materials.

In recent years, glass-nano composites materials are technically considered of great interest on account of their unique properties, such as high refractive index [9], high dielectric constants [2,9]. These glasses process several advantages such as absence of grain boundaries, isotropic properties, ease of thin film formation and greater stability to moisture as well as high chemical durability, low melting point and good mechanical properties like hardness, although empirical in nature can be correlated to young modulus, shear modulus, tensile strength, yield strength etc.

Chen and et. al. [10] shows that the intrinsic correlation between hardness and elasticity of materials correctly predicts Vickers's hardness for a wide Variety of crystalline materials as well as Bulk Metallic Glasses and also suggested if a material is intrinsically brittle Vickers's hardness linearly correlates with the shear modulus (Hv = 0.151G) and the hardness of crystalline materials can be correlated with the product of the squared Pagh's Modulus ratio (K) and the shear

modulus (G) [10]. Many researchers and scientists were studied Elastic and structural properties of the glasses by measuring sound velocity using the pulse-echo overlap technique [3, 8, 11, 12, 13] some of them are represented the relationship between hardness and Young modulus (E or Y) of the materials by using Ultrasonic pulse-echo method [8, 11, 12].

In the present study, we have reported the formation of coppermolybdate ($CuMoO_4$) nanoparticles embedded in the $xCuI$-$(1-x)(0.5CuO$-$0.5MoO_3)$ glass-nanocomposites and Silver-molybdate ($AgMoO_4$) nanoparticles embedded in the $xAgI$-$(1-x)MoO_3$ and $xAgI$ $(1-x)(0.5Ag_2O$-$0.5CuO)$ where x = 0.1, 0.2, 0.3, 0.4 and 0.5 were prepared from the reagent grade chemicals CuI, CuO and MoO_3. The mechanical properties of these as prepared samples were measured and calculated.

2. EXPERIMENTAL DETAILS

2.1. Sample Preparation

The Glass-nanocomposites samples with composition of $xCuO - (1-x)V_2O_5$; $xCuO - (1-x)MoO_3$; where x = 0.1; $xCuI$-$(1-x)(0.5CuO$-$0.5MoO_3)$ and $xAgI$-$(1-x)MoO_3$ where x = 0.1, 0.2, 0.3, 0.4 and 0.5 and $xAgI$-$(1-x)\{0.5Ag_2O$-$0.5CuO\}$, where x = 0.5, 0.6, 0.7, were prepared by the melt-quenching technique from the reagent grade chemicals CuI, AgI, V_2O_5, Ag_2O CuO and MoO_3.The molecular weight percentage of high purity nano Cuprous Iodide(CuI), Cupric Oxide(CuO), Silver Iodide(AgI), Silve Oxide(Ag_2O) and Molybdenum Trioxide(MoO_3) nano powders were mixed and continuously ground in a mortar and pestle set with adding some Ethyl alcohol for better mixing and good homogeneity. Drier was used to remove the alcohol from the mixture. The dry powder mixture poured in an alumina or silicon carbide crucible and placed inside an electric muffle furnace and melted in the furnace from 700^0C to 800 ^{0}C temperature depending upon the composition. The melts have been equilibrated for 30 minutes and then the melt mixture was quickly quenched by pouring on to a stainless steel mold to make the partially transparent molybdate glass-nanocomposites plates of thickness ~ 1 mm were obtained under pressure and then annealed for 24 hours in air to avoid the mechanical strain developed during the quenching process. The as prepared samples were chemically stable but hygroscopic so the samples were preserved in a moisture free sample container (racicator). The amorphous nature of the as prepared samples was confirmed by X-ray diffraction technique by an X-ray diffractometer and the X-ray diffraction (XRD) patterns of the samples have been recorded.

For achieving precise hardness measurement values, the polished and cleaned specimen surface should be set up perpendicularly to the direction of indentation of a hardness testing machine. A mixture of cold mounting powder and cold mounting liquid is used for mounting the samples and the mounted glass samples were polished using different graded emery (sand) papers to produce smooth parallel opposite surface and perpendicular to the diamond indenter axis for better indentation. Finally the samples were made smooth surface by using the cloth felted polishing machine.

2.2. Characterization of samples

The amorphous nature of the glass samples was confirmed by X-Ray Diffraction (XRD) using X'Pert Pro Panalytical diffractometer. After grinding all the samples into powder form, the glass transition temperature (Tg) of these samples were determined by differential scanning calorimetry.

2.3. Vickers hardness (Hv)

The traditional Vickers hardness tests were carried out under 1,5,7,10,15, 25 and 50gf indenter load respectively in different samples and the indenter diagonals were measured using a reflected light microscope (OLYMPUs, Japan) 20x resolution. H_V measurements were made with a standard Vickers hardness tester (Leco Micro hardness tester, Model No 700, loading range 1gf – 100gf. Five indentations were made on each specimen and a distance at least 40 times greater than the indentation depth was kept between the centre of each indentation and the edge of the sample.

3. RESULTS AND DISCUSSION

Previously we have reported that during X-ray diffraction pattern shows there is no peaks which indicates the amorphous nature of all the glass nano composite samples [13-15].The micro-hardness of the as-prepared glass-nanocomposites has been evaluated from Vickers hardness (H_V) tester using the relation

$$H_v = 2PSin\ (136^0/2)/d^2 \quad (1)$$

where, P is the applied load and d is the arithmetic mean of the two diagonals d_1 and d_2 [15].

From experimental data analysis it has been found that the variation of H_V as a function of applied indentation test loads ranging from 0.00981 to 0.981N for the samples. For each load five or more number of indentations were used to calculate the Vicker's microhardness(H_v) is based on the average of the measured diagonals for a particular load.

It has been found that the maximum ±5% difference between the calculated hardness values and the machine reading. The vicker's micro hardness values H_v in GPa of different compositions surface are against the different loads are shown in Fig. 1. All the graphs show that due to increase of indentation load the hardness remains constant. Also it has been found that increase the CuI doped or AgI doped increase the mole weight percentage but decreases the hardness as shown in Fig. 1.

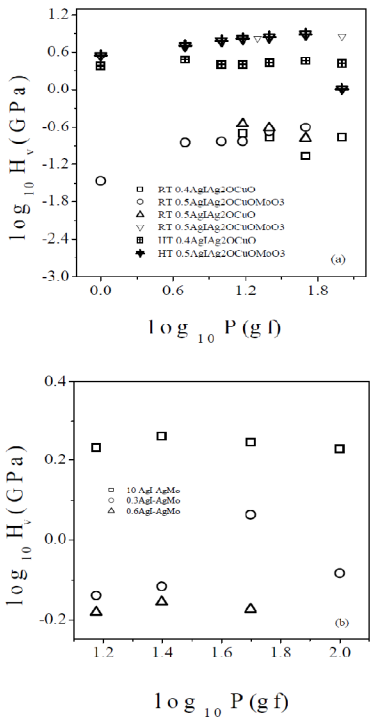

Fig: 1(a,b) Graph of Hardness versus Shear Modulus of different room temperature glass nanocomposites and heat treated glass nanocomposites

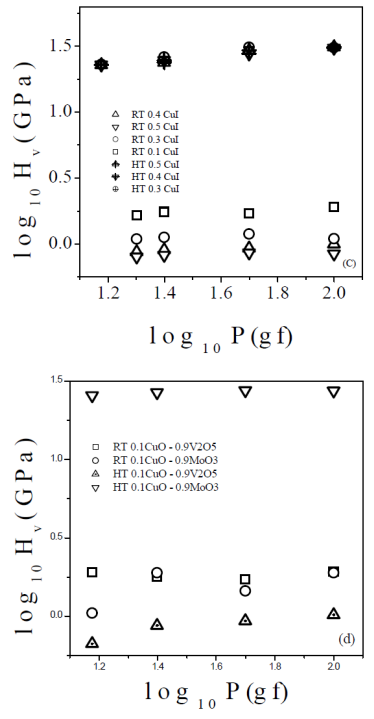

Fig: 1(c,d) Graph of Hardness versus Shear Modulus of different room temperature glass nanocomposites and heat treated glass nanocomposites

From the calculated shear modulus values of the as prepared samples are more than the metal alloy like Aluminum, Brass, copper magnesium, nickel steel etc. The Graphs on hardness verses Shear modulus of different room temperature glass nanocomposites and heat treated glass nanocomposites are shown in fig 4. Which represent that increases the load increase the shear modulus. On the other hand except 0.1CuO - 0.9V_2O_5 there is a noticeable change of hardness in case of heat treated samples. From the experiment it has been found that the hardness of the heat treated samples increases more than steel as recorded table 1.

Table 1(a): Hardness values of room temperature and heat treated as prepared samples

0.1CuO - 0.9V2O5		
Room Temp.	**Heat Treated**	
Load (N)	(Hv in Gpa)	(Hv in Gpa)
0.14715	1.906453	0.666949
0.24525	1.795734	0.873721
0.4905	1.71347	0.935998
0.981	1.92975	1.021906

Table 1(b): Hardness values of room temperature and heat treated as prepared samples

0.1CuO - 0.9MoO_3		
Room Temp.	**Heat Treated**	
Load (N)	(Hv in Gpa)	(Hv in Gpa)
0.14715	1.04923	25.545
0.24525	1.895641	26.7480
0.4905	1.447771	27.4764
0.981	1.891059	27.3751

Table 1(c): Hardness values of room temperature and heat treated as prepared samples

0.3CuI-0.35CuO-0.35MoO_3		
Room Temp.	**Heat Treated**	
Load (N)	(Hv in Gpa)	(Hv in Gpa)
0.1962	1.09263	23.04096
0.24525	1.12065	26.17359
0.4905	1.19327	31.11692
0.981	1.09635	30.9244

Table 1(d): Hardness values of room temperature and heat treated as prepared samples

0.4CuI	**0.3CuO**	**0.3MoO_3**
Room Temp.	**Heat Treated**	
Load (N)	(Hv in Gpa)	(Hv in Gpa)
0.1962	0.871851	22.90763
0.24525	0.905793	23.79891
0.4905	0.933302	29.40647
0.981	1.000051	31.13515

Table 1(e): Hardness values of room temperature and heat treated as prepared samples

0.5CuI-0.25CuO-0.25MoO_3		
Room Temp.	**Heat Treated**	
Load (N)	(Hv in Gpa)	(Hv in Gpa)
0.1962	0.801017	22.85461
0.24525	0.821528	24.66009
0.4905	0.856209	27.73614
0.981	0.840859	30.60431

Table 1(f): Hardness values of room temperature and heat treated as prepared samples

0.6AgI-0.4(0.3Ag_2O-0.7MoO_3)		
Room Temp.	**Heat Treated**	
Load (N)	(Hv in Gpa)	(Hv in Gpa)
0.14715	0.658781	12.02566
0.24525	0.69926	11.68462
0.4905	0.66969	11.76405

The variation of H_V with applied load shows that the H_V increases with increasing applied indentation test load and after the certain load hardness remain constant and all curves are parallel to each other. For brittle materials, it is common observation that cracking occurs during indenter loading half cycle [6]. Fig. 2 shows the variation of hardness H_v with indentation load P for all the compositions. It is clear from Fig. 2 that for the particular composition the hardness remains same within limited range of P.

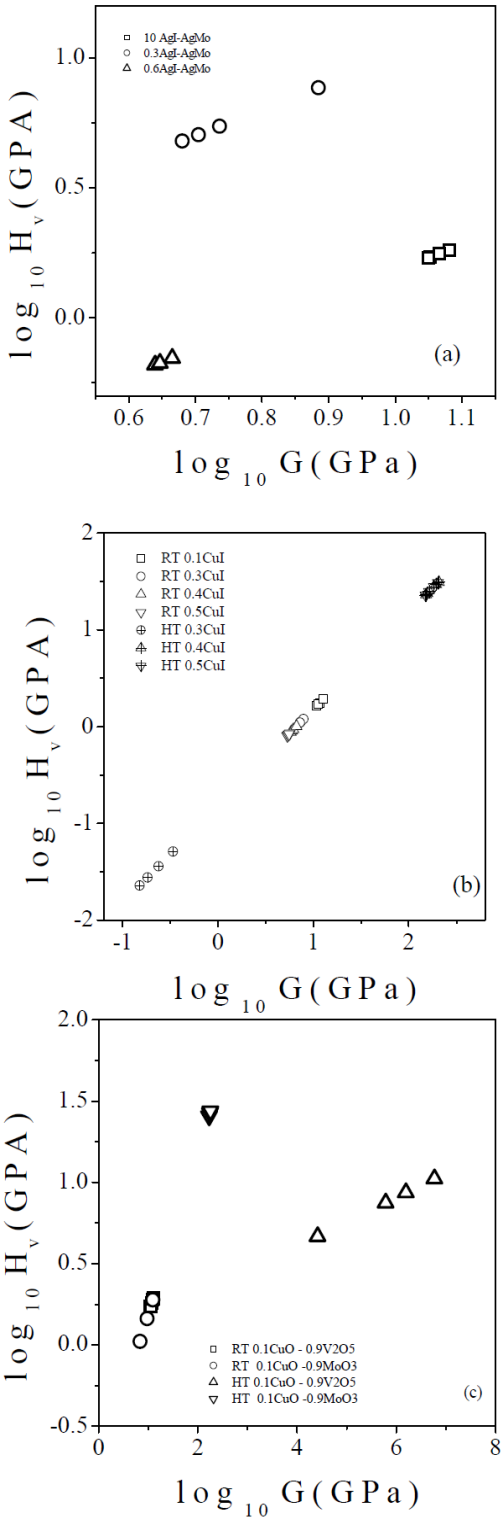

Fig.2 Graph on hardness verses Shear modulus of different room temperature glass nanocomposites and heat treated glass nanocomposites

This directly indicates that average diagonal (d) should be increased. We may conclude from the above fact that the hardness of the samples under investigation is not anisotropic in nature. As the CuI and AgI content decreases, the hardness is found to increase. When the CuI and AgI content is high, H_v shows saturation. So we also conclude that the hardness (H_v) is strongly dependent upon composition but independent on applied load for all the $xCuI-(1-x)(0.5CuO-0.5MoO_3)$ and $xAgI - (1-x)MoO_3$ It has also observed from the X-ray diffraction analysis that more number of $CuMoO_4$ nanoparticles is present in composites with x = 0.1 and density of them decreases with the increase of x. As a consequence, the microhardness changes. Thus the microhardness of the resultant composite depends upon both, the content of CuI, AgI or CuO and the growth of the $CuMoO_4$ or $AgMoO_4$ nanoparticles in the glass matrix [14].

4. CONCLUSIONS

Among the various indentation hardness testing methods, the Vickers microhardness(H_v) testing method has been employed in the present investigation. The microhardness behaviour of all the Glass nano composites samples are studied in terms of various parameters. It has been observed that the vicker's hardness (H_v) is strongly dependent upon composition but independent on applied load (P) for all the glass-nanocomposites within limited range of load (P). The variation of Vickers micro hardness numbers (H_v) with indentation loads of all the studied specimens, show an increase of hardness up to a certain load which is different for different compositions and then remains constant and independent of load. After heat treatment hardness drastically increases which is more than steel so we can conclude that the heat treated samples density increases.

5. ACKNOWLEDGEMENT

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REFERENCES

[1] Mechanical Metallurgy Laboratory 431303, "Tensile Testing" May- Aug 2007, T. Udomphol

[2] P. Zhang, S.X. Li, Z.F. Zhang, "General Relationship between Strength and hardness". J. Mat. Sc. and Engg. A,529,(2011) 62-73.

[3] Fazil O. Sonmez, Ahmet Demir, "Analytical relations between hardness and strain for cold formed parts", J. Mat. Processing Technology 186 (2007) 163–173.

[4] Vickers Hardness Measurements: Practices and Calibration Check, The University of British Columbia, Internal Technical Bulletin, v1.0, July 22, 2010

[5] Zbigniew H. Stachurski. "Testing materials' hardness science, technology and applications", Materials Forum, Volume 30 – (2006), 118-125.

[6] Metallographic and Materialographic Specimen Preparation, Light Microscopy, Image Analysis and HardnessTesting, Kay Geels In collaboration with Daniel B. Fowler, Wolf-Ulrich Kopp, and Michael Rückert, ASTM Stock No. MNL46. 623-643.

[7] Solid Mechanics and its Applications, Volume 190, Joshua Pelleg Materials Engineering, Springer (2013), 36 – 50.

[8] R. Palani and G. Srinivasan. "Elastic Properties of Ba^{2+} and Mn^{2+} metal ion doped lithium Borate Glasses using Pulse-echo Technique" International J. of recent Scientific research, Vol 3,Issue 11' (2012), 992 – 996.

[9] Material Science, S.L. Kakani, Amit Kakani, New Age International (P) Ltd., Publishers (2004) 249

[10] Xing-Qiu Chen, Haiyang Niu, Dianzhong Li, Yiyi Li, "Intrinsic Correlation between Hardness and Elasticity in Polycrystalline Materials and Bulk Metallic Glasses", Cond-mat.mtrl-sc (2011).

[11] Tosawat Seetawan, Vittaya Amornkitbamrung, Thanusit Burinprakhon, Santi Maensiri, Ken Kurosaki, Hiroaki Muta, Masayoshi Unob, Shinsuke Yamanaka. "Mechanical properties of Ag-doped Na1.5Co2O4", Journal of Alloys and Compounds 403 (2005) 308–311.

[12] Emad S. Al-Hasani. "Study of Tensile Strength and Hardness Property for Epoxy Reinforced With Glass Fiber Layers" Eng. & Technology, Vol.25, No.8, (2007), 988-996.

[13] S. Laila, A.K. Suraya, A.K. Yahya. "Effect of Glass Network Modification on Elastic and Structural Properties of Mixed Electronic-Ionic 35V2O5-(65-x)TeO2-(x)Li2O Glass System", Chalcogenide Letters vol. 11, no. 2, (2014),91-104.

[14] H.S. Güder, E. Sahin, O. Sahin, H. Göçmez, C. Duran and H. Ali Çetinkara, "Vickers and Knoop Indentation Microhardness Study of β-SiAlON Ceramic", Acta Physica Polonica a, no 6, Vol. 120 (2011),1026-1032.

[15] Arun Kr. Bar, Sanjib Bhattacharya and Debasish Roy. "Structural Study of Molybdate Glass-Nanocomposites", J. of Advanced Physics Vol. 2, pp. 2013,(1-4).