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Mechanical Properties Study of Borosilicate Glass Loaded with Vanadium and Cobalt by Nanoindentation Technique †

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Abstract: Nanoindentation test is used to investigate the mechanical properties of borosilicate glass with composition (mol%) 40 $Na_2B_4O_7$ − 40 SiO_2 − (20− x) V_2O_5 − x Co_2O_3, with x = 0, 1, 3, and 5 mol% for samples A, B, C and D respectively were prepared by melt quenching technique at 1100 °C. A load-displacement curve was plotted and used to extract some mechanical properties of the glass samples. The creep deformation behavior of the glass composition was studied; the maximum creep rate was observed for the sample that contains the highest vanadium oxide content, while the creep rate decreased with decreasing vanadium oxide content in glass samples. The hardness and the reduced modulus of elasticity were obtained. The Maxwell-Voigt model was applied to investigate the relaxation kinetics and deformation of the bulk glass.

Keywords: borosilicate glass; nanoindentation test; mechanical properties

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1. Introduction

Adequate glass election for different applications is decisive for successful product design. The glass's physical properties determine its capability to combat damage and prevent failure. The material surface properties are usually correlated to their performance as a final product. Nanoindentation testing is a powerful, complex, and substantial method to investigate small-scale material surface properties. The mechanical properties of the glass have a serious role in the success of the final product, so the material's hardness, strength, impact, and abrasion resistance should be taken into account when selecting the material. On a microscopic level, glass is elastic in nature. In contrast, on a macroscopic level, glass is very stiff, which means a very small strain is obtained at a typically applied stress which will not cause a shift out of its specified dimensions. So, Nanoindentation or scratch techniques can be used to identify the hardness of glass, where the other mechanical properties can be extracted to understand its nature. Creep behavior and viscoelastic deformation were investigated for La and Ce based bulk metallic glasses (BMGs) using Nanoindentation technique at room temperature the time dependent was described using kelvin model, the results were compared with that obtained from uniaxial compression and a great agreement was obtained [1]. Deformation and mechanical properties were studied using Nanoindentation experiments with a Berkovich tip on Zirconium based bulk metallic glass, the effect of loading rate and cyclic loads on Nanomechanical properties were studied. A significant effect of loading rate on Pop-in which becomes predominant at higher loads, hardness becomes peak load-independent according to the Oliver-Pharr method, an increase in elastic modulus with load takes place [2]. This study aims to investigate the mechanical properties of borosilicate glasses with different concentrations of vanadium and cobalt oxides by a Nanoindentation technique.

2. Materials and Methods

Borosilicate glass with composition $40SiO_2 - (20 - x)\ V_2O_5 - x\ CO_2O_3 - 40\ Na_2B_4O_7$ with x = 0,1,3 and 5 mol% for samples A, B, C and D respectively, prepared by melt quenching technique at 1100 °C then the resulting melt was poured on a cupper plate to quench it and annealed at 300 °C for 5 h to get rid of internal stresses. An extensive discussion of the methodology may be found in our earlier publication [3], and XRD verified the amorphous nature of all glass samples.

The mechanical properties (hardness, load depth, young's modulus, creep) were investigated using Nano test Vantage instrument according to ASTM-E-2546 [4]. Each sample was performed for four times and then averaged. A gradual increase in the applied load up to 400 mN with 10 mN/s loading rate. This maximum load was held for 20 secs then unloading with the same rate takes place.

3. Results

3.1. Load vs. Displacement Curve

Figure 1 exhibits the load-displacement curve for glass samples with different vanadium-cobalt concentrations. Four tests were conducted for each glass sample and then averaged for final results. All samples show a maximum load of 400 mN for 20 sec holding time. By increasing the load, the tip penetrates more depth with an increasing vanadium oxide content and decreasing cobalt oxide content, indicating more elasticity to glass samples with increasing vanadium oxide con. Sample A shows about 1300 nm of penetration depth while sample B reaches about 3500 nm, sample C indicates about 4500 nm, and the most elastic sample D indicates about 4600 nm penetration depth. Samples A and B show pops Ins. which means the presence of a very hard point of defect inside the sample that occurred during preparation which appears as shear bands under the indenter tip [5,6].

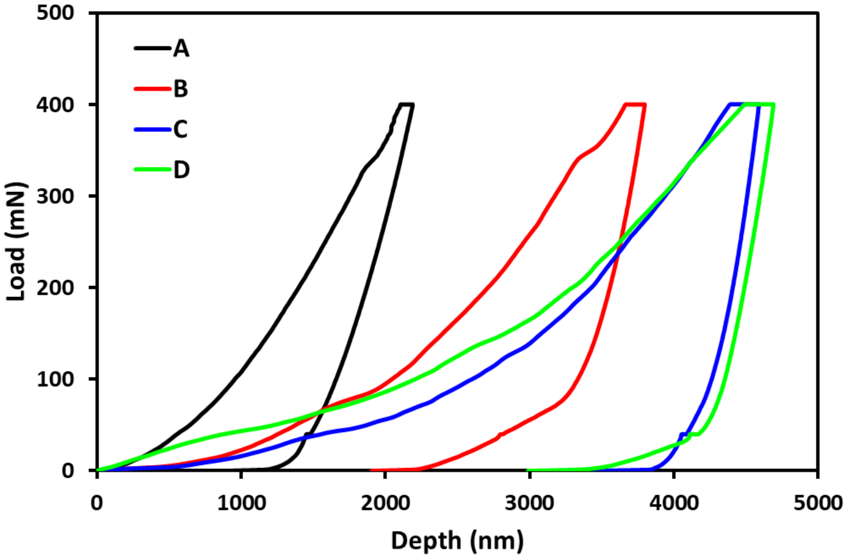


Figure 1. Load-displacement curve of glass samples.

3.2. Elastic Modulus and Hardness Measurments

The stiffness of the samples can be obtained from the slope of the upper part of the unloading curve S = dP/dh, from which we can obtain the reduced modulus from Equation (1)[7]:

$$E_r = \frac{\sqrt{\pi}}{2\beta}\frac{S}{\sqrt{A}} \tag{1}$$

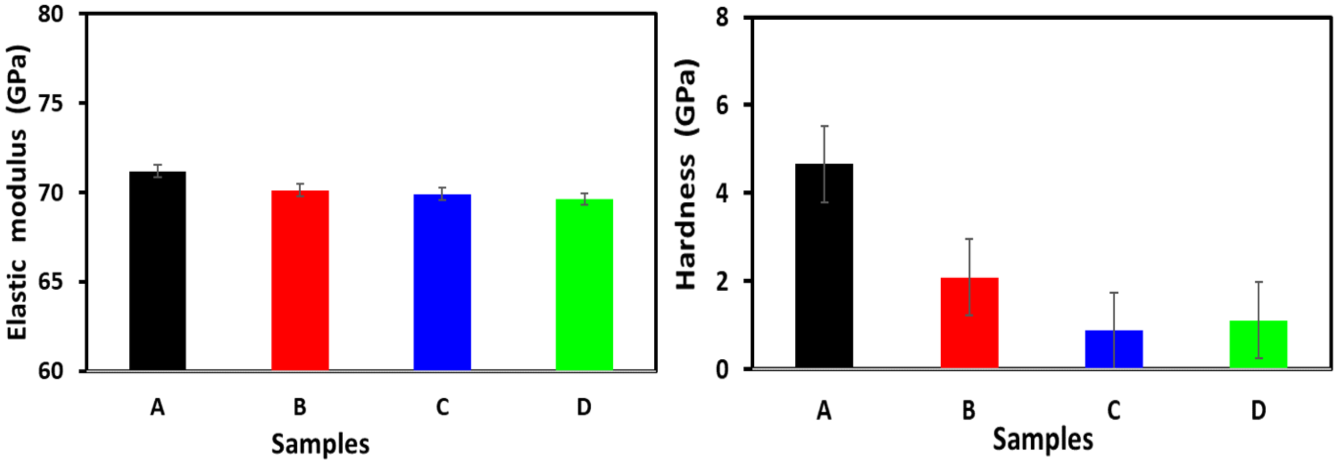
where Er is the reduced modulus of both the glass sample and indenter tip, and A is the project contact area [indenter tip area] and for Berkovich, β =1.034. To obtain the glass samples young's modulus we should apply Equation (2)[8]:

$$\frac{1}{E_r} = \frac{1-\nu^2}{E} + \frac{1-\nu_i^2}{E_i} \tag{2}$$

where E_i is elastic modulus of the indenter tip (1141 GPa) and ν_i = 0.07 which is the Poisson's ratio of indenter tip while E and γ are elastic modulus and Poisson's ratio for glass samples. The hardness (H) of all samples is calculated using Equation (3),

$$H = \frac{P_{max}}{A} \tag{3}$$

Figure 2a,b shows the elastic modulus and the hardness of glass samples, respectively. The elastic modulus slightly decreases as the vanadium oxide decreases. The decreases represent about 1.5%, 1.8%, and 2.3% for samples B, C, and D, respectively, compared with sample A. The sample with the lowest vanadium oxide has the largest stiffness value, the stiffness of the samples decreases with increasing vanadium oxide content, and so the hardness of the glass sample that contains the highest vanadium oxide content indicates greater hardness, while the hardness decreases with increasing vanadium content and shows 4.7 GPa, 2.1 GPa, 0.9 GPa and 1.1 GPa for samples A, B, C, and D. This represents 55%, 81%, and 76% decrease for samples B, C, and D, respectively, compared with sample A.



(**a**) (**b**)

Figure 2. (**a**) Elastic modules of glass samples; (**b**) Hardness of glass samples.

3.3. Creep Curve

The creep properties are crucial when they are used in structural parts by exhibiting the glass samples to a constant load at a constant temperature as a function of time[9]. A graph with three distinguished ranges was obtained (Figure 3). The first elastic region was obtained promptly after the load was applied, followed by a visco-elastic region characterized by a time-dependent decrease of the deformation rate and, finally, a pseudo-viscous range characterized by a constant rate of deformation.

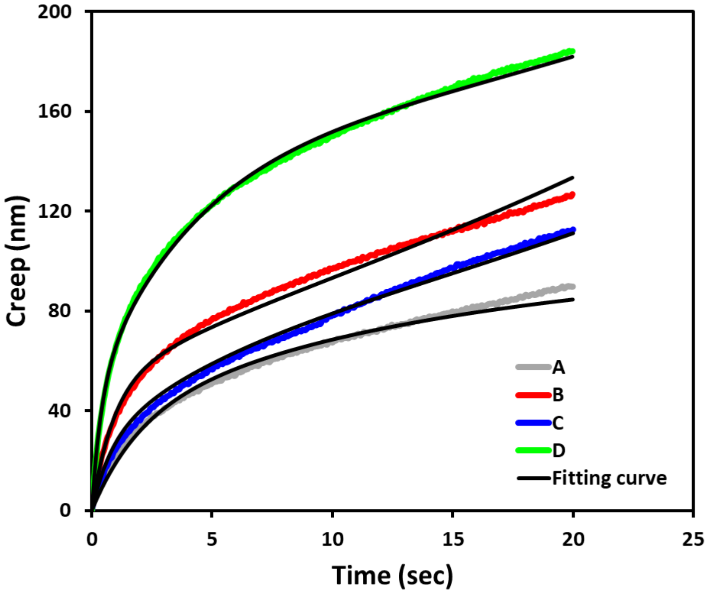


Figure 3. Creep of glass samples as a function of time.

Testing with nanoindenters induces elastic, anelastic, and viscoplastic strains in the glassy structure [10,11]. No macroscopic change or shear banding occurs in glass materials because of the anelastic strain, which is often correlated with the local atomic rearrangement of the glassy structure in the elastic zone below the yield stress. However, under prolonged external force, the glassy alloy exhibits viscoplastic strain [10,11]. The Maxwell-Voigt model may be used to characterize the viscoplastic strain as a function of indentation displacement, described by the following Equation:

$$h = \sum_{i=1}^{n} h_i \left(1 - e^{-t/\tau_i}\right) + \frac{t}{m} \tag{4}$$

where h_i is the indentation depth, t is the experimental time, τ_i is the characteristic relaxation time for the activation of the i-th anelastic process, and m is a constant proportional to the viscosity coefficient of the last dashpot. The stress-induced relaxation phenomena known as anelastic deformation follows the kinetics given by the first term of Eq (4). The second term stands for the creep's viscoplastic contribution.

As shown in Figure 3, the creep curves are fitted by two exponential decays, one for the anelastic deformation and one for the viscoplastic contribution (t/m). Table 1 displays the values of the fitting parameters for the creep curves. h_1 and τ_1 values characterize the first component of the anelastic deformation. In contrast, the h_2 and τ_2 values characterize the second component of the anelastic deformation. h_1 shows no discernible pattern, whereas h_2 rises as the vanadium content drops. As the ratio of vanadium drops, both τ_1 and τ_2 also decrease.

The anelastic component of creep can be evaluated in terms of a spectrum of relaxation times, and the isothermal relaxation spectra can be estimated using the equation (5)[12]:

$$L(\tau) = \left[\sum_{i=1}^{n} \left(1 + \frac{t}{\tau_i}\right) \frac{h_i}{t_i}\, e^{-t/\tau_i}\right] \frac{A_0}{P_0 h_{in}}\, t \tag{5}$$

where h_i, τ_i and t are the same parameters as in Equation (4), (A_0/P_0) is the inverse of the hardness H, and h_{in} is the maximum indentation depth.

As shown in Figure 4, all relaxation spectra have two peaks. The first peak with short relaxation times reflects hard region defects, while the second peak with long relaxation times reflects soft region defects. As the content of vanadium oxides drops, both peaks increase in intensity and move toward lower relaxation time. This is because the population of the relevant defects increases, making activation of the remaining defects simpler [13].

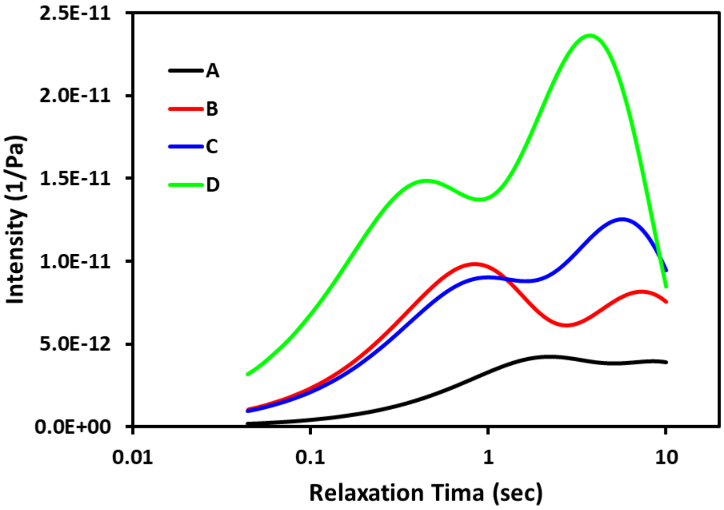


Figure 4. Spectra intensities of glass samples.

Table 1. Fitting parameters of creep curves of glass samples based on Maxwell–Voigt model.

Samples	h_1	τ_1	h_2	τ_2	m
A	35	1.9	50	11	2.58
B	50	0.9	50	9.0	3.2
D	27	0.8	59	7.0	3.74
C	48	0.4	110	4.6	3.96

4. Conclusions

The mechanical properties of any material are very important for final product selection. Samples of borosilicate glass loaded with vanadium and cobalt oxides show reasonable elastic behavior for different applications. The elasticity of the samples increased with increasing vanadium oxide content. The elastic modulus and stiffness slightly decreased as the vanadium oxide content increased. The hardness of the glass samples increased with vanadium oxide content. Three distinguished regions were appeared during creep testing as the first region was obtained promptly after applying the load followed by viscoelastic region characterized by time dependent decrease of the deformation, finally a pseudo- viscous region characterized by a constant deformation rate. The values of the fitting parameters for creep curves indicates that h_2 increases with vanadium oxide content decreasing and τ_1 and τ_2 decreased with this decreasing. All relaxation spectra show two peaks indicating hard and soft region defects.

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References

1. Wei, B.; Zhang, T.; Li, W.; Xing, D.; Zhang, L.; Wang, Y. Indentation Creep Behavior in Ce-Based Bulk Metallic Glasses at Room Temperature. *Mater. Trans.* **2005**, *46*, 2959–2962. https://doi.org/10.2320/matertrans.46.2959.
2. Marimuthu, K.P.; Lee, K.; Han, J.; Rickhey, F.; Lee, H. Nanoindentation of zirconium based bulk metallic glass and its nanomechanical properties. *J. Mater. Res. Technol.* **2019**, *9*, 104–114. https://doi.org/10.1016/j.jmrt.2019.10.034.
3. Hassaan, M.; Osman, H.; Hassan, H.; El-Deeb, A.; Helal, M. Optical and electrical studies of borosilicate glass containing vanadium and cobalt ions for smart windows applications. *Ceram. Int.* **2017**, *43*, 1795–1801. https://doi.org/10.1016/j.ceramint.2016.10.137.
4. ASTM E2546-15 Standard Practice for Instrumented Indentation Testing. *ASTM B. Stand.* **2015**, *1*, 1–23. https://doi.org/10.1520/e2546-15.
5. Anggono, A.D.; Elveny, M.; Abdelbasset, W.K.; Petrov, A.M.; Ershov, K.A.; Zhu, Y.; Yunusov, A.; Chupradit, S.; Mustafa, Y.F.; Surendar, A. Creep Deformation of Zr55Co25Al15Ni5 Bulk Metallic Glass Near Glass Transition Temperature: A Nanoindentation Study. *Trans. Indian Inst. Met.* **2021**, *75*, 673–680. https://doi.org/10.1007/s12666-021-02455-8.
6. Song, S.; Bei, H.; Wadsworth, J.; Nieh, T. Flow serration in a Zr-based bulk metallic glass in compression at low strain rates. *Intermetallics* **2008**, *16*, 813–818. https://doi.org/10.1016/j.intermet.2008.03.007.
7. Gong, J.; Miao, H.; Peng, Z. On the contact area for nanoindentation tests with Berkovich indenter: Case study on soda-lime glass. *Mater. Lett.* **2004**, *58*, 1349–1353. https://doi.org/10.1016/j.matlet.2003.09.026.
8. Tao, K.; Qiao, J.; He, Q.; Song, K.; Yang, Y. Revealing the structural heterogeneity of metallic glass: Mechanical spectroscopy and nanoindentation experiments. *Int. J. Mech. Sci.* **2021**, *201*, 106469. https://doi.org/10.1016/j.ijmecsci.2021.106469.
9. Raj, R.; Chyung, C. Solution-precipitation creep in glass ceramics. *Acta Met.* **1981**, *29*, 159–166. https://doi.org/10.1016/0001-6160(81)90096-1.
10. Gong, P.; Jin, J.; Deng, L.; Wang, S.; Gu, J.; Yao, K.; Wang, X. Room Temperature Nanoindentation Creep Behavior of TiZrHfBeCu (Ni) High Entropy Bulk Metallic Glasses. *Mater. Sci. Eng. A* **2017**, *688*, 174–179.
11. Anggono, A.D.; Elveny, M.; Abdelbasset, W.K.; Petrov, A.M.; Ershov, K.A.; Zhu, Y.; Yunusov, A.; Chupradit, S.; Mustafa, Y.F.; Surendar, A. Creep Deformation of Zr55Co25Al15Ni5 Bulk Metallic Glass Near Glass Transition Temperature: A Nanoindentation Study. *Trans. Indian Inst. Met.* **2021**, *75*, 673–680. https://doi.org/10.1007/s12666-021-02455-8.
12. Castellero, A.; Moser, B.; Uhlenhaut, D.I.; Dalla Torre, F.H.; Löffler, J.F. Room-Temperature Creep and Structural Relaxation of Mg–Cu–Y Metallic Glasses. Acta Mater. **2008**, *56*, 3777–3785.
13. Yuan, C.; Liu, R.; Pang, C.; Zuo, X.; Li, B.; Song, S.; Hu, J.; Zhu, W.; Shen, B. Anelastic and viscoplastic deformation in a Fe-based metallic glass. *J. Alloy. Compd.* **2020**, *853*, 157233. https://doi.org/10.1016/j.jallcom.2020.157233.