engineering proceedings

Proceeding Paper

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

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† Presented at the 3rd International Electronic Conference on Applied Sciences, 1–15 December 2022; Available online: https://asec2022.sciforum.net/.

Abstract: The nanoindentation test was used to investigate the mechanical properties of borosilicate glass with a composition (mol%) of 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. Samples were prepared using the melt quenching technique at 1100 °C. A load–displacement curve was plotted and used to extract select 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 contained the highest vanadium oxide content, and the creep rate decreased with a decrease in the vanadium oxide content in the glass samples. The hardness and 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

Citation: El-Deeb, A.S.; Taha, E.O.; Kader, M.M.A.; Naguib, H.M.; Hassaan, M.Y. Mechanical Properties Study of Borosilicate Glass Loaded with Vanadium and Cobalt by Nanoindentation Technique. *Eng. Proc.* **2023**, *31*, 81. https://doi.org/10.3390/ASEC2022-13816

Academic Editor: Nunzio Cennamo

Published: 5 December 2022

1. Introduction

Adequate glass election for different applications is decisive for successful product design. The physical properties of the glass 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 for investigating small-scale material surface properties. The mechanical properties of the glass have a serious role in the success of the final product, so the hardness, strength, impact, and abrasion resistance of the material should be considered when selecting the material. On a microscopic level, glass is elastic in nature. In contrast, glass is very stiff on a macroscopic level. This means that a very small strain is obtained at a typically applied stress, which will not cause a shift out of its specified dimensions. Therefore, nanoindentation or scratch techniques can be used to identify the hardness of glass, from which 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 the nanoindentation technique at room temperature. The time dependence was described using a Kelvin model, the results were compared with results 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 the loading rate and cyclic loads on the nanomechanical properties were studied. A significant effect of the loading rate on pop-ins becomes predominant at higher loads. Hardness becomes peak-load-independent according to the Oliver–Pharr method, and an increase in the 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 using a nanoindentation technique.

2. Materials and Methods

Borosilicate glass with a composition of 40SiO_2-(20-x) V_2O_5-x CO_2O_3-40 $Na_2B_4O_7$ with x = 0, 1.0, 3.0, and 5.0 mol% for samples A, B, C, and D respectively, was prepared using the melt quenching technique at 1100 °C. The resulting melt was poured on a copper plate to quench it and then annealed at 300 °C for 5 h to eliminate internal stresses. An extensive discussion of the methodology may be found in our earlier publication [3], and X-ray diffraction (XRD) verified the amorphous nature of all glass samples.

The mechanical properties (hardness, load depth, Young's modulus, and creep) were investigated using Nanotest Vantage instrument (Micro Materials Co., Wrexham, UK) according to ASTM-E-2546 [4]. Each sample was tested four times and the results were averaged. A gradual increase in the applied load up of to 400 mN with a 10 mN/s loading rate took place. This maximum load was held for 20 s, followed by unloading at the same rate.

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 the final results. All samples showed a maximum load of 400 mN for a 20 s holding time. By increasing the load, the tip penetrated to a greater depth with an increasing vanadium oxide content and a decreasing cobalt oxide content, indicating more elasticity to glass samples with an increased vanadium oxide content. Sample A shows approximately 1300 nm of penetration depth, while sample B reached approximately 3500 nm, sample C indicates approximately 4500 nm, and the most elastic sample, sample D, indicated a penetration depth of approximately 4600 nm. Samples A and B show pops-ins, which indicate the presence of a very hard point of defect inside the sample that occurred during preparation and appears as a shear band under the indenter tip [5,6].

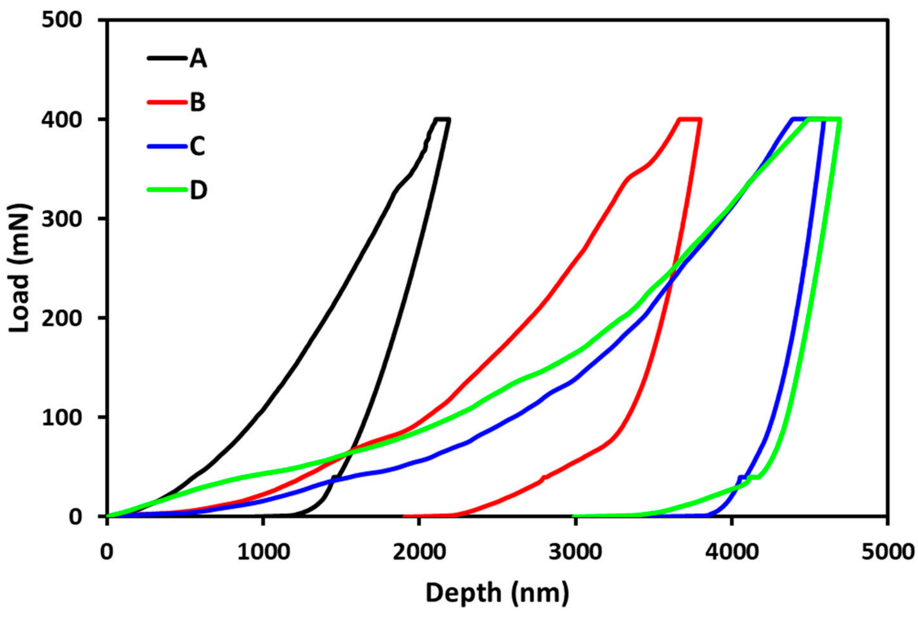

Figure 1. Load-displacement curve of glass samples.

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]:

$$E_r = \frac{\sqrt{\pi}}{2\beta}\frac{S}{\sqrt{A}} \quad (1)$$

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

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

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

$$H = \frac{P_{max}}{A} \quad (3)$$

Figure 2a,b show the elastic modulus and the hardness of glass samples, respectively. The elastic modulus slightly decreases as the vanadium oxide content 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 had the largest stiffness value, and the stiffness of the samples decreases with an increasing vanadium oxide content. Therefore, the hardness of the glass sample that contained the highest vanadium oxide content indicates a greater hardness, while the hardness decreased with an increasing vanadium content and demonstrated 4.7 GPa, 2.1 GPa, 0.9 GPa, and 1.1 GPa for samples A, B, C, and D. This represents a 55%, 81%, and 76% decrease for samples B, C, and D, respectively, compared with sample A.

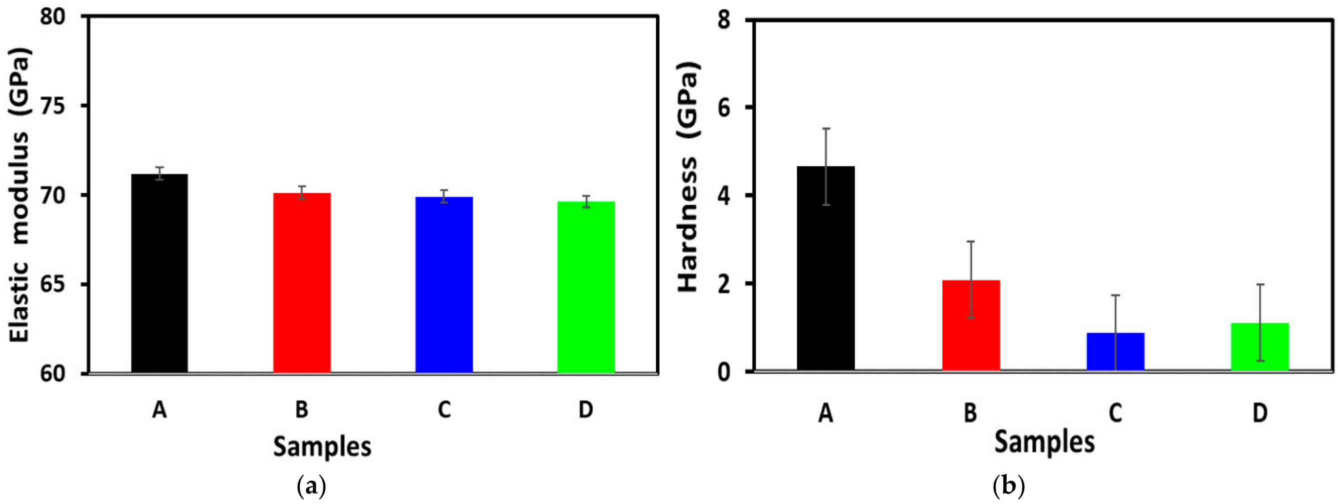

Figure 2. (**a**) The elastic modulus of glass samples and (**b**) the hardness of glass samples.

3.3. Creep *Curve*

The creep properties are crucial when they are used in structural parts. They are determined by exposing 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.

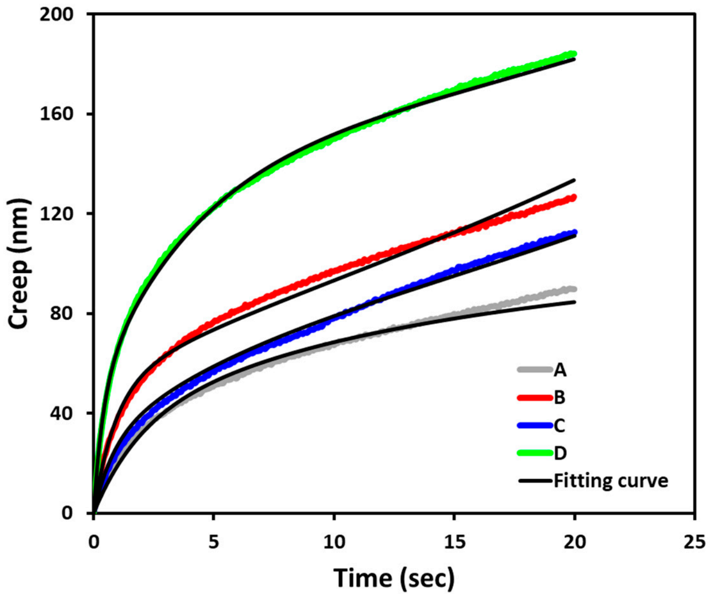

Figure 3. Creep of glass samples as a function of time.

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 [5,10]. The Maxwell–Voigt model may be used to characterize the viscoplastic strain as a function of indentation displacement, described by the following Equation:

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

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 provided by the first term of Equation (4). The second term represents the viscoplastic contribution of the creep.

As is 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. The 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. The h_1 value showed no discernible pattern, whereas h_2 rose as the vanadium content dropped. As the ratio of vanadium dropped, both τ_1 and τ_2 also decreased.

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

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) [11]:

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

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 is 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 decreases, both peaks increase in intensity and move toward a lower relaxation time. This is because the population of the relevant defects increases, making the activation of the remaining defects simpler [12].

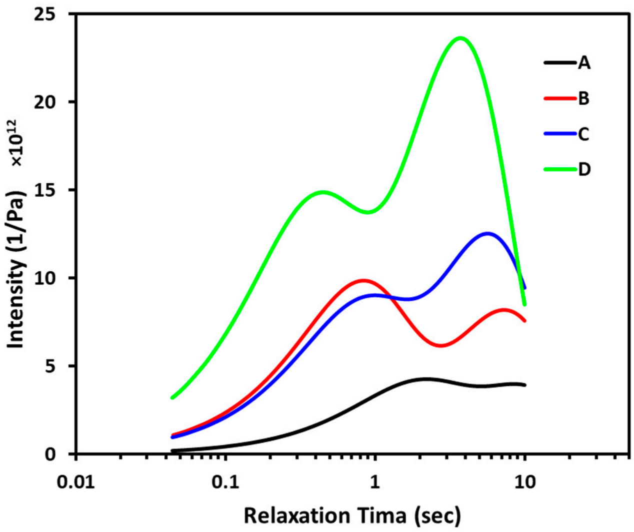

Figure 4. Spectra intensities of glass samples.

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 demonstrate reasonable elastic behavior for different applications. The elasticity of the samples increased with an 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 the vanadium oxide content. Three distinguished regions appeared during creep testing: the first region was obtained promptly after applying the load, followed by viscoelastic region characterized by a time-dependent decrease in the deformation. Finally, a pseudo-viscous region emerged, which was characterized by a constant deformation rate. The values of the fitting parameters for creep curves indicate that h_2 increases with a decrease in the vanadium oxide content and τ_1 and τ_2 decrease with this decreasing. All relaxation spectra show two peaks, indicating hard and soft region defects.

Author Contributions: Conceptualization, E.O.T. and A.S.E.-D.; methodology, M.M.A.K. and H.M.N.; software, H.M.N.; writing—original draft, A.S.E.-D. and E.O.T.; Review and editing, all authors; supervision, M.Y.H. All authors have read and agreed to the published version of the manuscript.

Funding: This research received no external funding.

Conflicts of Interest: The authors declare no conflict of interest.

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