

Elastic Studies of Cerium Doped Zinc Borotellurite Glass

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ARTICLE INFO

Article history:

Received 17 September 2025

Revised 23 December 2025

Accepted 24 January 2026

Online first

Published 01 March 2026

Keywords:

Tellurite Glass

Cerium

Elastic Properties

Zinc

Borotellurite

DOI:

10.24191/srj.v23i1.41624

ABSTRACT

Glass materials play a crucial role in modern applications due to their favorable properties relative to crystalline materials, including optical transparency, high durability, corrosion resistance, and resistance to ionizing radiation. Moreover, these properties can be further modified by doping rare-earth ions into the glass structure. In this study, cerium is chosen to be added into zinc borotellurite glasses as it can effectively reduce the effect of ionizing radiation such as gamma-ray compared to the other types of rare earth oxides. The glass samples with composition of $\{[(\text{TeO}_2)_{0.7}(\text{B}_2\text{O}_3)_{0.3}]_{0.7}[(\text{ZnO})_{0.3}]_{1-x}\{\text{CeO}_2\}_x$ (where x ranged from 0.00 to 0.05 mol) were synthesized using melt-quenching technique. The influence that cerium oxide has on the glass system was investigated by studying its elastic properties via ultrasonic pulse-echo technique. The findings revealed that all elastic moduli had an increasing trend, approximately 32-37% while the other elastic parameters possessed some variations with the dopant addition. The high elastic moduli ($L=77.59$ GPa, $G=24.16$ GPa, $E=61.55$ GPa and $K=45.39$ GPa) revealed that the insertion of CeO_2 led to the strengthening of the glass network which in turn produced glasses with higher rigidity. These findings highlight the significance of this study, demonstrating the glass's strong potential for applications in telecommunications and radiation shielding.

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1. INTRODUCTION

Today, glass is regarded as an essential material in everyday life. It differs from conventional solids in that its atomic structure is disordered, a characteristic described as amorphous (literally meaning “without form”) [1]. Owing to its excellent light-transmitting properties, glass has found extensive applications in various fields such as communications, electronics, and photonics. For instance, the development of glass-based optical fibers has transformed the telecommunications industry by replacing copper wires with fiber optics, thereby enabling the global transmission of high-quality, error-free data [2]. Not only that, glass has also been found to be useful for radiation shielding applications [3]. Its transparency, rigidity and ability to reduce or block radiation make glasses a potential substitute to replace existing lead-containing materials.

Tellurite glass has gained significant research interest because of its advantages. With its high rare-earth solubility, this glass addresses the limitations of silicate glass while its non-hygroscopic property overcomes the drawbacks of phosphate and borate glass [4 - 6]. In addition, it exhibits a high infrared transmission, low melting temperature, low phonon energy, and high refractive index [7 - 10]. However, TeO_2 glass is known to have poor glass-forming ability on its own; therefore, additional components such as B_2O_3 and ZnO are incorporated in this study to enhance its glass-forming properties. Meanwhile, CeO_2 was selected as the rare-earth compound because it has been reported to promote structural compaction at the atomic level, leading to the formation of high-density glass [11].

The elastic properties of glass are crucial as they can provide deeper understanding regarding the structure as well as the strength of the glass. According to Chang et al. [12], the elastic constants of a material are directly related to interatomic forces and the potentials in it. Generally, the increment of constants indicates that the forces between the atoms are also increase and vice versa. In the present study, the elastic properties of cerium-doped glass were studied using the non-destructive ultrasound technique. By propagating ultrasonic wave inside the samples, the information about the elastic parameter can be obtained as the wave's velocity is directly related to the elastic modulus, Poisson's ratio and density of the medium [13]. Moreover, the non-destructive nature of this testing method provides a significant advantage over other destructive techniques.

Recently, research on zinc borotellurite glass has increased considerably. Azlan et al. [14], Hajer et al. [15], Faznny et al. [16], Hazlin et al. [17] and Hasnimulyati et al. [18] has studied the optical properties of the glass using different types of rare earth oxide. Meanwhile, Eevon et al. [19] studied the elastic properties of Gd_2O_3 doped zinc borotellurite glass. Not only that, Mhareb et al. [20] also studied on the effect of gamma-ray on the structural and optical properties and durability of Nd_2O_3 doped zinc-bismuth-borotellurite glass. However, the effect on elastic properties of the glass when cerium is added has not been discovered. Hence, this research is being done to fill this gap. The addition of cerium into the glass system is expected to increase its elastic properties as CeO_2 has a high molecular weight and packing density. As a result, it will strengthen the glass network as it occupies the interstitial spaces between the atoms. This study can contribute to existing knowledge and help in the development of future advanced materials such as glass buffer coating in fiber-optic cable and radiation shielding glass for medical and nuclear industry.

2. EXPERIMENTAL METHODOLOGY

2.1 Sample Preparation

Glass samples with the composition $\{[(\text{TeO}_2)_{0.7}(\text{B}_2\text{O}_3)_{0.3}]_{0.7}(\text{ZnO})_{0.3}\}_{1-x}\{\text{CeO}_2\}_x$ (where x ranged from 0.00 to 0.05 mol) were synthesized using the conventional melt-quenching technique. In the mixture, tellurium (TeO_2) and boric oxide (B_2O_3) act as the glass former, zinc oxide (ZnO) acts as the glass modifier, while cerium oxide (CeO_2) acts as the dopant. The chemical oxides employed in this study were purchased from Alfa Aesar, with purities of 99.9%, 99.99%, 97.5%, and 99.99% for CeO_2 , ZnO , B_2O_3 , and TeO_2 , respectively. Initially, all oxides were weighed using a Dragon 204 Mettler Toledo electronic balance and thoroughly mixed and ground using agate mortar and pestle for 30 minutes to ensure homogeneity. Then, the mixture was put into an alumina crucible and preheated at 400 °C for one hour in a furnace to remove residual moisture, followed by melting at 900 °C for one hour in a second furnace. This temperature was selected as it represents the average temperature of all the combined components in the mixture. Meanwhile, a holding time of one hour was required to ensure that all components were fully melted. During the melting process, alumina dissolution is not a concern because alumina has a significantly higher melting temperature, approximately 2051°C [21]. The molten glass was subsequently poured into a stainless-steel mold that has been preheated to 400 °C and annealed at the same temperature for two hours. This process was carried out to eliminate remaining thermal stresses at a temperature corresponding to a glass viscosity of 10^{12} Pa·s [22]. Moreover, the temperature is lower than the reported glass transition temperature of cerium-doped glass [23]. The furnace was then switched off, allowing the samples to cool gradually to room temperature overnight. Finally, the glass specimens were removed, cut into slices of approximately 5 mm thickness, and polished with sandpapers of varying grades until smooth, parallel, and optically clear surfaces were obtained. The exact weight of the chemical compounds is as listed in Table 1.

Table 1. The weight of each chemical oxide used in the samples

CeO ₂ molar composition (mol)	Mass of TeO ₂ (g) ±0.0001	Mass of B ₂ O ₃ (g) ±0.0001	Mass of ZnO (g) ±0.0001	Mass of CeO ₂ (g) ±0.0001	Total Mass (g) ±0.0001
0.00	8.6715	1.6211	2.7074	0.0000	13.0000
0.01	8.5448	1.5974	2.6679	0.1900	13.0000
0.02	8.4192	1.5740	2.6287	0.3782	13.0000
0.03	8.2948	1.5507	2.5898	0.5646	13.0000
0.04	8.1716	1.5277	2.5514	0.7494	13.0000
0.05	7.9285	1.4822	2.4755	1.1138	13.0000

2.2 Ultrasonic Measurement

Ultrasonic measurement was used to investigate the elastic properties of the glass samples using pulse-echo technique. This testing was done using Ritec Ram-5000 Snap System equipped with 5 MHz Olympus Transducer and Sonotech Ultragel II as the couplant. Two types of transducers were used which are longitudinal and shear probe. The accuracy of this instrument is ± 0.0001 μs . In terms of sample size, the

specimen was cylindrical in shape with a diameter of 1 cm and thickness of 5 mm. The dimension of the sample was measured using digital vernier caliper. After the transit time was obtained, the ultrasonic velocity can be calculated using Eq. (1) [24].

$$V = \frac{2d}{t} \quad (1)$$

where V is the ultrasonic velocity, d is the thickness and t is the transit time.

3. RESULTS AND DISCUSSION

3.1 Ultrasonic Velocity and Elastic Moduli

Based on the data of ultrasonic velocity that is recorded in Table 2, it was found that the value had a generally increasing trend with a slight decrement at 0.01 and 0.04 mol of CeO_2 . The reduction of both longitudinal and shear wave velocities as the dopant was added can be attributed to the elevation of the glasses' total molecular weight. This was due to the fact that the movement of ultrasonic waves produced by the vibration of atoms in a medium. Hence, an increment in molecular weight caused the atoms to have greater inertia and make them difficult to vibrate. As a result, the acoustic velocity decreases [25]. Then, the increment of both velocities from 0.01 until 0.03 mol and at 0.05 mol of CeO_2 was due to the compaction of the glass samples. This was shown by the augmentation of density and fractal bond connectivity (d) as well as reduction of molar volume [26] as recorded in Table 2 and Table 3. As the glass became more compact, the distance between atoms in the glass network became closer. As a result, atomic vibrations can be easily propagated through the atomic network, thereby increasing ultrasonic velocities. Meanwhile, the reduction at 0.04 mol of CeO_2 was caused by the decrement in the connectivity of the glass system at this composition.

Table 2. Longitudinal wave velocity (V_L), shear wave velocity (V_S), longitudinal modulus (L), shear modulus (G), Young's modulus (E), bulk modulus (K), density (ρ) and molar volume (V_m) of $\{[(\text{TeO}_2)_{0.7}(\text{B}_2\text{O}_3)_{0.3}]_{0.7}[\text{ZnO}]_{0.3}\}_{1-x}\{\text{CeO}_2\}_x$ glass system

CeO ₂ molar composition (mol)	V_L (m/s) ±3.66	V_S (m/s) ±3.57	Elastic moduli (GPa)				ρ (kg/m ³) ±20	V_m (x10 ⁻⁵ m ³ /mol) ±0.001
			L ±0.30	G ±0.10	E ±0.94	K ±0.17		
0.00	3976.79	2242.08	57.41	18.25	46.24	33.08	3630	3.351
0.01	3954.60	2236.24	71.91	22.99	58.17	41.25	4598	2.562
0.02	3957.06	2240.41	72.29	23.17	58.58	41.39	4617	2.563
0.03	3999.52	2266.96	74.27	23.86	60.29	42.25	4643	2.561
0.04	3987.46	2251.48	74.24	23.67	59.93	42.68	4669	2.558
0.05	4066.57	2268.91	77.59	24.16	61.55	45.39	4692	2.557

Based on the published density [26] and ultrasonic wave velocity data, the glasses' elastic moduli can be calculated using Eq. (2) – (5) as listed below [27, 28].

$$L = V_L^2 \rho \quad (2)$$

$$G = V_s^2 \rho \quad (3)$$

$$K = L - \left(\frac{4}{3}\right)G \quad (4)$$

$$E = (1 + \sigma)2G \quad (5)$$

where L is the longitudinal modulus, V_L is the longitudinal wave velocity, ρ is the density, G is the shear modulus, V_s is the shear wave velocity, K is the bulk modulus, E is the Young's modulus and σ is the Poisson's ratio. All the data are as recorded in Table 2 and shown in Fig. 1 and Fig. 2..

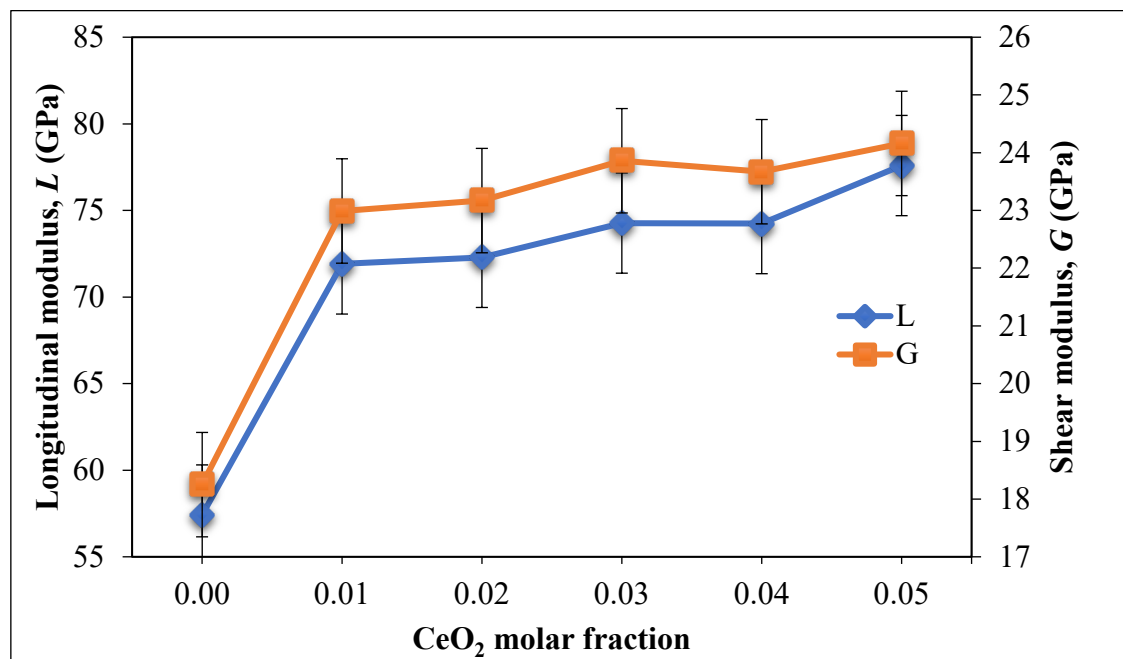


Fig. 1. Longitudinal and shear modulus of $\{[(\text{TeO}_2)_{0.7}(\text{B}_2\text{O}_3)_{0.3}]_{0.7}[\text{ZnO}]_{0.3}\}_{1-x}\{\text{CeO}_2\}_x$ glass system

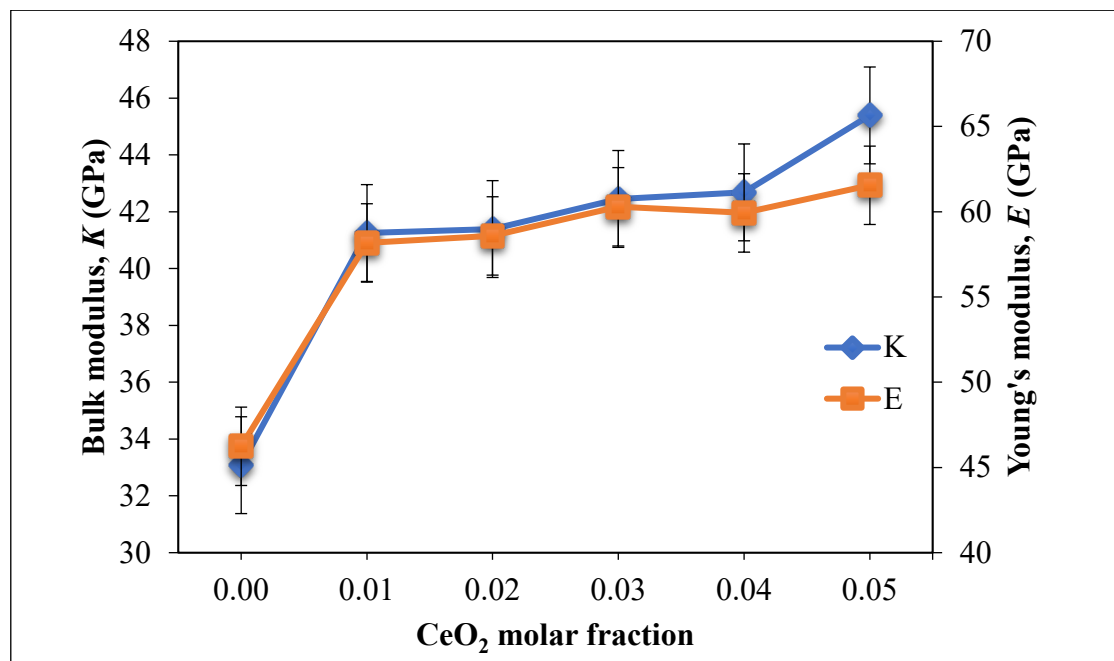


Fig. 2. Bulk and Young's modulus of $\{[(\text{TeO}_2)_{0.7}(\text{B}_2\text{O}_3)_{0.3}]_{0.7}[\text{ZnO}]_{0.3}\}_{1-x}\{\text{CeO}_2\}_x$ glass system

In the present study, it was found that all the elastic moduli possessed an increasing trend with the addition of cerium oxide despite the slight variation in the ultrasonic velocity. This indicates an elevation in the rigidity of the glass samples and the strengthening of their network [29, 30]. The reasons that might contribute to this increment were the rises of packing density and connectivity of the glasses. Packing density of the glass increased due to the insertion of CeO_2 which has a higher packing density ($16.6 \text{ cm}^3/\text{mol}$) than the other chemical oxides ($\text{TeO}_2 = 14.7 \text{ cm}^3/\text{mol}$; $\text{B}_2\text{O}_3 = 15.2 \text{ cm}^3/\text{mol}$; $\text{ZnO} = 8.0 \text{ cm}^3/\text{mol}$) [31] that were used in the study. As a result, the total packing density of the glass was increased which led to the strengthening of the samples. Moreover, the changes in the coordination number of tellurite and borate structural units can also result in the increment of packing density [32]. This is referring to the conversions of TeO_3 into TeO_4 and BO_3 into BO_4 , which causes the BO/NBO ratio to be increased especially at 0.01 mol of CeO_2 as shown in Table 3. This increase in connectivity is attributed to the higher coordination number of cerium oxide. As the connectivity of the glass network increased, it showed that the atoms were more bonded together and this enabled them to resist deformation better. After the value for each elastic modulus was compared to one another, the result has shown that the longitudinal modulus gave the highest value. This signified that the glass samples can stand force in one direction better than forces that acted in various directions. In addition, longitudinal deformation is associated with direct stretching or compression of interatomic bonds, while shear deformation is mainly accommodated through changes in bond angles, which generally require lower energy.

3.2 Poisson's Ratio, Microhardness, Debye Temperature, Mean Ultrasonic Velocity, Acoustic Impedance and Softening Temperature

Other elastic parameters that can be calculated are Poisson's ratio, microhardness, Debye temperature, mean ultrasonic velocity, acoustic impedance and softening temperature (Table 3). These parameters are calculated based on Eq. (6) – (13) as shown below [28, 32 – 36].

$$\sigma = \left[\frac{(L - 2G)}{2(L - G)} \right] \quad (6)$$

$$H = \frac{(1 - 2\sigma)E}{6(1 + \sigma)} \quad (7)$$

$$\theta_D = \frac{h}{k} \left(\frac{3N_A P}{4\pi m_v} \right)^{\frac{1}{3}} V_{mean} \quad (8)$$

$$V_{mean} = \left[\frac{1}{3} \left(\frac{2}{V_s^3} + \frac{1}{V_L^3} \right) \right]^{-\frac{1}{3}} \quad (9)$$

$$P = \sum_i x_i (m + n)_i \quad (10)$$

$$Z = V_L \rho \quad (11)$$

$$T_s = \frac{v_s^2 M_T}{c^2 P} \quad (12)$$

$$d = \frac{4G}{K_e} \quad (13)$$

where H is the microhardness, θ_D is the Debye temperature, h is the Planck's constant, k is the Boltzmann constant, N_A is the Avogadro constant, V_{mean} is the mean velocity, P is the number of atoms per unit formula, x is the molar fraction, m and n are the number of cation and anion in A_mO_n oxide, Z is the acoustic impedance, T_s is the softening temperature, M_T is the total molecular weight, c is constant which equal to $0.5074 \times 10^5 \text{ cm/s K}^{1/2}$ and d is the fractal bond connectivity.

Table 3. Poisson's ratio (σ), microhardness (H), Debye temperature (θ_D), mean ultrasonic velocity (V_{mean}), acoustic impedance (Z), softening temperature (T_s) and fractal bond connectivity (d) of $\{[(\text{TeO}_2)_{0.7}(\text{B}_2\text{O}_3)_{0.3}]_{0.7}[\text{ZnO}]_{0.3}\}_{1-x}\{\text{CeO}_2\}_x$ glass system.

CeO₂ molar composition (mol)	σ ± 0.0022	H (GPa) ± 0.031	θ_D (°C) ± 0.67	V_{mean} (m/s) ± 0.04	Z (kg/m²s) ± 0.009	T_s (K) ± 7.11	d ± 0.047	BO/ NBO ratio ± 0.01
0.00	0.2670	2.834	284.23	2494.16	1.444	733.70	2.207	0.19
0.01	0.2650	3.603	309.89	2487.06	1.818	733.59	2.229	4.57
0.02	0.2641	3.644	310.33	2491.44	1.827	740.04	2.239	4.32
0.03	0.2633	3.765	314.05	2520.72	1.857	761.49	2.248	2.47
0.04	0.2660	3.693	312.07	2504.31	1.862	754.89	2.218	3.95
0.05	0.2740	3.640	314.79	2526.15	1.908	770.44	2.129	3.45

Poisson's ratio (σ) is defined as the ratio of lateral to longitudinal strain of a sample when subjected to compression or elongation. In the atomic level, this parameter is commonly related to cross-link density and the dimensionality of a glass network. The σ value of a 3-dimensional structure is lower than 2 and 1-dimensional due to the higher number of bonds or cross-link density in the lateral direction [37]. According to Saddeek et al. [38], the dimensionality of glass network can be studied using the ' d ratio' which is the fractal bond connectivity. For a 3D network, the d ratio will be equal to 3 while a 2D and 1D network will has a d ratio of 2 and 1 respectively [38, 39]. However, this value is not a direct structural measurement and with an assumption that the glass structure is homogeneous and isotropic in nature. In the present study, the d ratio was in the range of 2.207 to 2.248 as shown in Table 3. Hence, it can be concluded that all the glass samples had a 2D dimensional structure. This is also in agreement with studies that have been reported previously by Halimah et al. [39] and Cheong et al. [40]. Furthermore, the increment of d ratio from 0.00 until 0.03 mol indicates the elevation in the number of bonds which in turn led to the reduction of Poisson's ratio at this composition. The value of σ which had a minimum value of 0.2633 and a maximum value of 0.2740 also meant that the glasses possessed a low cross-link density [41, 42]. In addition, when the glasses were subjected to stress, the glass network was distorted and the ions themselves were also deformed [12].

The parameter that indicates the amount of stress required to eliminate free volume in the glass network is the microhardness (H). Using Eq. (7), the parameter can be calculated based on the data of Young's modulus (E) and Poisson's ratio (σ). In the present study, it was found that the value of H increased until 0.03 mol of CeO₂ and decreased beyond that. The elevation of microhardness confirmed the increment in the rigidity of the glass system as also shown by the inclining trend of the elastic moduli. Moreover, the augmentation in the packing density of the glass also could contribute to the increment of H . As explained in the elastic moduli section, the incorporation of cerium oxide with higher packing density compared to the other chemical oxides caused the glass network to become more compact and rigid. As a result, a higher stress was needed to eliminate the free volume that was available in the glass system. On the other hand, the decrement of H at 0.04 and 0.05 mol of cerium oxide can be related to the reduction of average stretching force constant of the glasses. This was due to the fact that CeO₂ has a lower first order stretching force constant (111.45 N/m) [43] compared to the other oxides (TeO₂ = 219.00 N/m; B₂O₃ = 668.42 N/m;

$\text{ZnO} = 216.37 \text{ N/m}$ [44] used in the study. Hence, the strength of the network bond declined, and the stress needed to deform the structure was also diminished.

Another parameter that can also be calculated is the Debye temperature (θ_D). θ_D is directly related to atomic vibrations where it is defined as the temperature when all modes of vibrations in the network are excited [45]. Based on the data recorded in Table 3, the value of Debye temperature had an increasing trend with the insertion of CeO_2 into the glass system. The factors that might lead to this elevation were the number of atoms per unit volume (P/m_v), mean ultrasonic wave velocity (V_{mean}) and also the atomic ring size of the glass network [46 - 48]. As cerium oxide with eight coordination number was added into the glass network, the ratio P/m_v increased which in turn caused compaction to occur in the structure. This compaction then caused the atoms to require a higher amount of energy to be able to vibrate and resulted in a higher θ_D . The relation of V_{mean} to the variation of Debye temperature was reported by El-Aal et al. [47]. It was stated that the rise of θ_D implies the strengthening of the glass network and can be attributed to the increment of ultrasonic wave velocity. The reasons responsible for the increment were the augmentation of number of bond per unit volume as well as the network connectivity. In addition, the creation of a smaller ring size as cerium oxide is incorporated into the glass system might also results in the glasses' compaction and thus, higher Debye temperature.

Acoustic impedance (Z) gives information regarding the transmission and reflection of sound energy in the glass specimen [32] while softening temperature (T_s) sheds some light on the temperature when the glass starts to soften after being exposed to heat. In the present study, Z had an increasing trend with the addition of CeO_2 . This suggested that the glass became more rigid and increased in its connectivity. Moreover, the densification of the glass could also lead to the elevation of acoustic impedance. This is due to the fact that a denser substance has a higher molecular weight. Hence, the atoms will have greater inertia and higher resistance in changing their atomic motion. As a result, this caused greater impedance in the propagation of ultrasonic wave in the prepared glass. In terms of softening temperature, the data also showed an increasing trend but had a slight decrement at 0.01 and 0.04 mol of cerium oxide. This inclining trend was expected as the elastic moduli and microhardness of the glasses were increasing. Kannappan et al. [32] mentioned that the elevation of H will strengthen the softening points in a glass, hence causing the T_s to be increased as well. Meanwhile, the small reduction of softening temperature might be related to the creation of non-bridging oxygens (NBOs) in the glass system. Furthermore, the weakening of bonds inside the network structure revealed by the reduction of the average stretching force constant could also lead to the decrement of softening temperature.

4. CONCLUSIONS

$\{[(\text{TeO}_2)_{0.7}(\text{B}_2\text{O}_3)_{0.3}]_{0.7}[\text{ZnO}]_{0.3}\}_{1-x}\{\text{CeO}_2\}_x$ glass system was successfully prepared using melt-quenching technique with x is varies from 0.00 until 0.05 mol. The elastic properties of the glass were investigated via a non-destructive pulse-echo technique. From the findings, it is revealed that the ultrasonic wave velocities, Poisson's ratio, microhardness, Debye temperature and softening temperature have some variation as CeO_2 is inserted into the glass system. The variation can be attributed to the elevation of total molecular weight, densification of the glass, as well as the formation of bridging oxygen. Despite the slight fluctuation, the elastic moduli and acoustic impedance possess an increasing trend. The elastic moduli increased approximately 25-37% with CeO_2 addition. Hence, it can be said that even though there is some resistance in the flow of ultrasonic waves in the glass samples, it was not caused by the weakening of the network

structure, but instead by the compaction and strengthening of the glass network. In summary, it can be concluded that the incorporation of cerium oxide results in the changes of the network structure which in turn produces glass with higher rigidity and stability.

5. ACKNOWLEDGMENTS/FUNDING

The author gratefully acknowledged the financial support and laboratory equipment provided by Universiti Putra Malaysia, Universiti Pertahanan Nasional Malaysia with Short Term Grant UPNM/2022/GPJP/SG/3 and Ministry of Higher Education (MOHE) Malaysia.

6. CONFLICT OF INTEREST STATEMENT

The authors agree that this research was conducted in the absence of any self-benefits, commercial or financial conflicts and declare the absence of conflicting interests with the funders.

7. AUTHORS' CONTRIBUTIONS

Hasnimulyati Laoding: Conceptualisation, methodology, formal analysis, investigation and writing-original draft; Azuraida Amat: Conceptualisation, methodology, writing-review and editing; Siti Nasuha Mohd Rafien: Writing-review and editing; Azhan Hashim @ Ismail: Writing-review and editing; Nur Baizura Mohamed: Writing-review and editing; Ishak Mansor: Supervision.

8. DECLARATION OF GENERATIVE AI IN THE WRITING PROCESS

During the preparation of this work, the author(s) used CHATGPT in order to improve the readability and language quality of the work. After using this tool/service, the author(s) reviewed and edited the content as needed and take(s) full responsibility for the content of the publication.

9. DATA AVAILABILITY/SUPPLEMENTARY MATERIALS

The datasets used and/or analysed during the current study are available from the corresponding author on reasonable request.

10. ETHICS STATEMENT

The authors declare that this research did not involve human or animal subjects. All experimental procedures were performed following the institutional Safety, Health, and Environmental (HSE) protocols of Universiti Teknologi MARA.

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