การศึกษาสมบัติความยืดหยุ่นของกระจกหน้าต่างรีไซเคิลที่เจือด้วยคอปเปอร์(I)ออกไซด์ (Cu_2O) ด้วยเทคนิคคลื่นเสียงอัลตร้าโซนิกและฟูเรียร์ทรานสฟอร์มอินฟราเรดสเปกโทรสโกปี

Elastic Properties of Recycled Soda-lime Glasses Doped with Copper(I)oxide (Cu_2O) Studied by Ultrasonic Technique and Fourier Transform Infrared Spectroscopy

Y. Jaichueai, C. Bootjomchai[1], O. jaiboon[1], J. Laopaiboon[1] and R. Laopaiboon[1*]

[1]Department of Physics, Faculty of Science, Ubon Ratchatani University, 34190 Thailand

*Email: Raewatl@yahoo.com

บทคัดย่อ

วัตถุประสงค์ของงานวิจัยคือนี้ศึกษาโครงสร้างของกระจกหน้าต่างรีไซเคิลด้วยเทคนิคคลื่นเสียงอัลตร้าโซนิกและฟูเรียร์ทรานสฟอร์มอินฟราเรดสเปกโทรสโคปี ตัวอย่างแก้วจะถูกเตรียมในระบบ 90Recycled window glass – $10Na_2O$ – xCu_2O เมื่อ x คือ 0.001, 0.01, 0.1 และ 1.0 เปอร์เซ็นต์โดยโมล โดยหลอมที่อุณหภูมิ 1250 องศาเซลเซียส เป็นเวลา 4 ชั่วโมงและอบที่อุณหภูมิ 500 องศาเซลเซียส เป็นเวลา 2 ชั่วโมง จากนั้นวัดความหนาแน่น ความเร็วคลื่นเสียงตามยาวและความเร็วคลื่นเสียงเฉือนของตัวอย่างแก้วด้วยเทคนิคคลื่นเสียงอัลตร้าโซนิก โดยใช้หัวตรวจสอบความถี่ 4 เมกะเฮิร์ตซ์ ที่อุณหภูมิห้อง ความหนาแน่นและความเร็วคลื่นเสียงของตัวอย่างแก้วสามารถนำมาคำนวณสมบัติความยืดหยุ่นของตัวอย่างแก้วได้เช่น โมดูลัสตามยาว โมดูลัสเฉือน บัลก์โมดูลัส ยังโมดูลัส อัตราส่วนปัวซอง และความแข็งระดับไมโคร รวมไปถึงสมบัติทางกายภาพ เช่น ความต้านทานคลื่นเสียง อุณหภูมิการอ่อนตัว และอุณหภูมิเดอบาย สุดท้ายศึกษาโครงสร้างของตัวอย่างแก้วด้วยเทคนิคฟูเรียร์ทรานสฟอร์มอินฟราเรดสเปกโทรสโกปีในช่วงเลขคลื่นที่ 400 - 2000 ซม$^{-1}$ ผลที่ได้จากการศึกษาประกอบด้วยความเร็วคลื่นเสียงอัลตร้าโซนิก โมดูลัสความยืดหยุ่น อัตราส่วนปัวซอง ความแข็งระดับไมโคร ความต้านทานคลื่นเสียง อุณหภูมิการอ่อนตัว และอุณหภูมิเดอบายจะขึ้นอยู่กับปริมาณ Cu_2O ที่เจือและยืนยันผลด้วยฟูเรียร์ทรานสฟอร์มอินฟราเรดสเปกโทรสโกปี

คำสำคัญ: กระจกหน้าต่างรีไซเคิล เทคนิคคลื่นเสียงอัลตร้าโซนิก สมบัติความยืดหยุ่น ฟูเรียร์ทรานสฟอร์มอินฟราเรดสเปกโทรสโกปี

Abstract

The purpose of this work was to study the elastic moduli and structural properties of recycled window glass using ultrasonic technique and Fourier transform infrared spectroscopy. The glass system of 90Recycled window glass – $10Na_2O$ – xCu_2O (where x is 0.001, 0.01, 0.1 and 1.0 mol%) were prepared by melt quenching technique at the melting temperature of 1250°C for 4 h and annealing temperature of 500°C for 2 h. Then, densities of glass sample with varying the Cu_2O dopant were measured. The longitudinal and shear velocities were measured by using ultrasonic technique at 4 MHz frequency at room temperature. Next the value of densities and the ultrasonic velocities data of glass samples were used to calculate the elastic properties such as longitudinal modulus, shear modulus, bulk modulus, Young's modulus, Poisson's ratio and micro-hardness as well as physical properties such as acoustic impedance, softening temperature and Debye temperature. Finally, Fourier transform infrared spectroscopy was measured in wave number range 400 - 2000 cm^{-1} to study structure of the glass samples. The results indicated that the ultrasonic velocity, elastic moduli, Poisson's ratio, micro-hardness, acoustic impedance, softening temperature and Debye temperature depended on the concentration of Cu_2O dopant and could be confirmed by Fourier transform infrared spectroscopy results.

Keywords: Recycled window glasses; Ultrasonic technique; Elastic moduli; Fourier transform infrared spectroscopy

Introduction

Nowadays glasses materials have been used extensively such as decorative window glass, glassware in the laboratory, optical lens, etc. Glass materials are interesting because it's insulator and transparent to visible light. Moreover, Soda-lime glass is relatively inexpensive, chemically stable, reasonably hard, and extremely workable. Because it can be resoftened and remelted numerous times, it is ideal for glass recycling. The composition of soda-lime glass is normally 60-70 % of silica (SiO_2), 12-15 % of soda (Na_2O) and 5-12 % of lime (CaO). The soda serves as a flux to lower the melting point of silica, and the lime acts as a stabilizer for the silica. Soda-lime glasses have lower melting point and higher coefficient of expansion. In addition, glasses have high strength and are corrosion resistance to chemicals. It also can be blown into an infinite number of shapes [1-3]. Moreover, it can be colored by impurities such as some transition metal oxides (TMOs). Transition metal impurities such as TiO_2, ZnO_2, MnO_2, NiO, V_2O_5 have an influence on structural properties, physical properties and elastic moduli [4-8]. Copper containing glasses have received significant interest for potential scientific and industrial application. The mechanic, electronic, optical properties of glass doped with copper oxide are depend on the concentration of copper ions in glass system [9-11].

The present study aims to investigate the effect of copper oxide on structural properties and elastic moduli including longitudinal modulus, shear modulus, bulk modulus, Young's modulus, Poisson's ratio and micro-hardness as well as acoustic impedance, softening temperature and Debye temperature. The network bonds will also be examined by FTIR spectroscopy.

Experimental and theoretical techniques

1. Preparation of the glass samples

Glass samples from the glass system of 90Recycled window glass – $10Na_2O$ – xCu_2O (where x is 0.001, 0.01, 0.1 and 1.0 mol %) were prepared by the melt quenching technique. The chemical composition analyses of the glass system are shown in **Table1.** The chemical composition of glass samples were weighed using a digital balance ($\pm$ 0.0001 g). The homogeneous mixtures were placed in ceramic crucibles and melted in electric furnace at the temperature 1250°C for 4 h. The molten glass was poured into stainless steel mould and annealed at 500°C for 2 h. Bulk glass were cut and polished using different silicon carbide grades in order to obtain a highly flatness for ultrasonic measurement.

Table 1. Quantitative analysis of chemical composition of recycles window glasses (RWG) by using WDXRF technique

Chemical composition	Concentration (%)
Na_2O	15.1700
MgO	3.4080
SiO_2	73.3900
Al_2O_3	0.5866
CaO	7.3390
Fe_2O_3	0.0635
TiO_2	0.0263
K_2O	0.0238

2. Density and molar volume measurements

The densities of all glass samples were determined by Archimedes' principle using n-hexane as an immersion liquid and applying the relation [12]

$$\rho = \rho_l \left(\frac{W_a}{W_a - W_b}\right) \tag{1}$$

where ρ_l is the density of n-hexane (0.661 g/cm^3), W_a and W_b are the glass sample weights in air and in n-hexane, respectively. The molar volume (V_a) of the glass samples was calculated using the relation [13]

$$V_a = \frac{M}{\rho} \tag{2}$$

where M is molecular weight of glass systems. ρ is densities of glass sample. x_i is the mole fraction of the component oxide i and M_i is the molecular weight and can be calculated according to the relation [14]

$$M = \sum x_i M_i \tag{3}$$

The glass packing density can be calculated using the relation [15]

$$V_t = \frac{\rho}{M} \sum x_i V_i \tag{4}$$

where V_i is given by,

$$V_i = \frac{4\pi N_A}{3}\left(x r_M^3 + y r_O^3\right) \tag{5}$$

where N_A is Avogadro's number, r_M and r_O are the ionic radii of the cation and anion of the oxide M_xO_y, respectively. The estimated error in molar volume and packing density of glass samples were $\pm$ 0.006 cm^3.mol^{-1} and $\pm$ 0.044 × 10^{-6} m^3, respectively.

3. Ultrasonic velocity measurement

Ultrasonic flaw detector, SONATEST Sitescan 230 was used to measure ultrasonic wave velocity at room temperature. The ultrasonic wave is generated from a ceramic transducer with a resonant frequency at 4 MHz and this transducer also acts as a transmitter-receiver at the same time. The ultrasonic velocity can be calculated by the relation [16]

$$v = \frac{2x}{\Delta t} \tag{6}$$

where x is the glass sample thickness and Δt is the time interval. Longitudinal and shear ultrasonic velocities were measured three times to check the reproducibility of the data. The estimated error of longitudinal and shear velocities measurement were $\pm$ 11 m.s^{-1} and $\pm$ 8 m.s^{-1}, respectively.

4. Determination of elastic moduli

The elastic moduli including longitudinal modulus (L), Shear modulus (G), bulk modulus (K), Young's modulus (E), Poisson's ratio (σ) and micro-hardness (H) as well as acoustic impedance (Z), softening temperature (T_s) and Debye temperature (θ_D) of glass samples have been calculated from the measured ultrasonic velocities and densities using the relations [17]

Longitudinal modulus:

$$L = \rho V_L^2 \tag{7}$$

Shear modulus:

$$G = \rho V_S^2 \tag{8}$$

Bulk modulus:

$$K = L - \frac{4}{3}G \tag{9}$$

Young's modulus:

$$E = (1+\sigma)2G \tag{10}$$

Poisson's ratio:

$$\sigma = \frac{L-2G}{2(L-G)} \tag{11}$$

Micro-hardness:

$$H = \frac{(1-2\sigma)E}{6(1+\sigma)} \tag{12}$$

Acoustic impedance:

$$Z = \rho V_m \tag{13}$$

Softening temperature:

$$T_s = \frac{V_S M}{C^2 \psi} \tag{14}$$

Debye temperature:

$$\theta_D = \left(\frac{h}{k_B}\right)\left(\frac{3\psi N_A}{4\pi V_a}\right)^{1/3} \tag{15}$$

where Z is acoustic impedance, *h* is Planck's constant, k_B is Boltzmann's constant, N_A is Avogadro's number, ψ is the number of atoms in the chemical formula, *C* is the constant of proportionality (507.4 $ms^{-1} K^{1/2}$) and V_m is the mean ultrasonic velocity defined by the relationship [12]

$$V_m = \left[\frac{3V_l^3 V_S^3}{V_l^3 + V_S^3}\right]^{1/3} \tag{16}$$

where v_l and v_S is longitudinal velocities and shear velocities, respectively.

5. Infrared absorption measurements

The glass samples were measured by a Fourier Transform infrared spectrometer. The powdered glass samples were mixed with KBr in the ratio 1:100 glass powder: KBr and then the sample were measured by Fourier transform infrared spectrometer at room temperature in the wavelength range 400–2000 cm^{-1}.

Table 2. Chemical composition, density and molar volume of glass system

Chemical composition (mol%)				ρ (g/cm^3)	V_a (cm^3/mol)
	RWG	Na_2O	Cu_2O	$\pm$ 0.0004	$\pm$ 0.006
S1	90	10	-	2.5525	23.487
S2	90	10	0.001	2.5678	23.347
S3	90	10	0.01	2.5695	23.337
S4	90	10	0.1	2.5712	23.371
S5	90	10	1.0	2.6085	23.531

Results and Discussion

1. Density and Molar volume

The chemical composition of glass, density and molar volume was shown in **Table 2** and shown in **Figure 1.** It can be seen that the density of glass samples are increase from 2.5525 to 2.6085 g.cm^{-3} with the increasing of Cu_2O in glass system, respectively. This can be attributed to the higher molecular weight of Cu_2O (143.08 g.mol^{-1}) than silica (60.09 g.mol^{-1}). The molar volume is defined as the volume occupied by the unit mass of the glass and can be used as a parameter to identity an open structure [18]. Initially, it can be seen that the molar volume was decrease with 0.000 – 0.001 mol% of Cu_2O due to the filling of Cu_2O dopant in the interstices of the glass network. Then, the increase in molar volume with 0.001 mol% of Cu_2O dopant lead to the breakdown in the ring type structure of the glass samples [19].

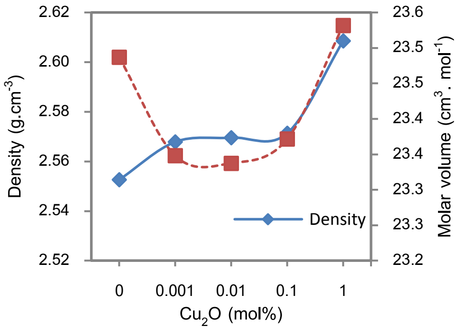

Figure 1. Variation of density and molar volume as a function of mol% of Cu_2O in the glass system

2. Ultrasonic velocity and elastic moduli

The longitudinal and shear ultrasonic velocity as a function of mol% of Cu_2O are shown in **Figure 2.** The velocities (v_l and v_s) increase with the increasing of Cu_2O dopant. The increases in ultrasonic velocities are related to the decrease in the number of non-bridging oxygen (NBO) and consequently the increase in connectivity of the glass network [20]. Therefore, ultrasonic velocity is a tool in revealing the degree of change in structure with composition of glasses. The value of Young's modulus (E) and bulk modulus (K) are shown in **Figure 3.**

Table 3 Longitudinal modulus (L), Shear modulus (G), Young's modulus (E) bulk modulus (K) and acoustic impedance (Z) of all glass samples

Sample	L(GPa) $\pm$ 0.112	G(GPa) $\pm$ 0.034	E (GPa) $\pm$ 0.048	K(GPa) $\pm$ 0.125	Z (10^{-6}kg.m^{-2}s^{-1}) $\pm$ 0.009
S1	86.510	29.860	73.756	46.697	11.839
S2	87.397	31.312	76.069	45.647	12.121
S3	88.450	31.955	77.615	45.843	12.240
S4	88.893	32.089	78.026	46.107	12.270
S5	90.510	33.246	80.554	46.181	12.560

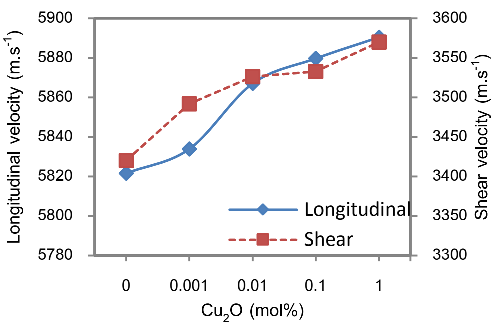

Figure 2 Variation of longitudinal and shear velocity as a function of mol% of Cu_2O in the glass system

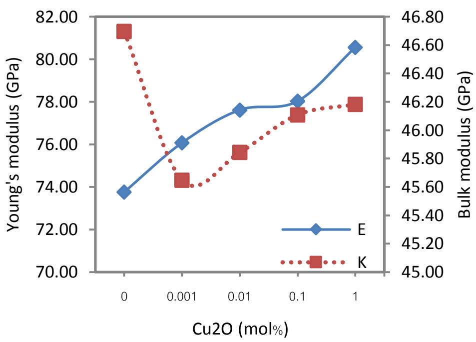

Figure 3 Variation of Young's modulus and bulk modulus as a function of mol% of Cu_2O in the glass system

It can be seen that the elastic moduli (longitudinal modulus, shear modulus and Young's modulus) increased with the increasing of concentration of Cu_2O dopant in glass system. This increase in elastic moduli reveals the decrease in number of non-bridging oxygen (NBO) in the glass network [20]. The value of Young's modulus increase from 73.756 up to 80.554 GPa. This increasing of Young's modulus is also related to the decrease in number of non-bridging oxygen (NBO) and lead to the increase in rigidity of glass system. The behavior of bulk modulus is associated with the change in the cross-linkage coordination of the glass network [22]. Therefore, the decrease in bulk modulus (0.00 – 0.001 mol% of Cu_2O dopant) indicate more formation of number of non-bridging oxygen and this lead to the decreasing in the cross-linkage and coordination of glass network. When the concentration of Cu_2O dopant increase from 0.01 mol% to 1.0 mol%, bulk modulus increase with the concentration of Cu_2O dopant in glass system. The value of Poisson's ratio was shown in **Figure 4.** Poisson's ratio is the measure of the cross-link density of the structure and reveals a cross-link density in glass structure. It was mentioned that the range of Poisson's ratio 0.1 to 0.2 is shown a high cross-link density while 0.3 to 0.5 is a low cross-link density [23].

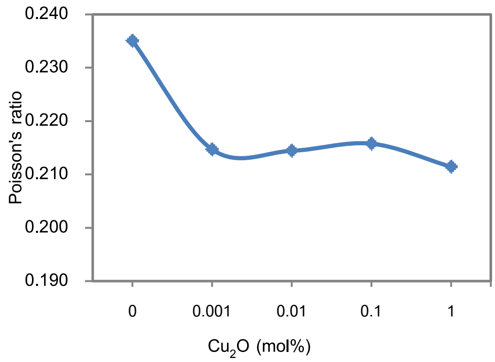

Figure 4. Variation of Poisson's ratio as a function of mol% of Cu_2O in the glass system

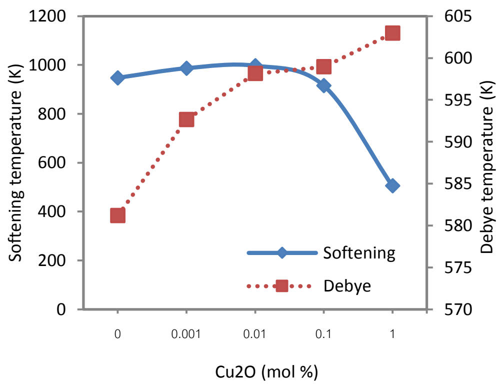

Figure 5. Variation of softening temperature and Debye temperature as a function of mol% of Cu_2O in the glass system

The decrease in Poisson's ratio with increasing mol% of Cu_2O dopant indicate that the increase in cross-link density of glass network and leads to the decrease in number of non-bridging oxygen [24]. **Figure 5** show the variation of softening temperature and Debye temperature with the concentration of Cu_2O dopant in glass system. Debye temperature (θ_D) is one of important parameter for materials and use to determine the elastic moduli and atomic vibrations. Softening temperature (T_s) is maximum temperature of glass before it permanently deforms. It can be seen that

Debye temperature increase from 581 K to 602.9 K and softening temperature decrease from 947 K to 505 K with increasing Cu_2O dopant. The increase in Debye temperature indicate that the increase in the rigidity of glass network and related to an increase in the ultrasonic velocities. Moreover, the decrease in softening temperature and increase in Debye temperature are related to the number of non-bridging oxygen in the glass network. The value of micro-hardness was shown in **Figure 6.** The micro-hardness are indicates the rigidity of glass network structure and this micro-hardness relate to Poisson's ratio and Young's modulus. The increase in micro-hardness from 5.27 to 6.39 GPa lead to the increase of resistance to downforce of glass system.

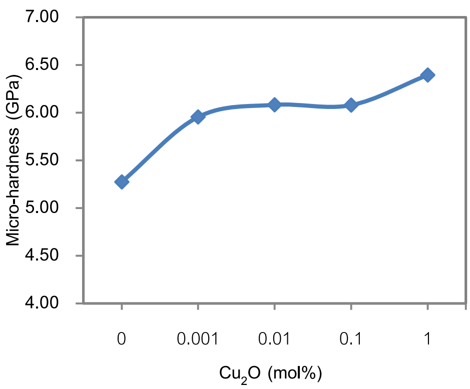

Figure 6 Variation of micro-hardness as a function of mol% of Cu_2O in the glass system

3. Fourier transforms infrared absorption

The FTIR spectra for the glass samples with the concentration of Cu_2O dopant (0.001, 0.01, 0.1 and 1.0 mol%) are shown in **Figure 7.** The glass samples were measured at room temperature in the wave number range 400–2000 cm^{-1}. The frequency bands from the glasses network vibrations appear in the range 400–1500 cm^{-1}. The vibration signals of FTIR spectra for the glass samples are related to network bonds in glass structure. The first vibration signals at around 470 cm^{-1} are assigned to the Si–O–Si bending modes of bridging oxygen. The peaks near 775-800 cm^{-1} are assigned to the vibrations of O-Si-O bonds. The peak at 960 cm^{-1} is assigned to the vibrations of non-bridging oxygen. The peak in the region range of 1050-1120 cm^{-1} is assigned to Si–O–Si anti symmetric stretching of Si-O-Si. The signal at approximately 1600 cm^{-1} is assigned to water molecular [26]. Increasing concentration of Cu_2O reveal slight the increase of the absorption bands in FTIR spectra. Moreover, the results from FTIR spectra confirm our conclusion from the values of elastic moduli studies.

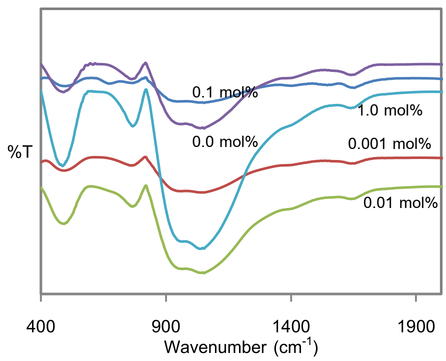

Figure 7 The IR spectra of the glass samples with vary concentration of Cu_2O in the glass system

Table 3 Depicts the detailed assignments of IR bands in sodium silicate glasses

Peak position (cm^{-1})	Assignment
460-480	Bending vibration of Si-O-Si Linkages [29]
640-680	Si-O-Si and O-Si-O bending [29]
775-800	Symmetric stretching vibrations of O-Si-O bonds [29,30]
960	Vibrations of non-bridging oxygen(NBO) [29]
1050–1120	Anti symmetric stretching of Si-O-Si linkages [29,30]
1400–1460	Carbonate group [29,30]
1630–1645	Molecular water [29,30]

Conclusions

Results of ultrasonic velocity in glass system indicate the formation of bridging oxygens with the increase of Cu_2O concentration in glass system. The elastic moduli are related to change in the structure of the glass. The glass structures are rigid at the higher values of Cu_2O concentration in glass system. The FTIR spectra also support the structural changes of glass system.

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