

CHARACTERIZATION OF HOLMIUM IN TELLURITE GLASS

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ABSTRACT

A series of holmium tellurite glasses $(\text{Ho}_2\text{O}_3)_x(\text{TeO}_2)_{100-x}$ were prepared by using normal quenching method. Holmium (III) oxide add as network modifier in the tellurite base glass and hence its effect the structural of the tellurite glass. The density and refractive index of this glass system increase when the holmium content increase. The molar volume of holmium tellurite glass decrease as holmium content increase. This is because of non bridging oxygen (NBO) was formed in the glass network. The XRD pattern show the structure of the glass sample and the result of the FTIR shows the bonding of the glass sample formed.

Keywords: Holmium (III) oxide; tellurite glass

INTRODUCTION

Tellurite glasses are interest for many researcher due to the high refractive index, high third-order nonlinear optical and low photon energies [1]. This tellurite glasses can use as a fiber optic material. Nowadays, rare earth ions such as Er^{3+} , Tm^{3+} and Ho^{3+} with certain glasses convert IR to ultraviolet or visible light through upconversion process [2]. There are not much information on the characteristic of holmium tellurite based glass with other properties. In this research will concentrate on the physical properties analysis of this binary holmium tellurite glasses. The density, molar volume, refractive index, FTIR and XRD for holmium tellurite glasses were determined.

MATERIALS & METHODS

Starting material tellurite (II) oxide, TeO_2 (Aldrich 99.5%) and holmium (III) oxide (Aldrich 99.0%) were used for synthesize the glass sample. A series of the glass sample were prepared by using normal melt quenching method. The composition of the Holmium (III) oxide with $x = 3, 5, 8, 10, 11.5$ and 12 in mole% add with tellurite (ii) oxide was weighed by using digital weighing machine with accuracy of $\pm 0.01\text{g}$ and mixed together by using mortar and pestle. The mixture was then poured into alumina crucible and put into electric furnace set with 100°C for period of 30 minutes. After that, the temperature increased with the increasing rate $10^\circ\text{C}/\text{min}$ until 1000°C and maintained the temperature for 1 hour. When the melting was complete, the molten

liquid was cast into a stainless steel cylindrical shape mould which had been preheated at 350°C for 30 minutes. The sample was annealed at 350°C in second furnace for 2 hours then the furnace was turn off and cooling down with room temperature. Two pieces of cylindrical shape of the glass sample were cut with required size. One piece of the sample was ground and polished for measurement of density and refractive index and another piece will be grind into powder form for XRD analysis and FTIR analysis. The densities for the glass samples were measured by using The Archimedes principle, and distill water use as the immersion liquid. The optical properties such as refractive indices (n) were determined by using EL X-02C high precision ellipsometer. The arrangement of samples glass bonding were investigated by using Fourier transform infrared spectroscopy (FTIR) analysis and the structures of holmium tellurite glasses were investigated by using X-ray diffraction (XRD) technique. All the measurements were run at room temperature.

RESULTS AND DISCUSSION

Density, molar volume and refractive index

The density of the sample increase with the holmium content added. The densities range from 5.14 g/cm³ to 5.93 g/cm³. The refractive index taken at wavelength 632.8 nm (Table 1) increased from 1.88 to 1.94.

Table 1: Variation of the weight fraction of TeO₂ and Ho₂O₃, molar mass, density, molar volume and refractive index of the (Ho₂O₃)_x(TeO₂)_{100-x} glasses system

| Sample | TeO ₂ (mol%) | Ho ₂ O ₃ (mol%) | Molar mass (g/mol) | Density (g/cm ³) | Molar Volume(cm ³) | Refractive index in wavelength 632.8nm, n |
|---------|----------------------------|--|-----------------------|---------------------------------|-----------------------------------|--|
| TH 3 | 97 | 3 | 16614.78 | 5.14 | 3232.45 | 1.88 |
| TH 5 | 95 | 5 | 17051.30 | 5.30 | 3217.23 | 1.89 |
| TH 8 | 92 | 8 | 17706.08 | 5.51 | 3213.44 | 1.90 |
| TH 10 | 90 | 10 | 18142.60 | 5.64 | 3216.77 | 1.92 |
| TH 11.5 | 88.5 | 11.5 | 18469.99 | 5.76 | 3206.60 | 1.93 |
| TH 12 | 88 | 12 | 18579.12 | 5.83 | 3186.81 | 1.94 |
| TH 13 | 87 | 13 | 18797.38 | 5.93 | 3169.88 | 1.94 |

Figure 2 shows that the molar volume decreases and the density increases as holmium ion content increases. This agrees with the density definition, that is the mass of glass sample divide by the molar volume of the glass sample.

Besides that, there might be ion holmium substituted inside the glass network and make the glass matrix become denser, the intermolecular spacing decreases [3] and decrease in molar volume and an increase in density.

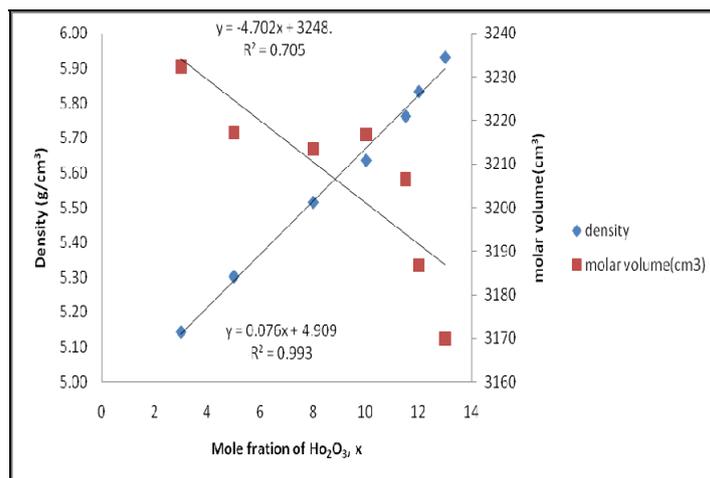


Figure 1: Variation of the weight fraction of Ho₂O₃ with density and molar volume of the (Ho₂O₃)_x(TeO₂)_{100-x} glasses system

The refractive index of the holmium tellurite glass increase when the ion holmium content increases as shown Table 1. The range for the refractive index is between 1.97 and 2.12. Refractive index depends on the polarization of the ions in the molecular structural. Since ions Ho³⁺ which has high polarity, is added into glass network and this will increase the refractive index. Furthermore refractive index also depend on the disorder in the glass sample such as non-bridging oxygen. Yanfei Chen et al. state that the polarization of non-bridging oxygen (NBO) higher than the bridging oxide [6] and this will cause the refractive index to increase; and this same phenomena occur in this holmium tellurite glass system when the ion holmium increased.

XRD

In Figure 2, the XRD pattern show that TH 3, TH 5, TH 8, TH 10 and TH 11.5 are in amorphous phase. For sample TH12 and TH 13 are in crystalline phase. In this research, the glass samples in amorphous phase was studied.

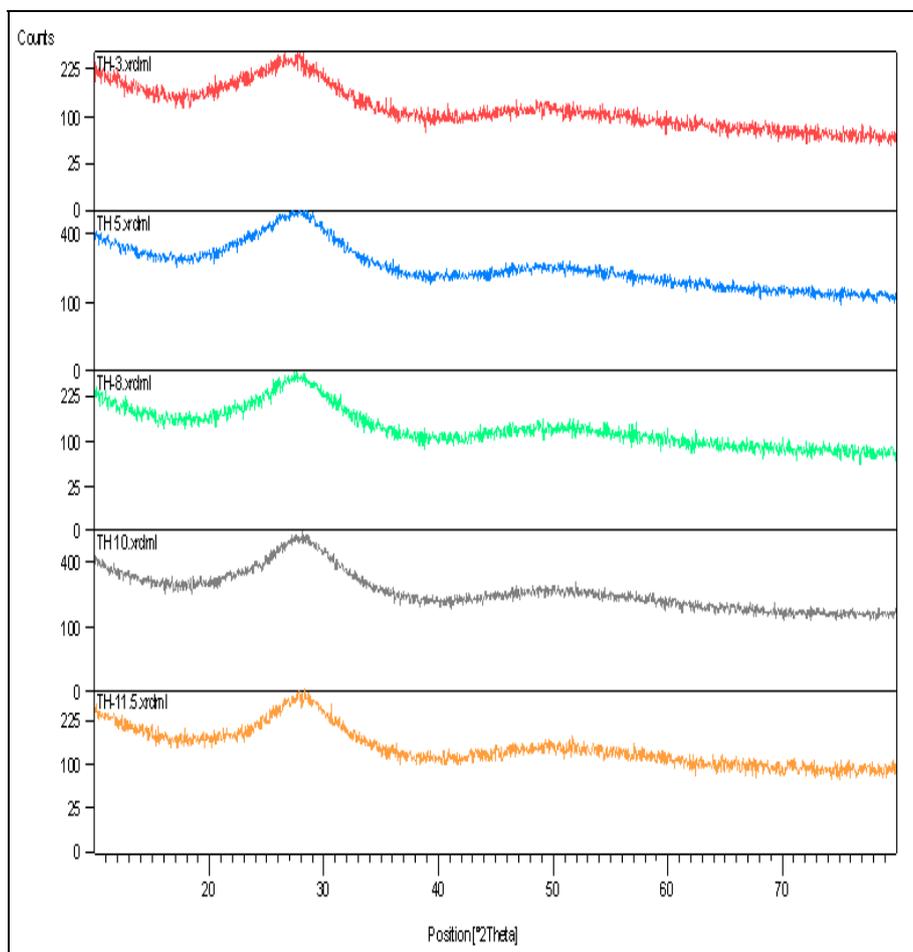


Figure 2: XRD analysis of the $(\text{Ho}_2\text{O}_3)_x(\text{TeO}_2)_{100-x}$ glasses system

FTIR result

From the FTIR analysis shown in Figure 3, the TeO_2 is the glass former while Ho_2O_3 is the glass modifier in binary holmium tellurite glasses system. The wavenumber for the holmium tellurite glass system is shifted slightly to the low wavenumber when the ion Ho^{3+} is increased. When holmium ions content increase, TH3 consists a band at 700.00cm^{-1} , then band shift to low frequencies 663.72cm^{-1} , 652.15cm^{-1} , 643.29cm^{-1} and 648.76cm^{-1} for TH5, TH8, TH10 and TH11.5 respectively. There is a diffuse band in the range of $700\text{-}640\text{ cm}^{-1}$, due to the disordered structure exit and considers the vibration modes of both TeO_3 and TeO_4 entities. The vibration modes of the bonds in both TeO_{3+1} unit are also found at around 580cm^{-1} where TeO_{3+1} unit consist of intermediate coordination of the tellurium atoms between 3 and 4 are found in the all glass samples. As Ho^{3+} content increases, the network modifier ions in the tellurite glass enhance the breaking of axial Te-O-Te linkages in the trigonal $[\text{TeO}_4]$ bipyramids (tbp). This causes the appearing of $[\text{TeO}_3]$ trigonal pyramid (tp) units and the formation of the

non-bridging oxygens [7].

Table 2: The reference of the wavenumber with the selected mode

| Wavenumber(cm^{-1}) | Mode |
|--------------------------------|---------------------------------|
| 650-660 | Te-Oax in $[\text{TeO}_4][4,5]$ |
| 775 | Te-Oeq in $[\text{TeO}_4][4,5]$ |

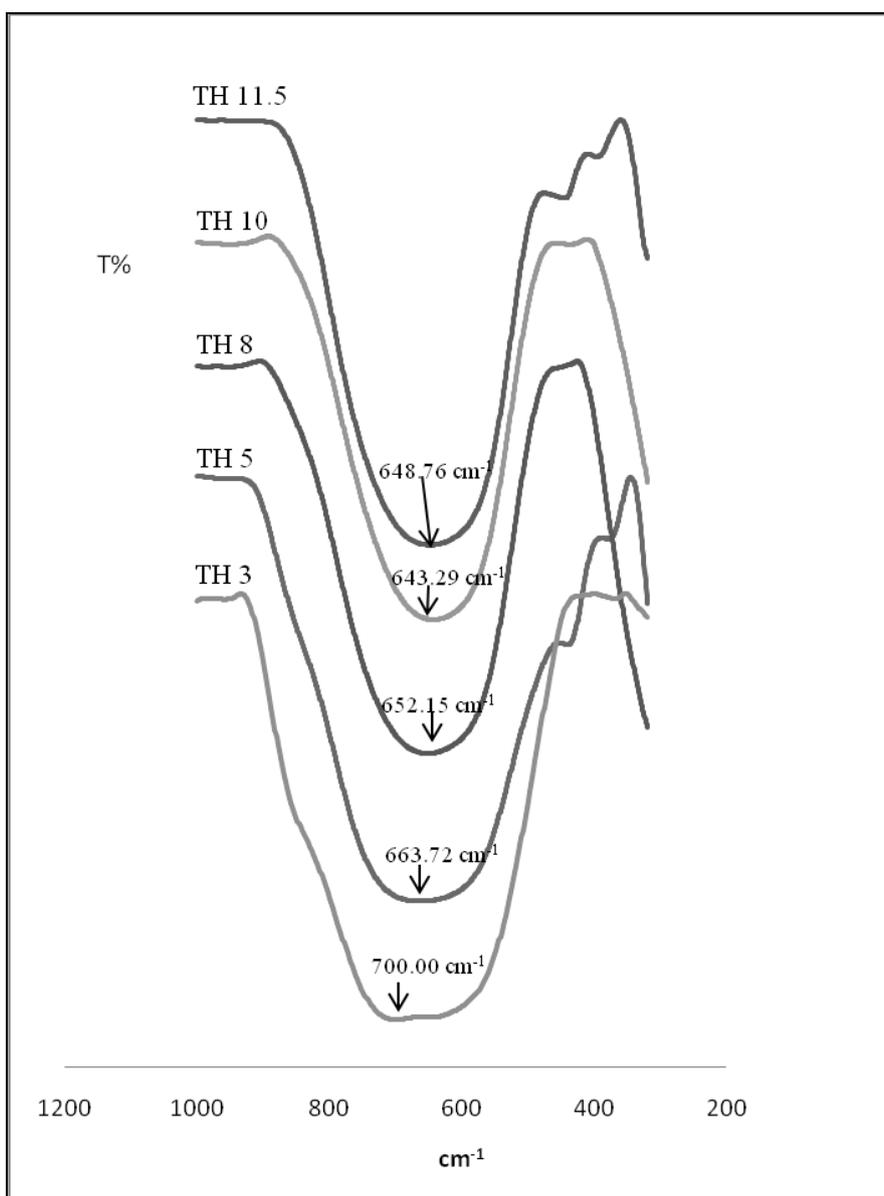


Figure 3: FTIR analysis of the $(\text{Ho}_2\text{O}_3)_x(\text{TeO}_2)_{100-x}$ glasses system

CONCLUSION

When holmium ion content is increased, the density of the glass samples increases, and the molar volume of the glass sample decreases. The refractive index increases due to the increase of high polarity of ion Ho^{3+} content into tellurite base glasses, and an increase in the forming of non bridging oxygen. The phase range of glass amorphous forming is found from 3 to 11.5 mol% of ion holmium content in tellurite base glass.

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