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Yangon University**

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FOREWORD

I would like to take a great opportunity to express my warmest welcome to the publication of the Vol.2, No.2 of the Journal of the Asia Research Centre, Yangon University.

Yangon University, a research-oriented higher education institution, has a multi-disciplinary research laboratory namely the Universities' Research Centre with a good stock of modern advanced research facilities. The academic staff from all departments of Yangon University are engaged in research programmes with objectives: to conduct research at international level, to enhance skills, creativity and capabilities in the performance of research, and to create new generation of outstanding researchers. A great deal of research has made excellent contributions towards the development of the nation as well as the development of the academic concerns.

Yangon University is a member of ASEAN University Network and has a good cooperation in education with the universities in ASEAN member countries. Since its establishment on the 19th August 2002, the Asia Research Centre, Yangon University has been making the financial support to the researchers from Yangon University and other institutions to conduct a good number of outstanding research projects. The principal financier of the Asia Research Centre is the Korea Foundation for Advanced Studies (KFAS).

This journal accommodates 17 research papers resulted from the outstanding research projects in Science, Earth Science and Life Science conducted by the academic departments of Yangon University. I would like to express my appreciation and congratulations on the concerted effort of the researchers who have made a great deal of excellent contributions to this issue.

I also would like to express my heartfelt thanks to Prof. Dr Kim Jae Youl, President of the Korea Foundation for Advanced Studies for his continued support to the Asia Research Centre, Yangon University.



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CONGRATULATORY MESSAGE

Dear Colleagues,

On the occasion of the publication of the second volume of the *Asia Research Journal, Yangon University*, I wish to forward congratulations to the Asia Research Center(ARC-YU) and all researchers at Yangon University.

The Asia Research Center at Yangon University is one of sixteen ARCs established in the region with the aim of providing financial support for local research projects, publication of research findings, and other academic activities, thereby encouraging Asian scholars to explore new ideas and innovations. Yangon University and The Korea Foundation for Advanced Studies co-established the center in 2002 and immediately began supporting Myanmar scholars. This has enabled scholars to embark on investigations in their field of interest and research topic with the confidence that the Asia Research Center is in full support. Such encouraging atmosphere has boosted the scientific spirit and resulted in many important research papers throughout the past seven years.

The *Asia Research Journal, Yangon University* was published once in 2004, which compiled the research summaries between the years 2002 and 2004. Subsequent research results have been synopsisized for Volume 2 in this recent publication so that the information can be circulated among all interested parties and related scientists. While this new volume will be a source of inspiration for many students and scholars, the indispensable information will contribute to the academic upgrading of Myanmar and the Asian region.

I envision this journal becoming an influential channel of international academic exchange that will effectively disseminate research achievements of Myanmar scholars and contribute to strengthening the scientific foundation for social development in Myanmar. I wish to thank Rector Tin Tun, leaders of Yangon University, Director Pho Kaung and others at the Asia Research Center for their leadership and guidance. Their dedication in bringing academic excellence in higher learning has been exemplary and commendable.

I send my best wishes for the publication.

Sincerely,



Jae-Youl Kim, PhD
President

Effect of Thermal Annealing on $x\text{B}_2\text{O}_3 - (95-x)\text{TeO}_2 - 5\text{Fe}_2\text{O}_3$ Glasses

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Abstract

Compositions of $x\text{B}_2\text{O}_3-(95-x)\text{TeO}_2-5\text{Fe}_2\text{O}_3$ glass system (with $x = 0, 10$ and 20) treated by isothermal annealing have been investigated using X-ray powder diffraction (XRD), Differential Thermal Analysis (DTA), Fourier Transform Infrared (FT-IR) and Raman spectroscopic techniques. The XRD data reveals the formation of a paratellurite ($\alpha\text{-TeO}_2$) and iron tellurite on isothermal annealing. The changes in glass transition temperature (T_g) and crystallization temperature ($(T_c)_{\text{max}}$) with composition x are interpreted in terms of the mixed-former effect using data of DTA. FTIR and Raman spectra of this glass system have been analyzed.

Key Words: Tellurite Glass, XRD, TG-DTA, FT-IR, Raman

1. Introduction

Inorganic oxide glasses are of commercial application due to their structural versatility and well-known application. Amongst these borate glasses have been of particular scientific interest because of the presence of boron anomaly in them. They have been extensively studied by techniques such as nuclear magnetic resonance (NMR) and X-ray, ultraviolet and infrared (IR) spectroscopy. However little work has been done on borates containing Fe_2O_3 , addition of which often makes them electronically conducting. Tellurite glasses (based on TeO_2) have not been studied in as much detail as other oxide glasses.

Tellurite glasses have optical non-linearity and high transparency in the visible-to-IR region. Relatively low glass transition temperature (T_g) is of advantage for the preparation of so-called advanced materials, e.g, an "optical memory" device that may be utilized in the field of optoelectronics^[6]. Changes of optical properties can be used as the "optical memory" effect. The structure and properties of oxides glasses depend strongly on the nature and concentration of constituent oxides^[5]. Iron tellurite glasses are particularly attractive for crystallization and structural changes. It would be of interest to see the effect of addition of Fe_2O_3 to the conditional glass former in presence of varying concentration of another glass former B_2O_3 . The effect of thermal annealing on the glasses in $\text{B}_2\text{O}_3\text{-TeO}_2\text{-Fe}_2\text{O}_3$ system has also been investigated. The changes have been monitored by various techniques such as X-ray diffraction (XRD), Differential Thermal Analysis (DTA), Fourier Transform Infrared (FTIR) and Raman spectroscopic techniques, and the observed results are discussed in the present work.

Experiment

Iron tellurite glasses containing B_2O_3 having the compositions, $x\text{B}_2\text{O}_3 - (95-x)\text{TeO}_2 - 5\text{Fe}_2\text{O}_3$ with $x = 0, 10$ and 20 have been prepared by a conventional melt-quenching method. The mixtures of the starting materials are H_3BO_3 , TeO_2 and Fe_2O_3 (5mol %) reagent grade in the appropriate amounts have been mixed thoroughly in an agate pestle-mortar and then put into porcelain crucible. After being pulverized, sinter first at about 300°C for a few hours to convert boric acid (H_3BO_3) into boric oxide (B_2O_3). This process prevents foaming on melting. After that it was melted at about 1000°C for an hour in an electric furnace. The homogeneous glass samples were prepared by quenching the melt onto the cold copper plate and were pressed by another copper plate.

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Heat treatment (isothermal annealing) was given at 400°C for 0 min, 100 min and 200 min in an electric furnace. Each sample was characterized by its XRD pattern. Rigaku Multiflex-2kW diffractometer was used at a scanning rate of 2 degree min^{-1} with a Cu-K α source. The IR transmission spectra were measured on an FT-IR spectrometer (FT-IR 8400 Shimadzu) by the KBr disk method.

The values of the glass transition temperatures (T_g) and crystallization peak temperatures (T_c) have been determined by Differential Thermal Analyzer (DTA which conducted at heating rate of 15°C min^{-1} using Shimadzu's apparatus DTG 60AH. An aluminium (Al) pan was used as the standard sample. All measurements have been carried out heating under N₂ atmosphere (50 ml min^{-1}).

To complement the thermal analysis, Raman spectrum for the glass samples have been taken with scanning time of 20 sec. Raman spectra have been measured in the back scattering geometry using Ocean optics RSI 2001 S Raman spectrometer. The excitation light source used a solid-state diode laser with the constant output of 500 mW at 514nm.

3. Results and Discussion

Homogeneous glass samples could be prepared in the $x\text{B}_2\text{O}_3-(95-x)\text{TeO}_2-5\text{Fe}_2\text{O}_3$ system where in the Fe_2O_3 content is 5mol% and B_2O_3 content varies corresponding to difference x values: $x = 0, 10$ and 20 . The XRD pattern of the reagent mixture (95TeO_2 plus $5\text{Fe}_2\text{O}_3$) is shown in Fig. 1. The XRD pattern is generally observed in several oxide glasses with an "amorphous" structure. The formation of homogeneous glass samples was confirmed by XRD, as illustrated in Fig. 2.

Isothermal annealing of the $95\text{TeO}_2-5\text{Fe}_2\text{O}_3$ glass at a temperature close to T_c (about 400°C) resulted in precipitation of crystalline particles. So, XRD pattern of this sample heated at 400°C for annealing time 0min and 200min is shown in Fig. 3. Several diffraction peaks indicates that the crystallized phase consists of paratellurite ($\alpha\text{-TeO}_2$) and iron tellurite of which diffraction peaks are marked with open and closed circles.

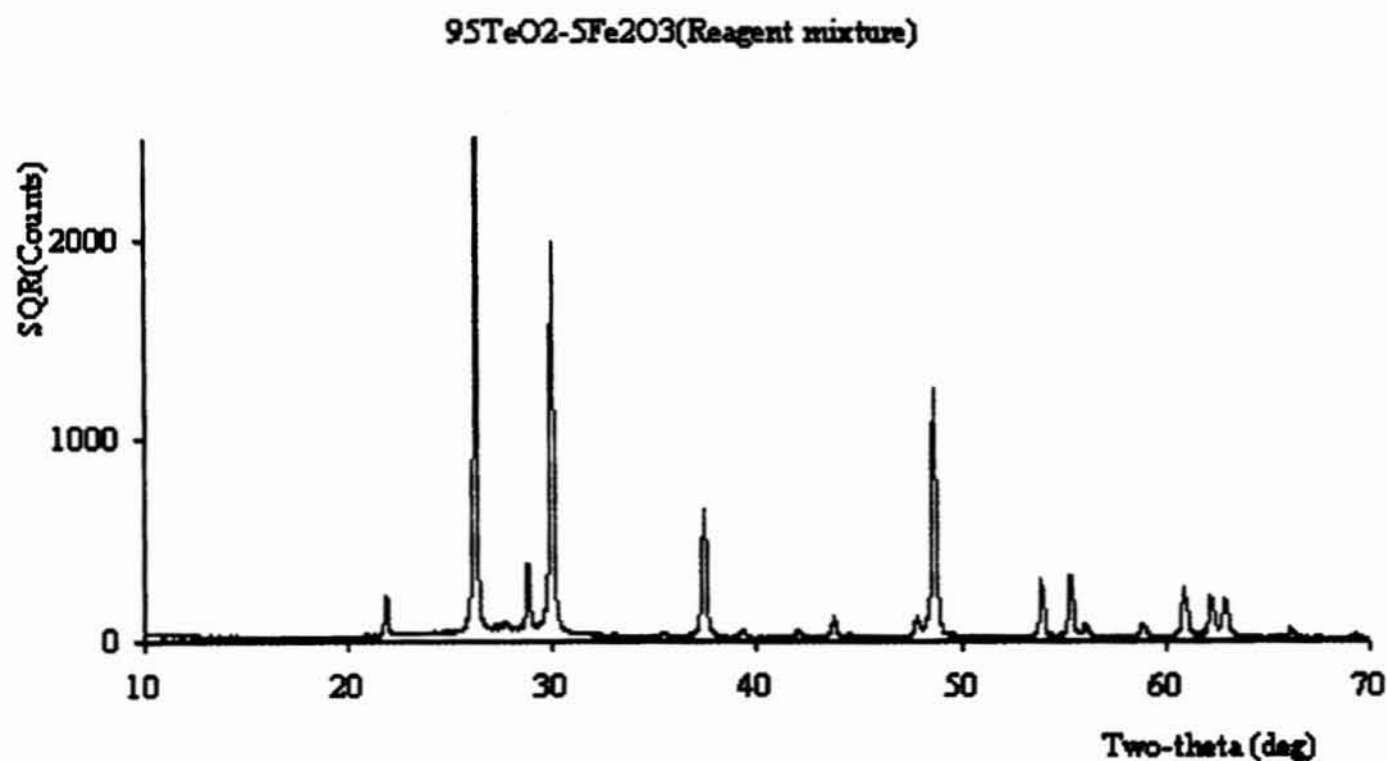


Figure 1 The XRD pattern of the reagent mixture (95TeO_2 plus $5\text{Fe}_2\text{O}_3$)

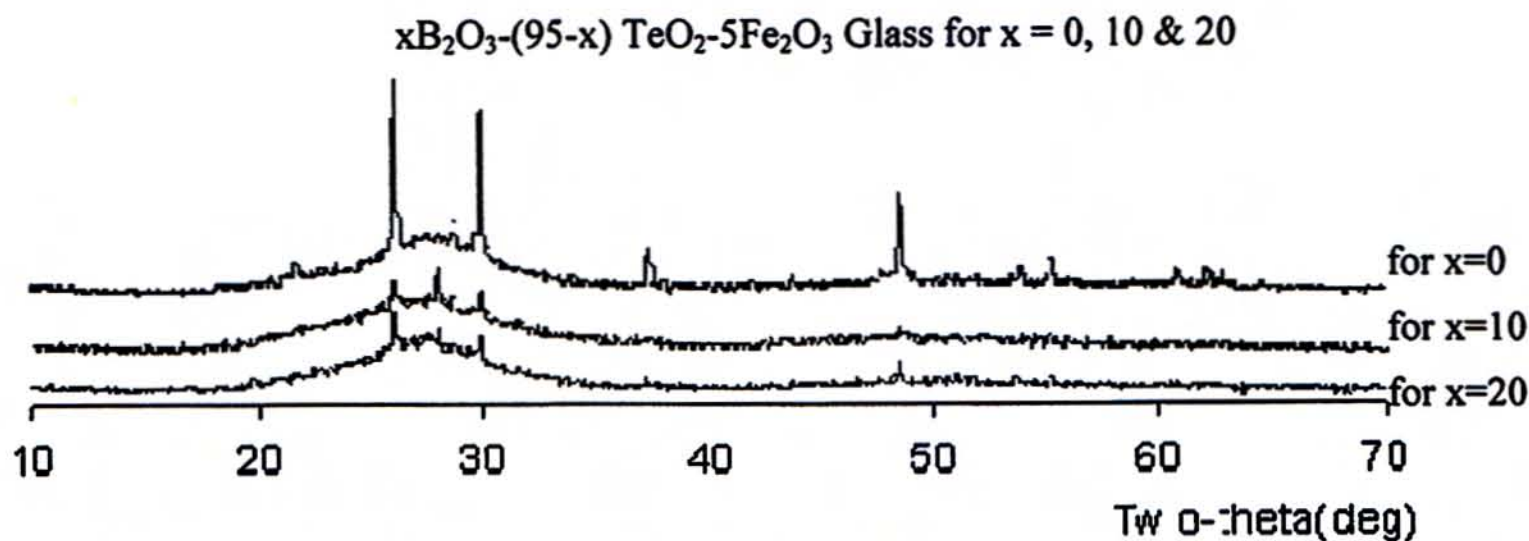


Figure 2 XRD pattern of $x\text{B}_2\text{O}_3-(95-x)\text{TeO}_2-5\text{Fe}_2\text{O}_3$ Glass for $x = 0, 10 \text{ \& } 20$

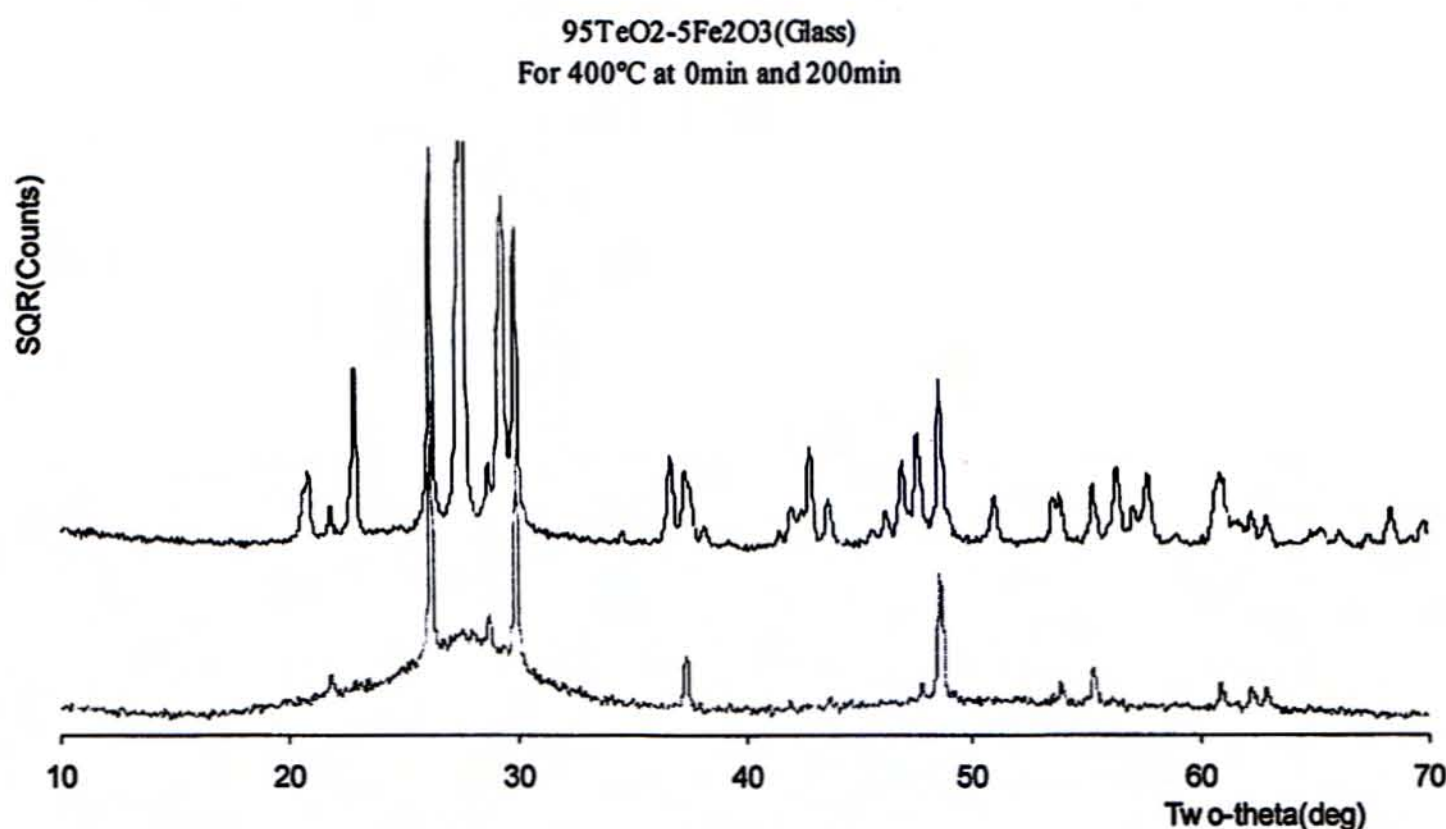


Figure 3 The formation of homogeneous glass samples confirmed by XRD

The DTA curves of $x\text{B}_2\text{O}_3 (95-x)\text{TeO}_2 5\text{Fe}_2\text{O}_3$ for $x = 0, 10$ and 20 glass are shown in Fig 4. The values of glass transition temperature (T_g) and the maximum crystallization temperature $(T_c)_{max}$ are presented at the table 1. Heating rate was kept at 15°Cmin^{-1} .

Table 1 Glass transition temperature (T_g) and the maximum crystallization temperature $(T_c)_{max}$ with value of x

x	$(T_g)^\circ\text{C}$	$(T_c)_{max}^\circ\text{C}$
0	334	411
10	340	427
20	364	458

The variation in glass transition temperature (T_g) and maximum crystallization temperature $(T_c)_{max}$ of the $x\text{B}_2\text{O}_3-(95-x)\text{TeO}_2-5\text{Fe}_2\text{O}_3$ glasses with composition are shown in Fig 5(a) and (b) respectively. It can be seen that (T_g) and $(T_c)_{max}$ passes through a maximum value only for B_2O_3 ($x = 20$) based glass. These glasses exhibit a continuous increases in (T_g) and $(T_c)_{max}$ with increase of B_2O_3 content. In general, it is found that the higher values of (T_g) and $(T_c)_{max}$, the more increase the value of x .

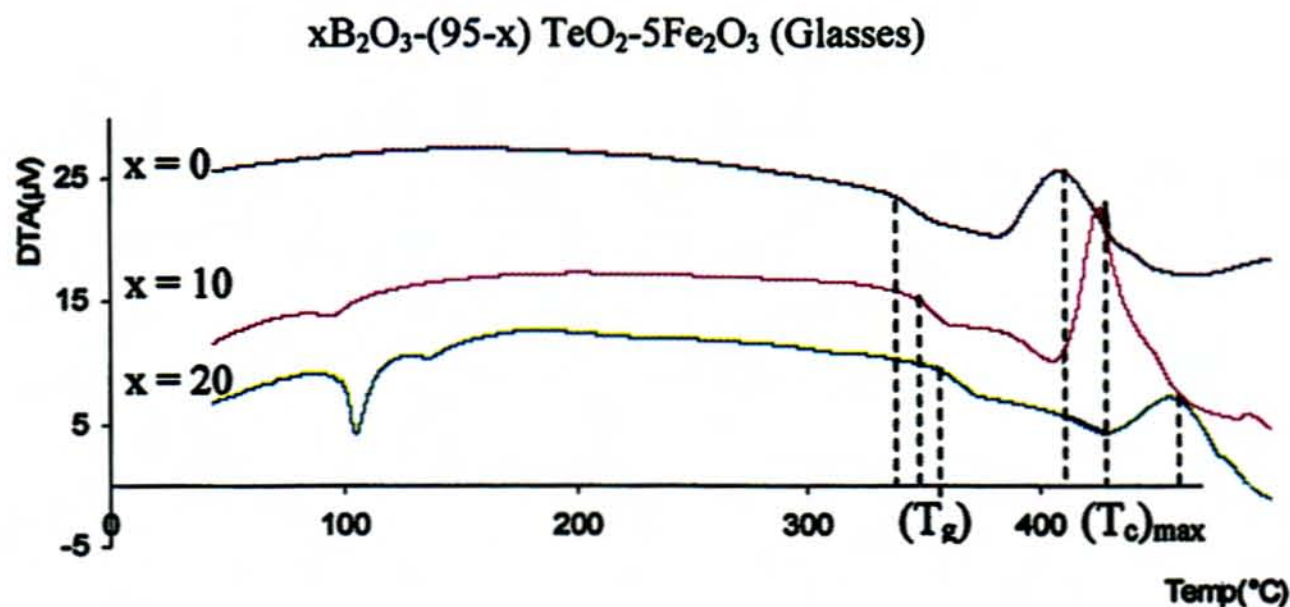


Figure 4 The DTA curves of $x\text{B}_2\text{O}_3-(95-x)\text{TeO}_2-5\text{Fe}_2\text{O}_3$ for $x=0, 10$ and 20

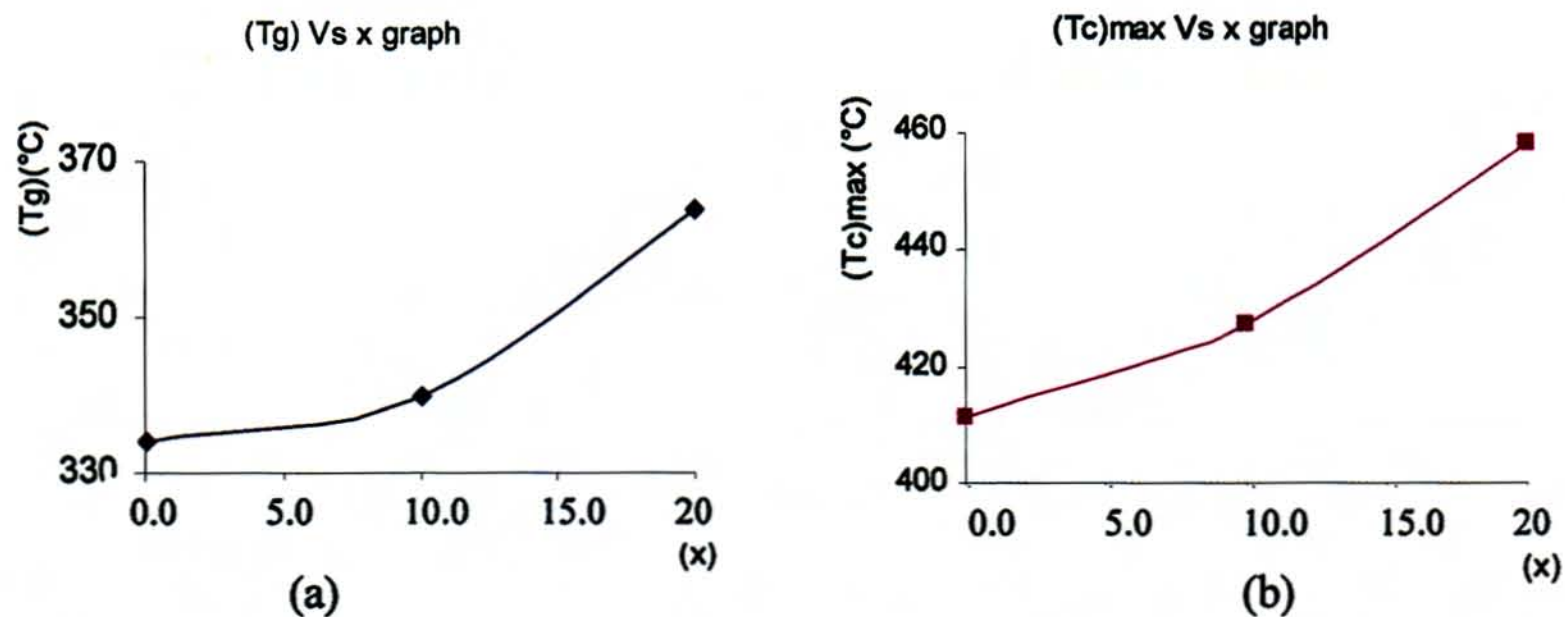


Figure 5 (a) and (b) The variation in (T_g) and $(T_c)_{\max}$ of the $x\text{B}_2\text{O}_3-(95-x)\text{TeO}_2-5\text{Fe}_2\text{O}_3$ glasses with composition x

The IR transmittance spectra of iron tellurite glasses in the system for various values of x (0, 10 and 20) are shown in Fig. 6. The mid infrared spectra of these glasses system in the region $500-1200\text{ cm}^{-1}$ are shown. In this figure two broad bands appeared at the wavelength of $500-800\text{ cm}^{-1}$ and $1000-1200\text{ cm}^{-1}$ respectively.

The formation of $\alpha\text{-TeO}_2$ is also observed in the IR transmission spectra. The peak appearing around 780 cm^{-1} in Fig. 7(a-d) is ascribed to the asymmetric Te-O_{ax} stretching band of TeO_2 . The IR spectrum of TeO_2 (reagent) is shown in Fig. 7(d) for comparison. The main peak due to the symmetric Te-O_{eq} band becomes sharp with the annealing, as is shown in Fig. 7(a-c). (O_{ax} and O_{eq} indicate the oxygen atoms at axial and equatorial sites, respectively). The bands are observed to be sharper on annealing suggesting thereby the increase in crystallinity.

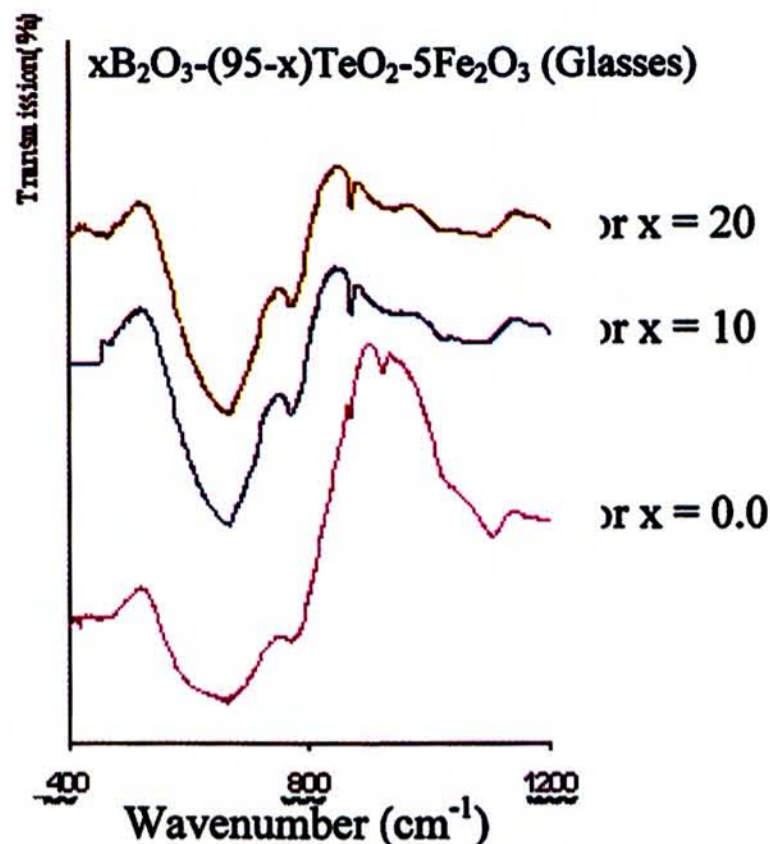


Figure 6 IR transmission spectra of $x\text{B}_2\text{O}_3-(95-x)\text{TeO}_2-5\text{Fe}_2\text{O}_3$ glasses with $x = 0, 10$ & 20

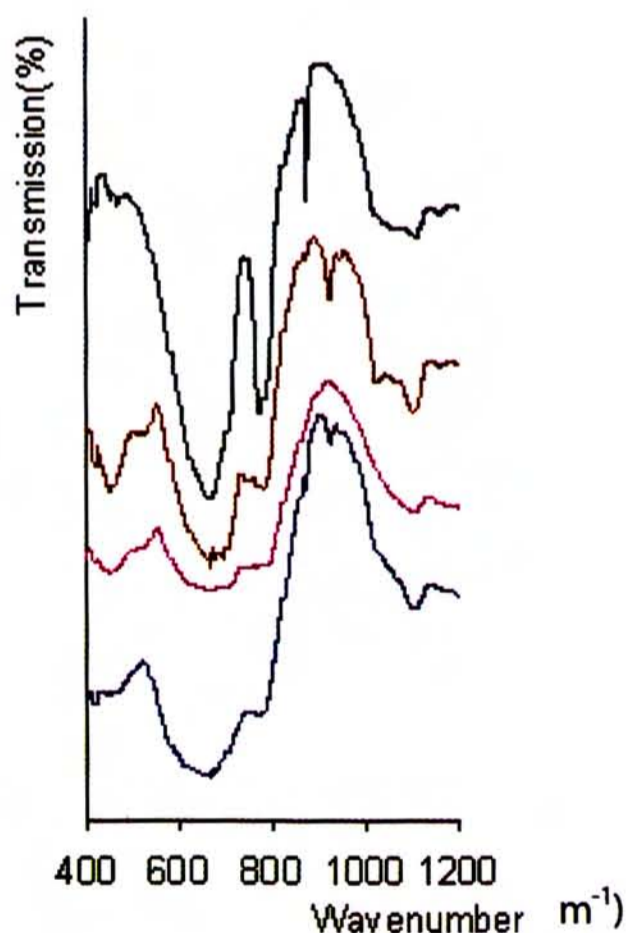


Figure 7 The FT-IR spectra of $95\text{TeO}_2-5\text{Fe}_2\text{O}_3$ glass annealed at for different time intervals: 400°C with (a) 0min, (b) 100min, (c) 200min & (d) IR spectrum of reagent mixture of 95TeO_2 and $5\text{Fe}_2\text{O}_3$

Raman scattering spectra from the $x\text{B}_2\text{O}_3-(95-x)\text{TeO}_2-5\text{Fe}_2\text{O}_3$ glasses system for different values of $x = 0, 10$ and 20 were observed after isothermal annealing process. The spectra for various samples are shown in Fig. 8. In this figure, the scanning time is fixed at 20 sec. It can be seen that glass to crystal transition occurs at the wavenumber about 644 cm^{-1} . So, the transition peak shifts towards higher wavenumber for glasses containing B_2O_3 content.

Raman scattering spectra from the isothermally annealed samples at 400°C for different time periods (0min, 100min and 200min) were observed and are plotted in Fig. 9. After isothermal annealing, it is found that the intensity is increased with the annealing time.

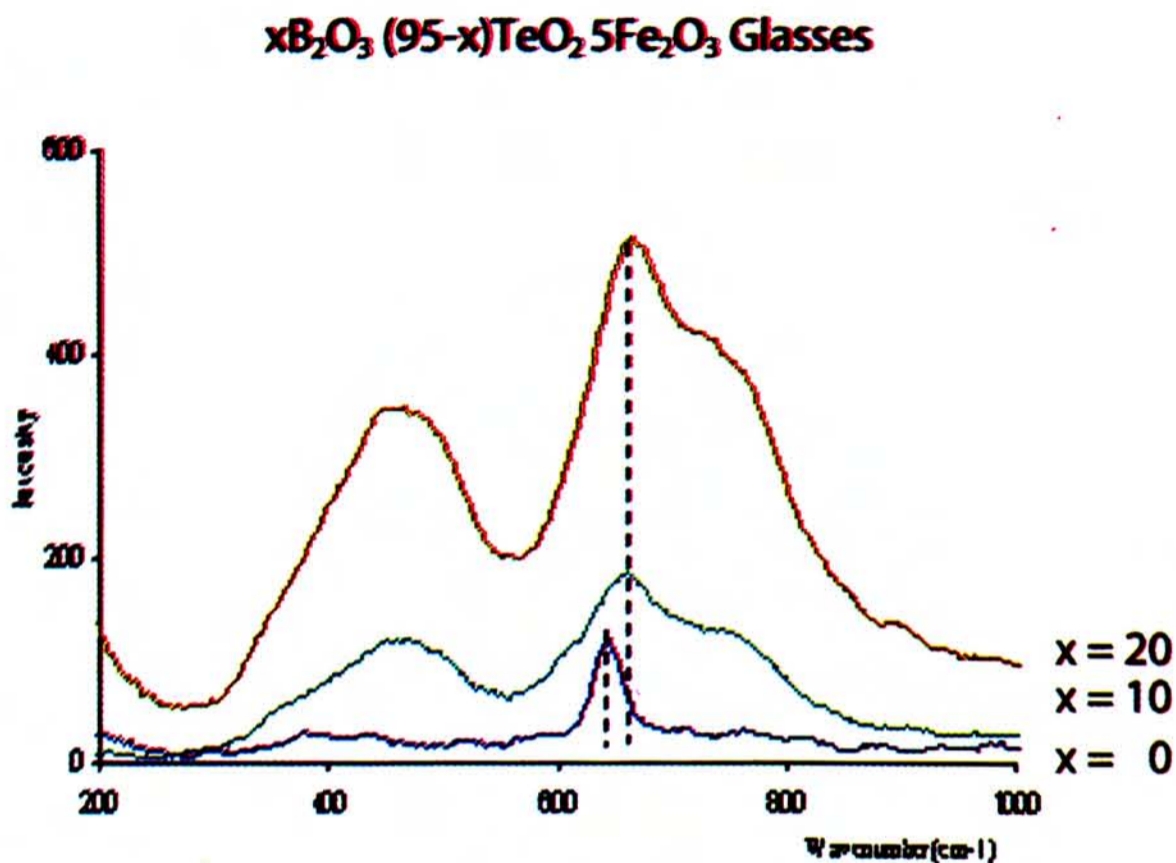


Figure 8 The Raman spectra of $x\text{B}_2\text{O}_3$ (95-x) TeO_2 5 Fe_2O_3 glasses for different values of x

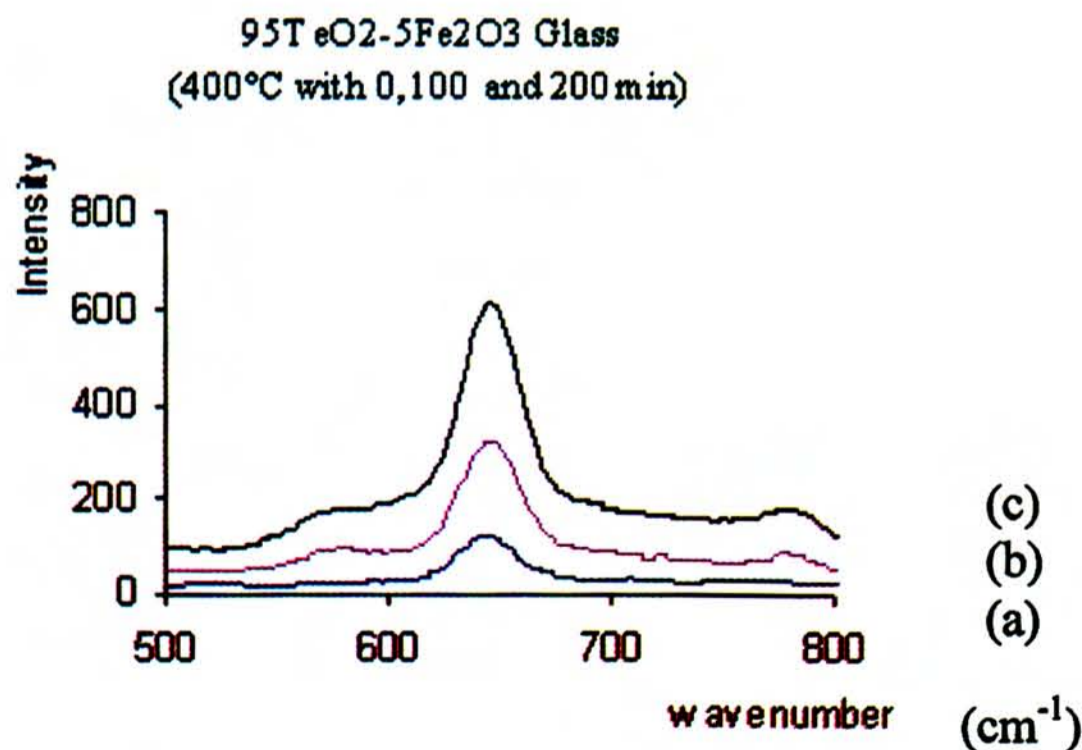


Figure 9 The Raman spectra of 95 TeO_2 5 Fe_2O_3 glass annealed at 400°C for different time intervals: (a) 0min, (b) 100min, (c) 200min

4. Conclusion

The series of glass samples having compositions: $x\text{B}_2\text{O}_3$ (95-x) TeO_2 5 Fe_2O_3 with $x = 0, 10$ and 20 have been prepared using standard melt-quenching technique. The effect of annealing as a function of annealing time at a temperature of 400°C (above glass transition temperature) has been investigated. The changes have been monitored by XRD, DTA, IR and Raman spectroscopic techniques. The sample with $x = 0$ is found to exhibit in its XRD pattern, sharp peaks corresponding to crystalline TeO_2 superimposed on broad hump which is a characteristic of glassy phase. The intensities of these sharp peaks are found to decrease with the addition of increased amount B_2O_3 (i.e. x).

The presence of glassy phase was supported by the DTA studies also value of T_g was found to lie between 330°C and 360°C . The observed data show that small amount of Fe_2O_3 (5mol%) helps in dissolving a part of TeO_2 , conditional glass former, into glassy phase while the remaining TeO_2 is left behind in the crystalline phase. Addition of another glass former B_2O_3 is found to decrease the intensity of peaks of TeO_2 indicating thereby the reduction in the amount of crystalline TeO_2 . This might be due to lower concentration of TeO_2 in the sample and /or a part of crystalline TeO_2 might have entered into the already existing glassy phase. This is because TeO_2 is a conditional glass former and Fe_2O_3 has been reported to act as glass former or glass modifier in Na_2O - B_2O_3 glasses system.

Thermal annealing is found to induce crystallization of the glassy phase. The XRD patterns of annealed samples indicate the presence of iron-telluride crystallites in addition to TeO_2 crystals (which were present in the unannealed samples also). IR and Raman spectroscopic data of the present samples seem to support the present findings.

Acknowledgement

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