INVESTIGATIONS OF TEMPERATURE DEPENDENT OPTICAL PROPERTIES OF TELLURIUM BASED GLASS MATERIALS FOR PHOTONICS AND BIOMEDICAL APPLICATIONS

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Master Thesis

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I certify that in my opinion the thesis submitted by Taufiq ABDULLAH titled "INVESTIGATIONS OF TEMPERATURE DEPENDENT OPTICAL PROPERTIES OF TELLURIUM BASED GLASS MATERIALS FOR PHOTONICS AND BIOMEDICAL APPLICATIONS" is fully adequate in scope and in quality as a thesis for the degree of Master of Science.

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Taufiq ABDULLAH

ABSTRACT

M. Sc. Thesis

INVESTIGATIONS OF TEMPERATURE DEPENDENT OPTICAL PROPERTIES OF TELLURIUM BASED GLASS MATERIALS FOR PHOTONICS AND BIOMEDICAL APPLICATIONS

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Institute of Graduate Programs
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The oxide based glass materials have been using as potential components in the field of optical fiber communication, sensor technologies, photonic crystal fiber, fiber glass, photonics glass and many other related areas. These type of glass materials have amorphous nature with very good transparent properties. The basic purpose of this experiment is to design a unique glass network composed of $70TeO_2$-$15ZnCl_2$–$10Nb_2O_5$–$5TiO_2$ to define the optical properties, thermal properties and structural properties. In this research work, the proposed optical glasses were produced using the melt quenching method. The optical properties and optical band gap properties was analyzed using UV-VIS spectra for ultraviolet absorption of the glass molecules. The thermal properties were examined using Differential Thermal Analysis (DTA). Different thermal parameters including the transition temperature of the glass, the melting temperature of the glass, the onset temperature and the temperature of crystallization were derived from the DTA investigation. The crystallographic structure was analyzed using Scanning Electron Microscope (SEM) and X-Ray Diffraction (XRD). The mechanical and structural properties was determined using Micro Vicker hardness respectively.

Key Words : Tellurium glass, optical glass materials, photonic glass, fiber glass, DTA, UV spectra, thermal hardness, metal oxide glass materials

Science Code : 92503

ÖZET

Yüksek Lisans Tezi

TELLÜR ESASLI CAM MALZEMELERİN FOTONİK VE BİYOMEDİKAL UYGULAMALARDA SICAKLIĞA BAĞLI OPTİK ÖZELLİKLERİNİN İNCELENMESİ

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Metal oksit bazlı cam malzemeler, optik fiber iletişimi, sensör teknolojileri, fotonik kristal elyaf, cam elyaf, fotonik cam ve diğer birçok ilgili alanda potansiyel bileşenler olarak kullanılmaktadır. Bu tür optik cam malzemeler çok yüksek şeffaflığa ve şekilsiz bir yapıya sahiptir. Bu deneyin temel amacı, $70TeO_2$-$15ZnCl_2$–$10Nb_2O_5$–$5TiO_2$'den oluşan bir cam ağının sentezlenmesidir Bu çalışmada optik özellikleri, termal özellikleri ve yapısal özellikleri tanımlamak için önerilen cam ağı geleneksel eriyik söndürme yöntemi kullanılarak hazırlanmıştır. Optik özellikler ve optik bant aralığı özellikleri, cam moleküllerinin ultraviyole absorpsiyonu için UV Görünürlük spektrumları kullanılarak analiz edildi. Termal özellikler Diferansiyel Termal Analiz (DTA) kullanılarak incelenmiştir. DTA araştırmasından cam geçiş sıcaklığı Tg, erime sıcaklığı Tm, başlangıç sıcaklığı Tx ve kristalleşme sıcaklığı Tp dahil olmak üzere farklı termal parametreler belirlenmiştir. Kristalografik yapı, Taramalı Elektron

Mikroskobu (SEM) ve X-ışını Kırınımı (XRD) kullanılarak analiz edildi. Mekanik ve yapısal özellikler sırasıyla Mikro Vicker sertliği kullanılarak belirlendi.

Anahtar Kelimeler : Tellür cam, optik cam malzemeler, fotonik cam, fiberglas, DTA, UV spektrumları, termal sertlik, metal oksit cam malzemeler.

Bilim Kodu : 92503

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SYMBOLS AND ABBREVITIONS INDEX

SYMBOLS

T : Temperature

Ɛ : Dielectric constant

R : Electrical Resistance

TiO_2 :Titanium dioxide

$ZnCl_2$: Zinc chloride

Nb_2O_5 : Niobium oxide

TeO_2 : Tellurium dioxide

ABBREVITIONS

log : logarithmic

DTA : Differential Thermal Analysis

FT-IR : Fourier Transform Infrared Spectroscopy

IR : Infra Red

SEM : Scanning Electron Microscope

UV : Ultraviolet

UV-VIS-NIR :Ultraviolet-Visible-Near Infra Red Absorption Spectrometer

XRD : X-ray Diffraction

LED : Light Emitting Diode

PART 1

INTRODUCTION

Metal oxide based glass materials have been using as a potential candidate in the field of optical fiber communication, sensor technologies, photonic crystal fiber, fiber glass, photonics glass, lasers, fiber optic cables, radiation shielding materials, biomaterials and tissue engineering and many other related areas. More specifically, the tellurium based glass materials have drawn attention of many national and international researchers in recent days. These type of glass materials have amorphous nature with very good transparent properties [1].

The basic purpose of this research is to develop a unique combination of optical glass materials. In this study, the proposed glass network composed of $70TeO_2$-$15ZnCl_2$–$10Nb_2O_5$–$5TiO_2$ in order to define the structural, optical and thermal characteristics [2]. The proposed glass network was made using the melt quenching method with annealing to strengthen the glass materials [3,4].

In order to investigate the optical studies, optical energy band gap, UV-Visibility spectra for ultra violet ray of the molecules of the optical glass materials was analyzed [5]. The thermal studies of the optical glass materials were investigated by using the differential thermal analysis technique [6]. Different thermal parameters including the transition temperature of the glass T_g, the melting point T_m, the onset temperature T_x and crystallization temperature T_p were derived through the DTA investigation [5,7]. Thermal properties and in addition to its basic properties, especially its resistance to thermal shock according to high technologies, therefore, its properties can be improved by varying the temperature of the proposed glass network.

The crystallographic structure was examined using Scanning Electron Microscope (SEM) and X-Ray Diffraction (XRD). FT-IR spectroscopy was examined for the glass network as well. The mechanical and structural properties was determined using Micro Vicker hardness respectively.

Based on the tellurium based glass study, efficient and advanced materials for technological innovations of optical glass materials can be produced for photonics and biomedical applications.

In this study, the first chapter is "Introduction" and a short summary of the study is under this title it is given. The second chapter is “Glass Science and Technology" and the tellurium based glass materials discussed in this study explained with a wide literature review. The third chapter is “Materials and Methods” includes the preparation of materials, devices, samples used in this study. In the fourth chapter “Results and Discussions” describes the data obtained from the different analysis of samples and the data obtained are represented graphically and visually for data driven evaluation along with comparisons with similar studies and experiment conducted previously. In the fifth and final chapter, “Conclusion” includes where the final results of the study are evaluated, by interpreting the findings obtained as a result of the experiments and analyzes.

PART 2

GLASS SCIENCE AND TECHNOLOGY

Optical and infrared(IR) glasses, which are among the indispensable materials today, more specially in diverse fields such as lenses, windows, glass fiber, sensors, optical fibers, biomaterials, bio-glass, photonic glass and so on. They have different areas of use, such as technological and decorative. Glasses synthesized by sand, alkali at high temperatures with alkaline rare earth element and other glass-making components the Multi-component materials are generally known as transparent, amorphous materials [8]. Glasses; glass system called meshing agent although it contains one or more components that have the function of the main component in it other class oxides entering into its composition, on the other hand, have a network structure that affects the formation of a glassy structure. They can be defined as regulators [9].

The same component can also be used for different functions in the glass network system. For example, alumina (Al_2O_3) can be used in the glass-making function in aluminate-based glasses, while silicate (SiO_2) can be used as a property modifier in most glasses. It can be tinted by adding various components (Fe_2O_3, ZnO, $ZnCl_2$, PbO, Bi_2O_3, P_2O_5) to the glasses or it can be painted. Thus, in the works of art, in the production of vitrified glass or they can be used as decorative glasses.

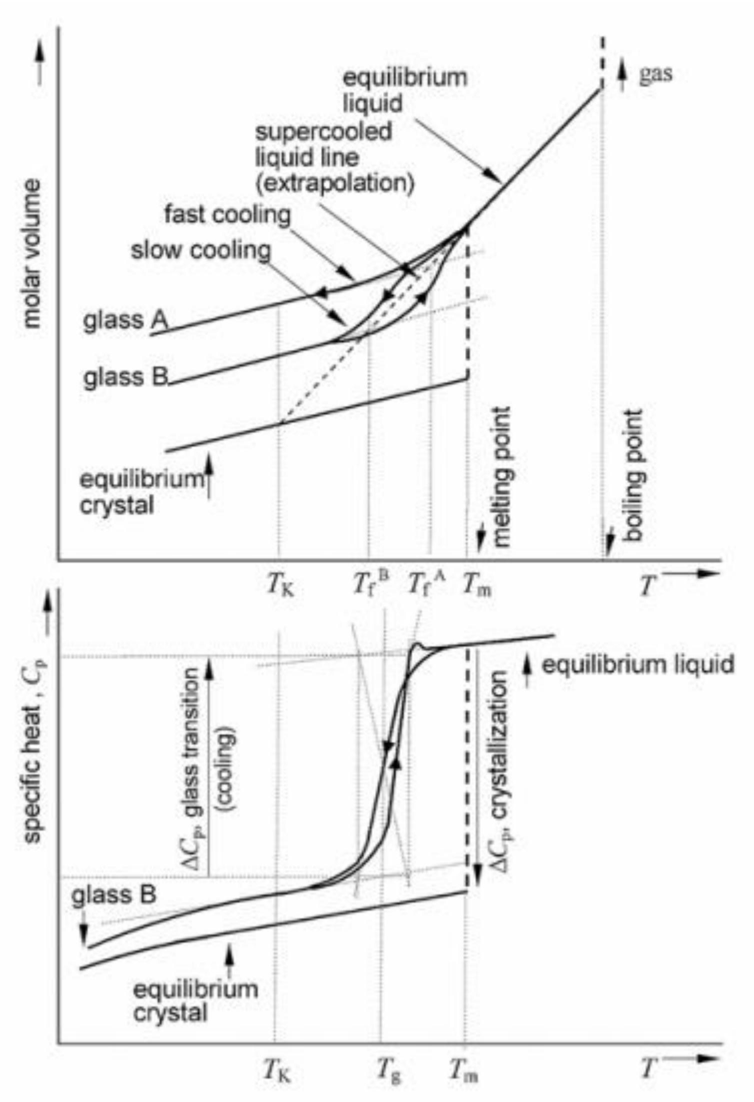

Figure 2.1. Schematic diagram of glass transition, specifically
molar volume and specific heat with respect to temperature [10].

Simple plot designed for the transformation of glass is graphically explained as the function of molar volume with respect to temperature. Glasses, A and B, can be obtained by reducing the cooling rate with imaginary temperature decreases ($T_f{}^a$ and $T_f{}^b$). T_k approximation can be found as an projection limit at zero cooling rate. Hysteresis effect is shown only for glass B. Isobaric heat capacity (C_P) showing a step-like nonlinear variation with some hysteresis in TG analysis [10].

Although glasses have a fragile structure, they can also be used with other alloy type materials in comparison, they are durable in terms of chemical, mechanical, structural properties and glass it has survived from the first historical periods when its production was made to the present day and still there are many examples of glass used. Because it is a product that can be sterilized, it can be used in medical tools and equipment (medicine storage) bio-glass materials are used. In addition, cleaning in everyday life is also a regular part, since it is easy to use tools such as bowls, plates, bottles, jars and jugs, and glass materials can also be used in the production of tools.

2.1. MECHANICAL PROPERTIES OF GLASSES

The glasses are mechanically flexible at the same time as in fiber optic cables however, this situation can be disrupted by ion exchange or annealing by forming a pressurized surface layer or by acid polishing or protective coating. They can be strengthened by removing surface defects. The optical and structural characteristics mainly stand on the specific purpose of uses and based on which the components are chosen for industrial production [11,12]. These are advantageous because they are both environmentally friendly and easy to recycle as materials. For this reason, glass materials are being used today in different purposes including decorations, safety shielding and against nuclear radiation and so on.

2.1.1. Viscosity

Viscosity is the most important mechanical property of glass materials. Inside the glass, a kind of internal decoupling that occurs between the fluid molecules found and in the opposite direction to this flow, it can be defined as the friction force. In addition, viscosity, optical, structural and in addition to its mechanical properties which is a basic property of glass that should be known. Different types of glass materials can be formed according to the viscosity of the glass at temperatures that can be shaped and annealed.

2.2. ELECTRICAL PROPERTIES OF GLASS MATERIALS

In general, the glasses present a good electric resistance and good dielectric characteristics. Moreover, they have significant dielectric constant, but still this is not yet higher enough for special sophisticated optical memory devices and optical communication [13]. Considering the different glass groups, chalcogen semiconductor glass materials have been highly utilized in optical memory devices due to their high electrical properties.

2.3. OPTICAL PROPERTIES OF GLASS MATERIALS

Some basic optical parameters including Ultraviolet, Visible and Infrared Transmission, Rayleigh scattering of the inclusion depend on various factors. Infrared spectrum (IR) band and UV band, optical band-gap; they are the values that the transmission is operated [12,13]. Reflection on the surface of glass materials occurs diffusely or regularly. At the same time, the refractive property of glass materials is due to the fact that it depends on the refractive index. For example, silicate glasses while the refractive index is 1.5, the refractive indices of chalcogen and tellurite glass materials are around 2.0. Glass materials with a high refractive index are usually photonic crystal fiber and photonic devices.

2.4. THERMAL PROPERTIES OF GLASS MATERIALS

Thermal expansion is one of the most basic and distinctive properties of optical glass materials. The easy measurability of this property and the concentration in the glass components since it is sensitive to its proportions, so that it can be monitored regularly. In optical glass materials research, it is the most used control parameter in ensuring the control of production which can also be defined as the resistance of glasses against thermal changes. The most important factor in thermal shock resistance is the thermal expansion coefficients of glasses. The fact that this coefficient is small is also a factor of the thermal temperature difference that the glass can withstand. Glasses that are heated expand by an amount proportional to the thermal expansion coefficient. This modification of volume can also influence the glass density and refractive index as well [13].

2.5. DENSITY OF GLASS MATERIALS

The glass density is dependent on the chemical component ratio [14]. The buoyancy method, the Archimedes principle and the pycnometric method are mostly used to calculate the density measurement methods. The optical glass sample is first in air and then in a liquid whose density is known a fast and reliable measurement method based on weighing, as the principle of Archimedes it is known. In this measurement method, the liquid used is normally distilled water and the experiment is conducted using a platinum wire [15].

By the weight of a container filled with a liquid whose density is known, its weight is a known, the measurement method based on the glass being placed in this liquid is known as the pycnometric method. The temperature of the optical glass sample placed in a liquid whose density is preset the measurement method based on the collapse of the container to the bottom with its change is the buoyancy method. In this method, a standard density known in advance for measurement the glass is used, and the sample to be measured is also close to the density of that standard glass.

2.6. TELLURIUM BASED GLASS MATERIALS

Although the glass sector is known as the area where tellurium minerals or compounds are used the most considering components in optical glass materials, tellurium-based glasses show a diverse characteristics along with a multiple fields of applications. For this reason, many researchers all over the world are curious to study on tellurite glass.

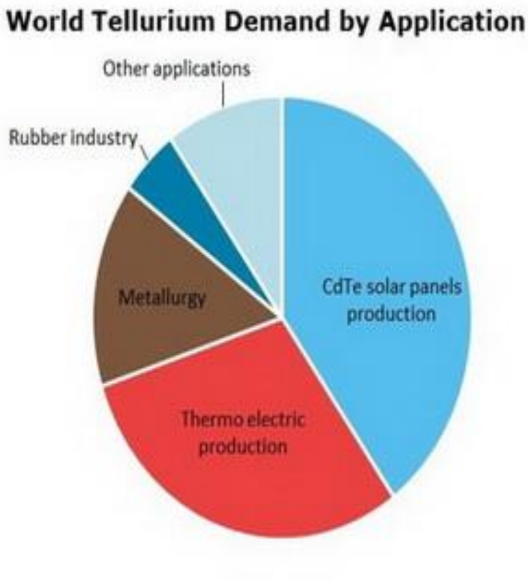

Figure 2.2. World Tellurium Demand By Applications [16].

Even in Türkiye, there are some leading institutions and companies contributing the optical glass research including tellurium based glass, boron based glass, silicon based glass and so on. For example, ASELSAN, Rare Earth Elements Research Institute (NATEN), Boron Research Institute (BOREN), Sisecam and many others who conduct research on tellurium-based glass materials, boron mineral-based materials, silicon-based glass materials and etc. Glasses with tellurium content are increasing for research and developments in many countries recent days. The biggest factor in the increase of these researches is based on the fact that tellurium based glasses have good mechanical properties, resistance to corrosion and thermal stability, outstanding optical properties and so on.

The figure 2.2. represents the world tellurium consumption usage data based on industries, purposes, field of areas and production. From the diagram, it is clear that the most portion of tellurium usage goes to cadmium tellurite based solar cell application that nearly forty percentage of the total supply all over the world. Following that, the thermoelectric production is on the second rank of world tellurium demand and others belong to metallurgy, nano technology, materials engineering, rubber, polymer science and other applications [16]. It is known that there are approximately 176 types of tellurium mineral species on the earth. They are the most used in the glass industry, solar Photovoltaic energy, photonics industry, fiber optics industry and are the main ones in this study is tellurium dioxide, which is denoted by TeO_2.

2.7. BIOMEDICAL APPLICATIONS OF TELLURIUM GLASS MATERIALS

In studies on tellurium-based glasses, these glasses have a good compatibility with textures an eye implant that it can provide and supports cell proliferation, controlled drug release. It can be used in biomedical applications such as tissue and bone formation. A high amount of tellurium that may be released during the dissolution of tellurium-based glasses materials. There is a concern that with its ion, it can become toxic to cells and tissues. The concerns about the toxicity of tellurium-based glasses on cells and tissues etc. which can be mitigated by the results shown. Tellurite glasses have proved more biomedical, bio-cell characteristics including a very good antibacterial properties in comparison with other metal based glasses. For this reason, Tellurium based glasses have a become a very potential candidate for medical implant, biomaterials, bioceramic coating, tissue engineering and many others [17]. Tellurium-based glasses, antimicrobial additives for dental restoration materials as it is used, the coating can be used for medical materials. Antibiotics and cancer drugs, etc. in the transport of the drug in the drug release systems.

Moreover, Tellurium plays a vital role in living organs due to outstanding biological, structural, visual properties, it has been analyzed as nanotechnological particles. However, not enough research on the application of biomedical sectors was conducted yet. For this reason, Tellurium glass is on a trend topic in biomedical engineering, biomedical science, medical science and so on [18,19].

2.8. PHOTONIC APPLICATIONS OF TELLURIUM BASED GLASSES

It had been more than three decades, researchers had been using tellurium glass for optics, photonics, laser, optical communication, fiber optics, quantum dot applications, white LED and so on [20]. Since it has been proved that these type of materials have amazing refractive index, good transparency, high energy band gap and many other optical characteristics [21].

E. Vogel from Bell Labs have designed a glass fiber for fiber optics application with a very high level gain where in the synthesis of the glass, rare earth materials were doped in the glass network [21]. Another optical fiber amplifier was developed at NTT, Japan utilizing tellurium glass materials for photonics and quantum computing applications [21,22].

2.9. OPTICAL AND MECHANICAL APPLICATIONS OF TELLURIUM BASED GLASS MATERIALS

In recent days, Tellurium oxide based optical glass materials have been a trend in the field of fiber optics communication and photonics technology. These tellurium oxide based materials have some outstanding properties like high refractive index, lower photon energy, high transmission rate and so on. These materials have excellent optical properties as well as mechanical properties. At 2015, H. Masai et al. have proposed a boron based glass for photoluminescence properties which has achieved outstanding performance for quantum communication [23].

At 2017 I. Kabalci et al. developed a tellurium based optical glass where aluminium oxide enhanced the hardness and structural properties [1]. Again at 2017, I. Kabalci. et al. have proposed ytterbium doped boron oxide glass materials for optical application which found to be efficient for thermal, hardness characteristics [24].

Furthermore, P. Kostka. et. al. have developed an erbium doped boron glass for photoluminescence applications [25].

E.A. Mohamed et al. have developed a lithium based nano glass for dielectric and ferromagnetic properties analysis [26]. Swapna et al. have studied vanadium doped tellurium based glass materials. They have studied the optical properties, structural properties and morphological analysis to evaluate the structural properties, cut off wavelength, molar volume, optical basicity, refractive index and electron polarizability [27]. K. Suresh et al. has studied the photoluminescence properties of ytterbium doped tellurite based glass materials using melt quenching methods for the application of solar cell. They have studied the optical properties, optical absorption, fluorescence, refractive index and glass density and quantum efficiency of the glass network [28].

In this research, a unique glass network combined with $70TeO_2$-$15ZnCl_2$–$10Nb_2O_5$–$5TiO_2$ was synthesized for the investigation of optical, thermal and structural properties based on temperature. The various types of thermal parameters were obtained through DTA analysis. Optical properties specially UV-VIS spectra was examined within the range of 190nm to 900nm. The electronic energy band gap was calculated applying Beer-Lambert Law. Furthermore, The crystalline properties, hardness properties, FT-IR spectroscopy and SEM were analyzed for structural morphology of the glass network.

PART 3

MATERIALS AND METHODS

In this study, the material samples, glass sample preparation, chemicals and analytical experiments used in the preparation of optical glass are given explained respectively.

3.1. RAW MATERIALS

The chemicals used in this experiment for the reagent grade of TeO_2, $ZnCl_2$, Nb_2O_5 and TiO_2. All these chemical components were 99.999% metal basis collected from Sigma Aldrich chemical science corporation. These are widely used as high purity commercial powders.

3.2. METHOD

A set of optical glass materials have been prepared following the general melt quenching method. The glass samples marked as TZNT1, TZNT2, TZNT3, TZNT4 and TZNT5 were prepared for the experiment. Each sample contains a total mass of 10 grams. The formula for the glass sample was $70TeO_2$-$15ZnCl_2$–$10Nb_2O_5$–$5TiO_2$.

In this method, based on the melting point of the components of the raw material mixture, samples were melted for a while at a production temperature of 750°C -1000 °C. After melting the glass samples, it was annealed at 300°C for 24 hours.

The resulting glasses are then different methods such as sawing, grinding and polishing required for characterization techniques it is prepared with. The vast majority of glasses are on conventional melt quenching it is produced by this method.

3.3. SAMPLE PREPARATION

All glass samples with a size of 10 gram are at different temperature ranging between 750°C to 1000°C. It was mixed properly and then melted in a furnace named as Protherm electric arc furnace.

Figure 3.1. ProTherm Furnaces at Ceramics Department,
Fine Arts Faculty, Usak University

After that, the melts were collected into a steel plate and it was quenched immediately by other steel plate. Furthermore, the glass samples were annealed at 300°C for 24 hours.

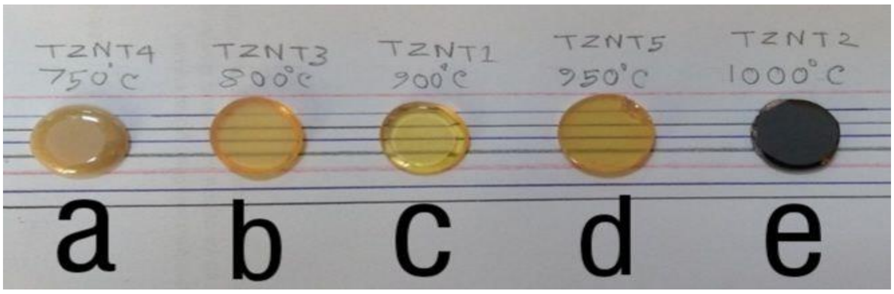

Figure 3.2. Prepared glass samples labeled as
TZNT1, TZNT2, TZNT3, TZNT4 and TZNT5.

For the synthesis of the optical glass, the production temperature of the three glass samples TZNT1, TZNT2, TZNT3, TZNT4 and TZNT5 were 900°C, 1000°C, 800°C, 750°C and 950°C. During the synthesis of the optical glass materials, each samples powder composites were mixed thoroughly and melted in a platinum crucible in an Electrical Arc Furnace model name, Pro Therm Furnaces. For the sample TZNT1, the sample was heated at 900°C where the temperature increasing rate was 10 °C/min upto 900°C and waiting an additional one hour inside the furnace it is prepared with. Then the melted glass was quenched in air usig stainless steel block at room temperature.

Similarly, for the sample TZNT2, the sample production temperature was 1000°C, for the sample TZNT3, the production temperature was 800°C. For the sample TZNT4, the sample production temperature was 750°C and for the sample TZNT5, the production temperature was 950°C.

3.4 MEASURED DEVICES

Throughout the study, the following devices had been used to accomplish the experimental analysis.

1. **DENVER INSTRUMENT SI 234** (to measure the weight of the chemicals)
2. **PROTHERM FURNACES** (for preparation of the glass)

3. **PERKIN ELMER SPECTRUM TWO FT-IR SPECTROPHOTOMETER 102945** (to analyze FTIR experiment)
4. **DTA HITACHI STA 7300** (to analyze DTA)
5. **QUORUM Q150R ES ROTARY PUMPED SPUTTER COATER** (for making gold layer on the sample for SEM analysis)
6. **CARL ZEISS ULTRA PLUS GEMINI FESEM** (for SEM analysis)
7. **RIGAKU ULTRA IV XRD** (for XRD analysis)
8. **PERKIN ELMER LAMBDA 25 UV-VIS SPECTROPHOTOMETER** (to analyze UV-VIS)
9. **QNESS HARDNESS MEASUREMENT DEVICE** (to measure micro vicker hardness of the glass samples)
10. **MITUTOYO DIGITAL MULTIMETER 293-821-30** (to measure the thickness of the samples)

3.5. DENSITY MEASUREMENT

The density of the glass depends on the ratio of the components of the glass. The buoyancy method, the Archimedes principle and the pycnometric method are mostly used method for calculating the density of glass materials.

Figure 3.3. Density measurement using Pycnometric method at Chemical Engineering Laboratory, Faculty of Engineering, Usak University.

It is one of the main density measurement methods. The sample is first taken in measurement at the air environment, then fast and reliable measurement based on weighing in a liquid with a known density. This method is known as the Archimedes principle. In this measurement method, the liquid is usually water and the measurement is carried out using a silver or platinum wire. It is usually measured at room temperature using the density meter + or - 0.001 g/cm3 based on the standard margin of error, it is measured. The adhesion of air bubbles to the sample need to be removed during measurements because it has a low surface tension, which prevents it from being used as a lifting fluid distilled water is usually used.

3.6. SCANNING ELECTRON MICROSCOPE (SEM)

Scanning Electron Microscope is used to find out the crystallizations on both the surface and cross sections of glass samples and it is used to investigate crystallization processes. The size, shape, composition, crystal structure, other physical and able to reveal microscopic scale information about its chemical properties and it is a tool that can create enlarged images of the sample.

Figure 3.4. Scanning Electron Microscope at Iron and Steel Institute at Karabuk University.

3.7. UV-VIS SPECTRA

The basic working mechanism of ultra violet spectroscopy is shown in Figure 3.3. Sample characteristics. Depending on the specific wavelengths, it reflects or absorbs, while the rest by measuring the transmitted or reflected intensity value, the reflection and it is used in determining the permeability values.

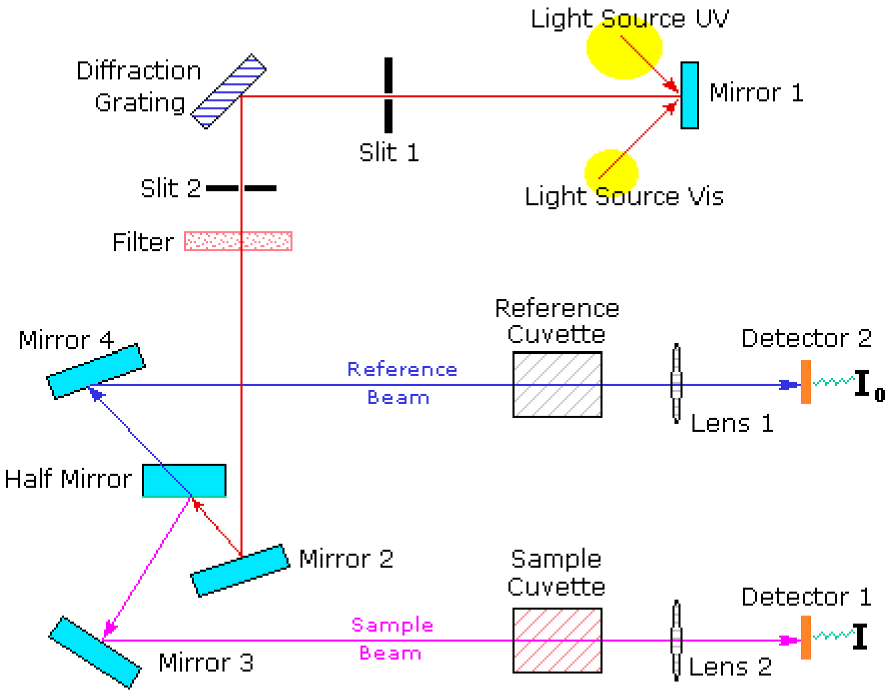

Figure 3.5. The basic principle of operation of the UV-VIS device [29].

Figure 3.6. The Perkin Elmer Lamda 25 UV-VIS spectrometer.

3.8. FOURIER TRANSFORM INFRARED PHOTOMETER (FT-IR)

Rays sent from the source found in Fourier transform infrared spectroscopy after passing through the sample, it is reassembled and the optical glass is decoded inside the sample. Certain characteristic frequencies of the sample are absorbed by the molecules it produces an absorption spectrum. The detector collects these signals and each it stores the spectrum in different locations. The resulting spectra are then called Fourier Transform, the analysis of the obtained spectra is found as output are made.

Figure 3.7. Parkin Elmer spectra FT-IR Spectrophotometer at Usak University [30].

Since calculations can be easily performed on modern computers since it is also quite easy to perform the Fourier transform, the spectra are very quickly it can be produced. Thus, it is possible to obtain better and faster data because most of the modern commercial infrared spectrometers are FT-IR spectrometers.

3.9. DIFFERENTIAL THERMAL ANALYSIS (DTA)

Thermal characteristics of glass materials are determined through DTA analysis. Its thermal properties will be investigated as shown in figure 3.6. below the material is thermodynamically exothermic with respect to any reference material and the endothermic parameters are determined according to the increasing temperature. For this process, the most known way is to determine the melting temperature. To detect the temperature changes and DTA is used to better understand the crystallization process. DTA; the temperature difference between the sample and the reference is reduced in time. Normally air is used as a reference material in DTA analysis for glass and ceramics.

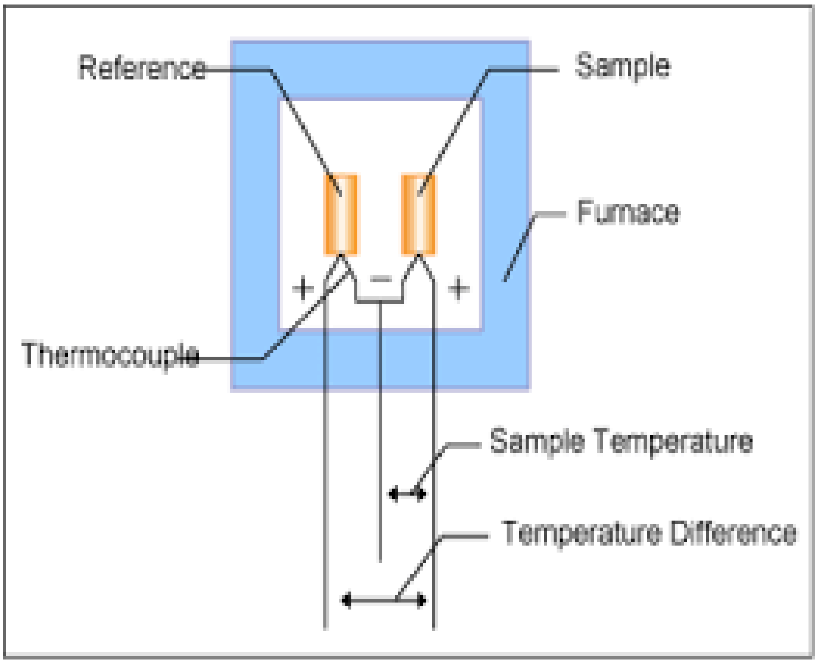

Figure 3.8. Working principle of DTA [31].

The difference in temperature versus time or temperature within a given atmosphere it is an analysis technique in which it is observed.

Figure 3.9. Differential Thermal Analysis device at
Iron and Steel Institute, Karabuk University.

DTA curves; glass transitions, crystallization, melting it provides information about temperatures such as system; sensors, amplifier, temperature sensor of the furnace and oven, computer and recording device or data it consists of a collecting device.

3.10. X-RAY DIFFRACTION (XRD)

The crystalline morphology of samples, thin films and nanoparticles has a highly accurate it allows it to be determined in the way. From a monochromatic source of X-rays it is composed of.

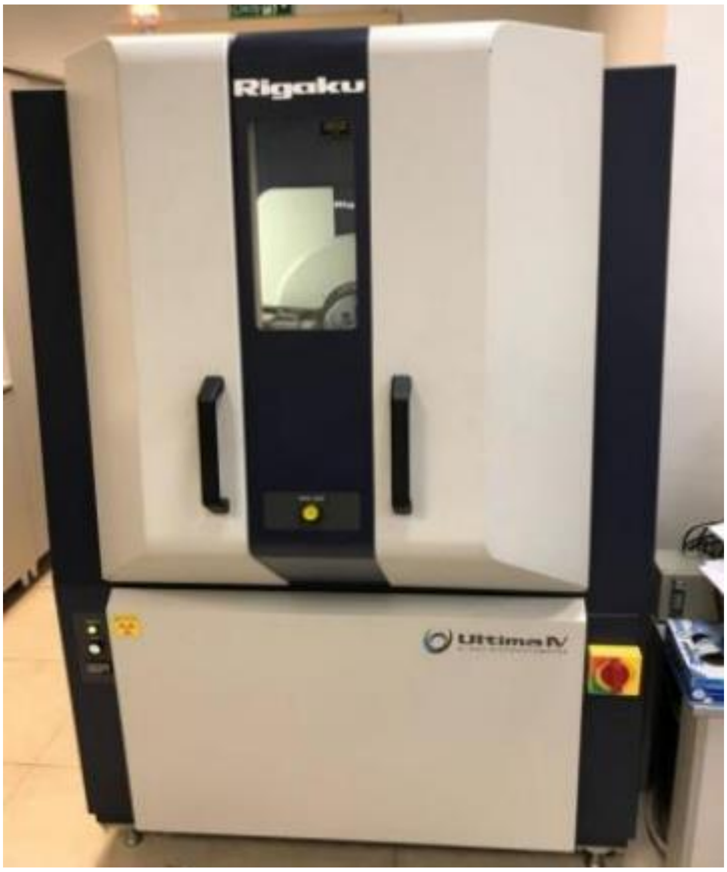

Figure 3.10. X-Ray Diffraction device at Iron and Steel Institute at Karabuk University.

3.11. MICRO VICKER HARDNESS

Microvicker hardness is one of the most popular method to define the hardness of glass, ceramics, metal, composite materials and many others. At first, imprint was taken of the sample in the micro hardness system which uses an intelligent machine vision technology. In case of cracks of the indentation found, then it is allowed to determine the resistance of the crack and diagonal length of the sample. Different scale levels including nano scale system is allowed by the system [32].

Figure 3.11. Micro Vicker Hardness Device at
Iron and Steel Institute at Karabuk University.

PART 4

RESULTS AND DISCUSSIONS

The glass samples labeled as TZNT1, TZNT2, TZNT3, TZNT4 and TZNT5 obtained in this study $70TeO_2$-$15ZnCl_2$-$10Nb_2O_5$-$5TiO_2$ glass network using the conventional melt quenching at room temperature in varying the production temperature. The production temperature of the five glass samples TZNT1, TZNT2, TZNT3, TZNT4 and TZNT5 were 900°C, 1000°C, 800°C, 750°C and 950°C. It was prepared using the extinguishing method. Among the obtained glass samples, three of them are are transparent, brown, homogeneous distribution and not brittle while the rest two became crystalline.

4.1. UV-VIS ANALYSIS

In the field of solid state physics and electronics, optical absorption in the UV regime is a very effective tool for defining the optical transitions and electronic band gap properties. Optıcal absorptions are mainly two types, direct energy band gap and indirect energy band gap. Deriving Tauc equation is a popular way to measure the optical band gap.

$$(\alpha h v)^{\gamma} = A(hv - Eg) \qquad [33]$$

here α is known as absorption coefficient, h is planck’s constant, ν is photon frequency, A is constant of proportionality and E_g is band gap.In case of direct transition, γ is considered to be two, on the other hand, in case of indirect transition, γ is considered to be half. Following the equation Beer-Lambert law, absorption coefficient can be calculated.

$$\alpha(w) = \frac{ln(Io/I)}{l} \qquad [34]$$

In the wavelength region of 410nm-900nm at room environment, the optical absorption of the glass samples was analyzed.

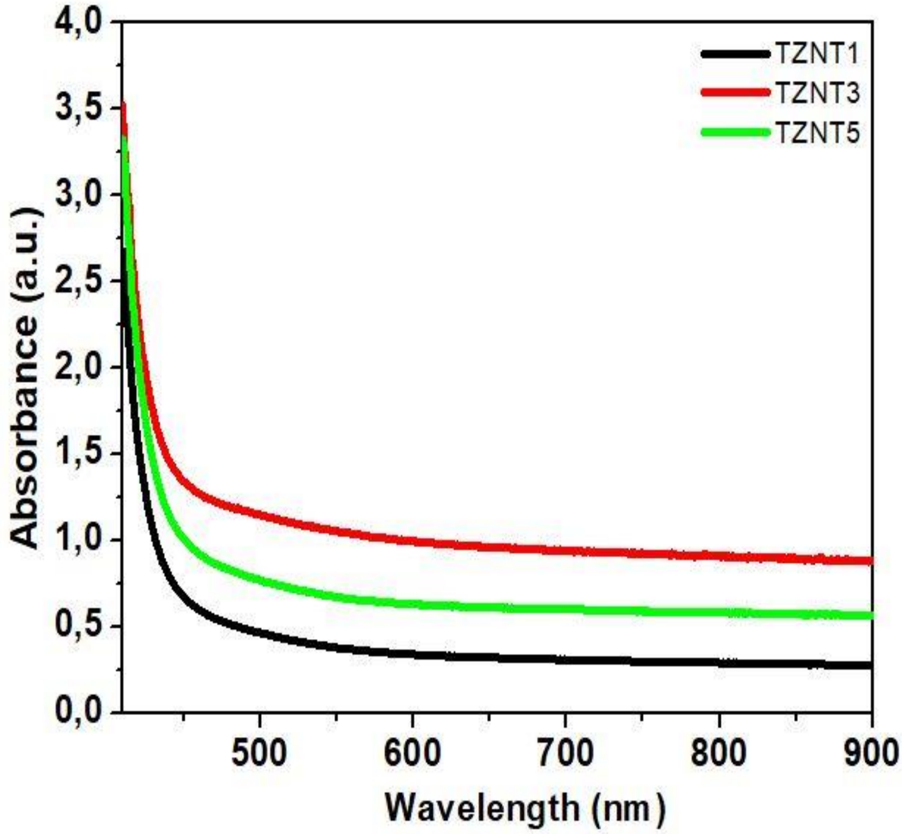

Figure 4.1. UV-VIS absorbance of three samples TZNT1, TZNT3 and TZNT5.

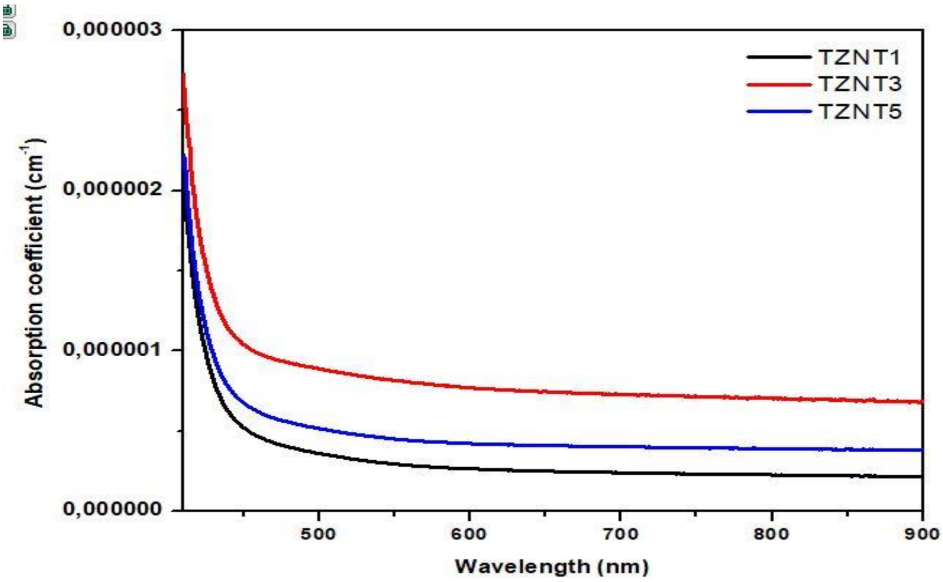

Figure 4.2. UV-VIS absorption coefficient of three samples TZNT1, TZNT3 AND TZNT5.

The energy band gap of the samples was defined using Tauc equation which is represented below.

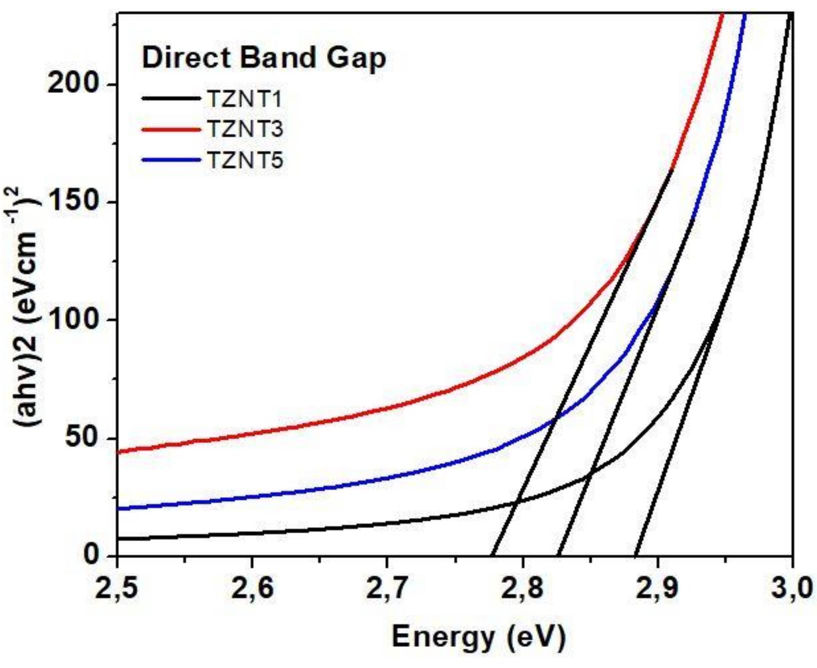

Figure 4.3. Direct band-gap of the glass samples labeled as TZNT1, TZNT3 and TZNT5.

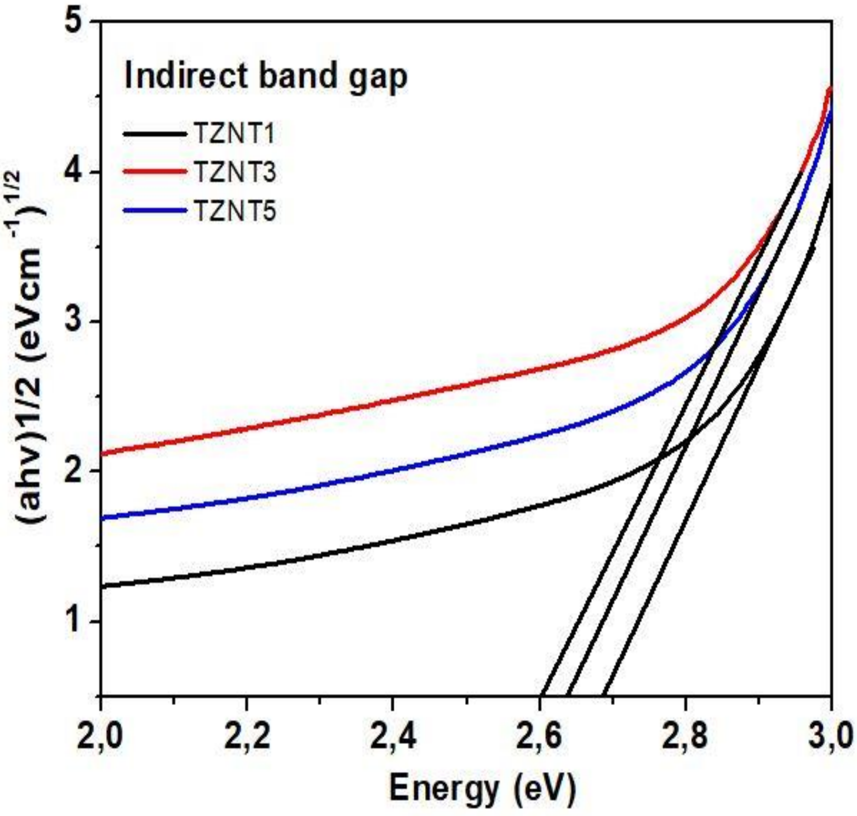

Figure 4.4. Indirect band-gap of the glass samples
labeled as TZNT1, TZNT3 and TZNT5.

4.2. DIFFERENTAL THERMAL ANALSIS (DTA)

DTA was examined within the temperature region 20°C to 800°C where the rate of heating was set up to be 10°C. The DTA plot is shown below figure 4.4 to determine the exothermic and endothermic reactions.

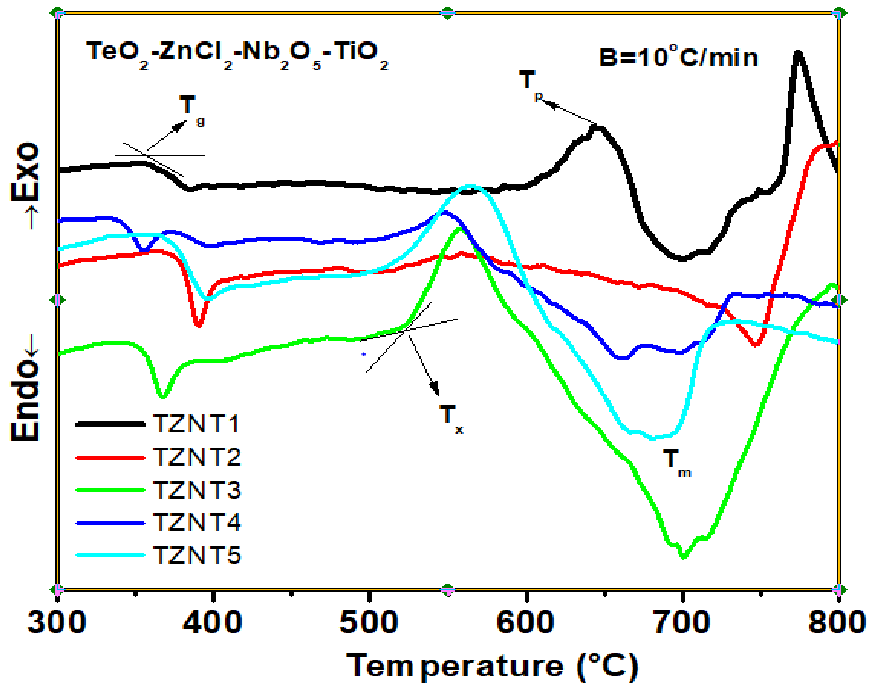

Figure 4.5. DTA analysis of the prepared glass samples.

4.3. SCANNING ELECTRON MICROSCOPE (SEM)

The surface morphology of the heat-treated samples is determined by any in order to determine the topography of the surface without etching, SEM is used to examine this.

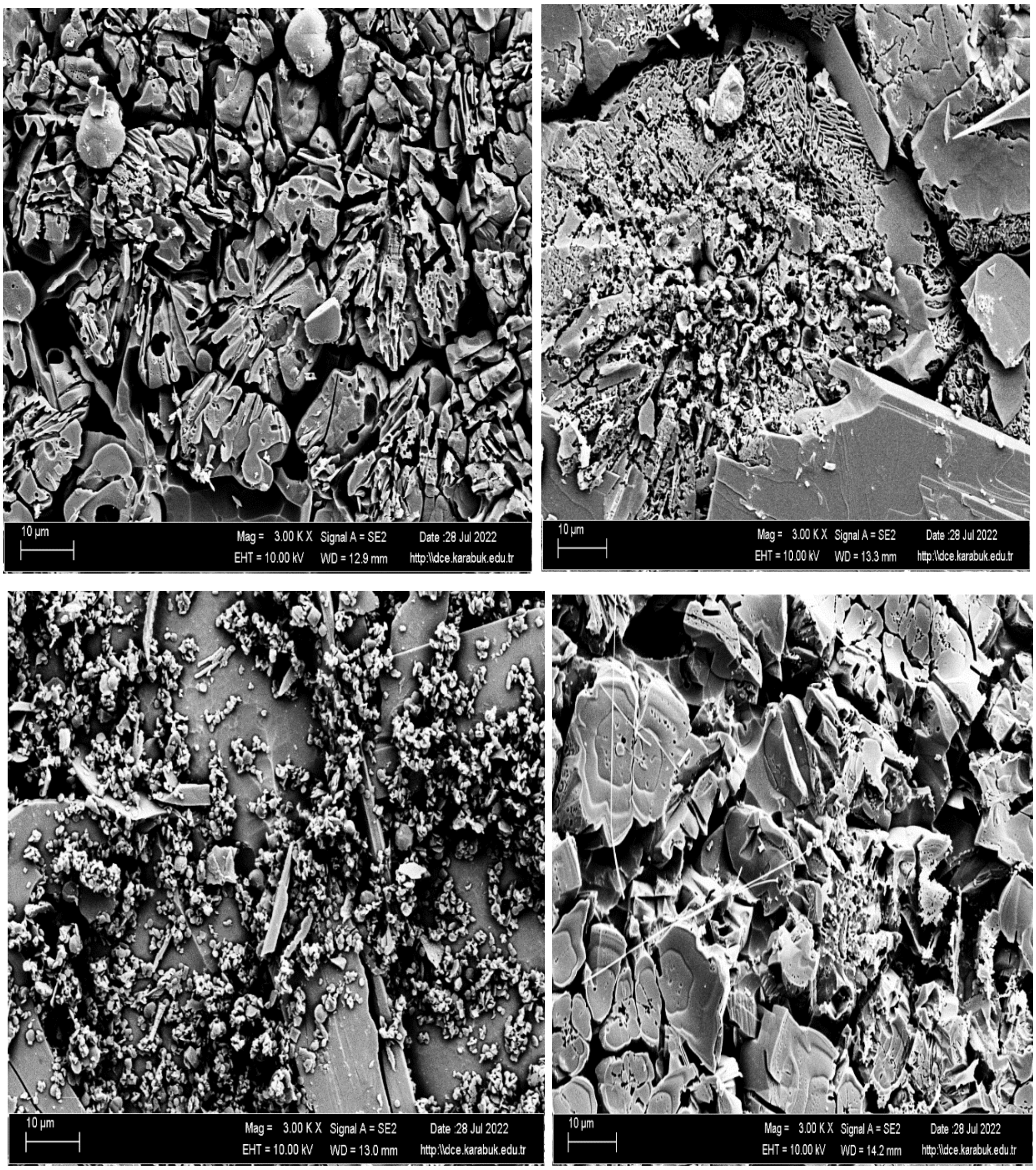

Figure 4.6. SEM images show the topographical differences of the samples TZNT1, TZNT2, TZNT3 and TZNT4 at 10μm.

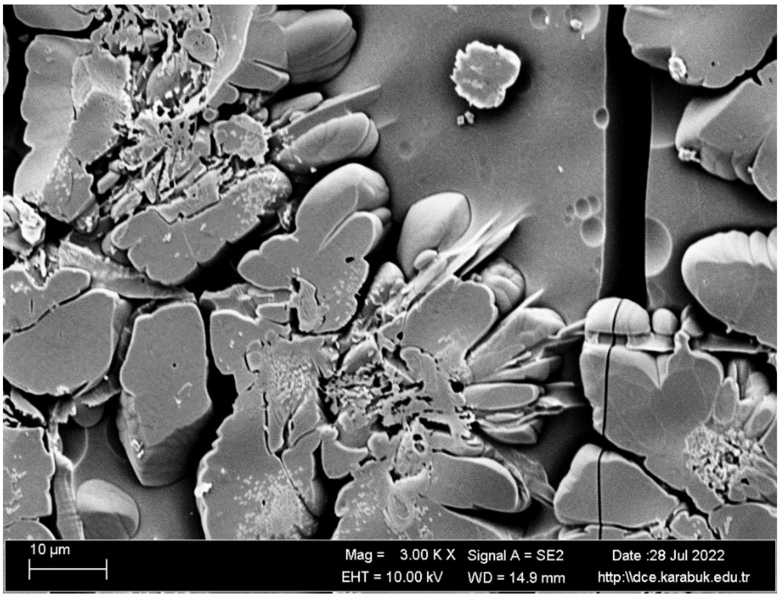

Figure 4.7. Topographical image of the sample
TZNT5 at 10μm.

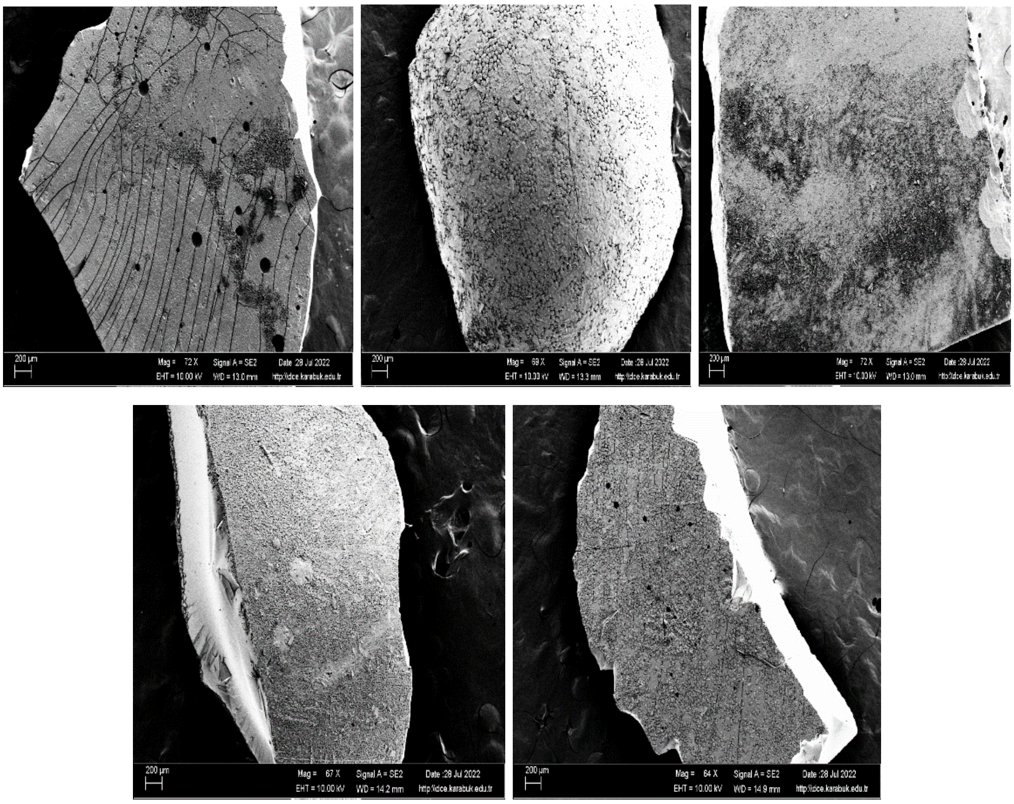

Figure 4.8. Topographical image of the sample
TZNT1, TZNT2, TZNT3, TZNT4 and TZNT5 at 200μm.

4.4. FOURIER TRANSFORM INFRARED SPECTROPHOTOMETRY(FT-IR)

Based on the data obtained from the thermal analysis, the glass samples TZNT1, TZNT2, TZNT3, TZNT4 and TZNT5 were melted in 500°C,500°C,610°C,650°C and 500°C for the preparation of the FT-IR analysis.

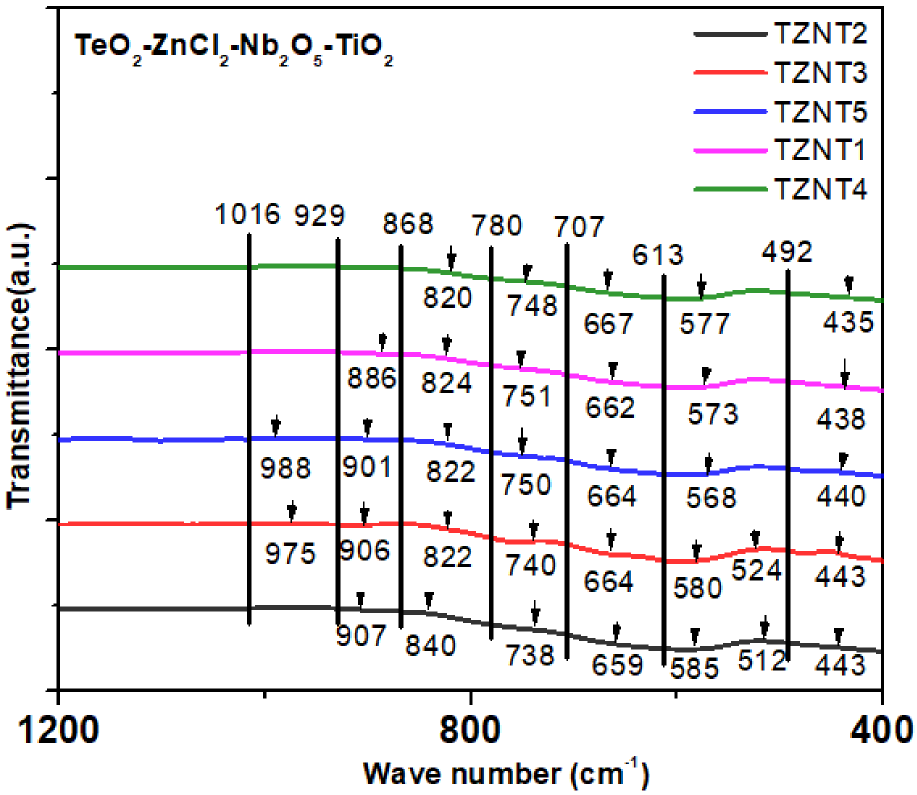

Figure 4.9. FT-IR plot of the five samples
TZNT1, TZNT2, TZNT3, TZNT4 and TZNT5.

4.5. X-RAY DIFFRACTION (XRD)

Based on the data obtained from the thermal analysis, the glass samples TZNT1, TZNT2, TZNT3, TZNT4 and TZNT5 were melted in 500°C,500°C,610°C,650°C and

500°C for the preparation of the XRD analysis. The glass was found to be have two crystal phase.

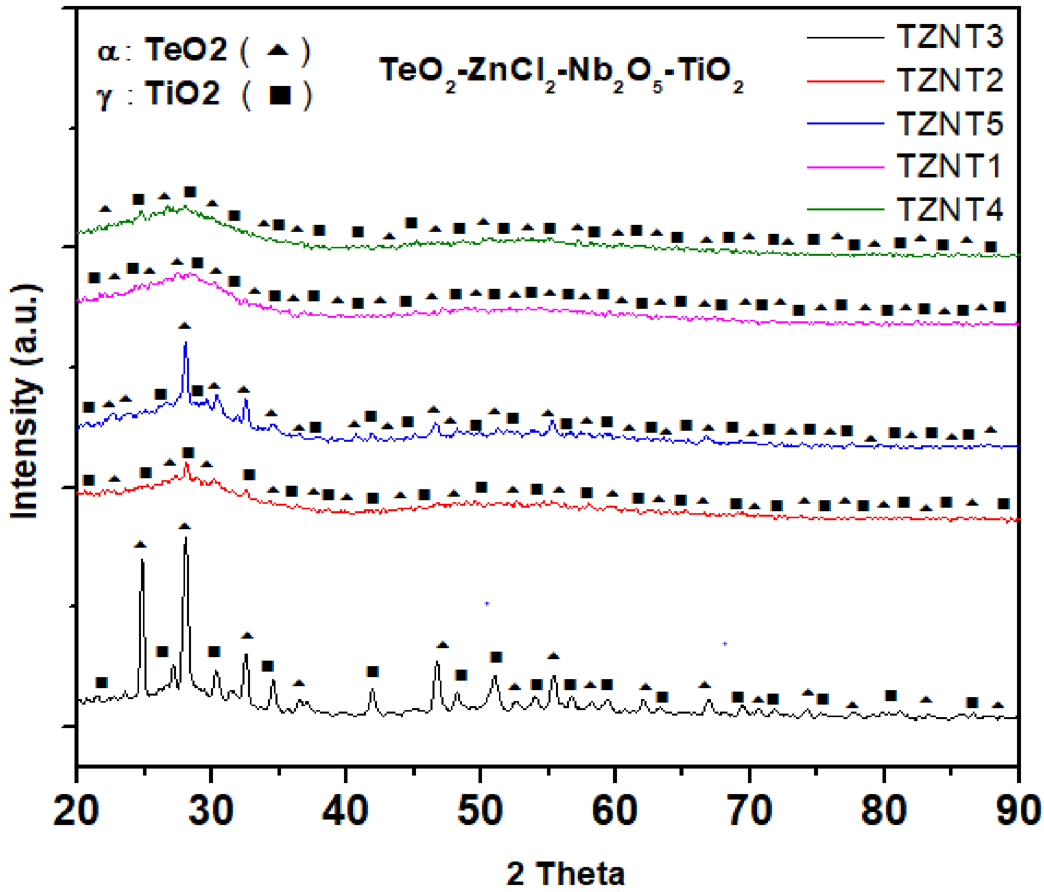

Figure 4.10. XRD of the glass samples
TZNT1, TZNT2, TZNT3, TZNT4 and TZNT5.

From the XRD analysis, two kind of crystalline phase was found, one is TeO_2 which is denoted to be alpha and the second one is TiO_2 which is denoted to be gamma. According to the graph, while increasing the amount of $ZnCl_2$ or Nb_2O_5 components, sharp peak got found which means the sample TZNT4 becomes much more amorphous in comparison with TZNT3, TZNT5, TZNT1 and TZNT2.

4.6. MICRO VICKER HARDNESS

Micro Vicker Hardness was tested for the samples TZNT1, TZNT2, TZNT3, TZNT4 and TZNT5. Among these samples, TZNT1, TZNT3 and TZNT5 glasses who were transparent have passed the test successfully. The first sample TZNT1 was proven to be the hardest among all five glass samples. The comparison result is shown below.

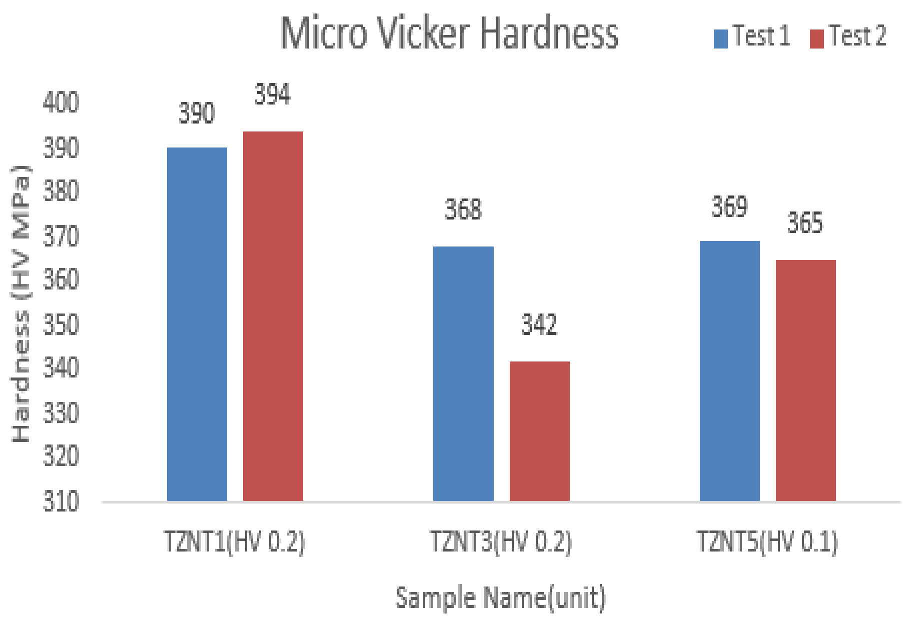

Figure 4.11. Micro Vicker's Hardness bar graph plot of three samples TZNT1, TZNT3 and TZNT5.

It is seen that he has the samples TZNT1, TZNT3 and TZNT5 were found to be hard as they passed the micro vicker hardness test. Among them, TZNT1 have highest micro vicker hardness result 394 HV 0.2 MPa. The second micro vicker hardness result goes to TZNT5 which is HV 369 0.1 MPa and the third one goes to TZNT3 which is HV 368 0.2 MPa.

PART 5

SUMMARY

Within the scope of this thesis, the designed optical glass samples, $70TeO_2$-$15ZnCl_2$–$10Nb_2O_5$–$5TiO_2$, in the glass system, in varying different production temperature where the production temperature of the samples labeled as TZNT1, TZNT2, TZNT3, TZNT4 and TZNT5 were 900°C, 1000°C, 800°C, 750°C and 950°C. The samples were made ready using the usual melt quenching method. Three of them TZNT1, TZNT3 and TZNT5 were obtained are transparent, colorless, homogeneous and non brittle while the rest TZNT2 and TZNT4 became crystal.

Differential thermal analysis (DTA), X-ray diffraction (XRD), Scanning Electron Microscope (SEM), Fourier Transform Infrared Ray spectroscopy (FT-IR), UV-VIS Spectra systematic experimental measurements such as absorption analysis, energy band gap analysis were performed. By the traditional melt quenching method of $70TeO_2$-$15ZnCl_2$–$10Nb_2O_5$–$5TiO_2$ glass network optical and structural properties were determined in this study.

In brief overview of the research,

1. According to the XRD analysis, the obtained glass samples are two crystal phases, TeO_2 and TiO2 phases.
2. The resulting glass TZNT1, TZNT3 and TZNT5 materials show good transparency with high optical properties. As these optical glass samples have high transparency, these features that could potentially be used in photonic and optical communication system and quantum computing.
3. Based on the thermal analysis, the sample TZNT5 have showed a tremendous thermal characteristics including exothermic and endothermic reaction.

4. It is seen that he has the samples TZNT1, TZNT3 and TZNT5 were found to be hard as they passed the micro vicker hardness test. Among them, TZNT1 have highest micro vicker hardness result 394 HV 0.2 MPa.
5. Such optical materials with the resulting high transparency are a very potential candidate materials for optical fiber communication, laser, photonics, medical imaging, optoelectronics, photonic crystal fiber, white LED application, quantum computing applications and many others.

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RESUME

Taufiq ABDULLAH has graduated first elementary education in Chuadanga district, Bangladesh. He completed high school education in Chuadanga Govt. College, Chuadanga. After that, he has completed undergraduate program in Electrical and Electronic Engineering from Bangabandhu Sheikh Mujibur Rahman Science and Technology University, Bangladesh at 2018. Then in 2020, he started assignment as a Graduate Research Assistant in Karabuk University Department of Biomedical Engineering. To complete M. Sc. education, he moved to Karabük University, where he has been still working as a R. A. for. Optical Glass Materials Lab.