SYNTHESIS, CRYSTALLIZATION KINETIC AND OPTICAL PROPERTIES OF EUROPIUM DOPED TELLURITE NANO - GLASS

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This thesis is specially dedicated to:

My beloved parents,
Sazali Idris, Zaini Zainuddin

My supportive siblings,
Addam, Lilli, Dianna, Djohan

My dedicated lecturers,

My endless spirits

and all my friends.

.....thanks......

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ABSTRACT

Tellurite glass of composition (80-x) TeO_2 – 5 Na_2O – 15 MgO – (x) Eu_2O_3, with the concentration region of $0 \leq x \leq 2.5$ mol% was prepared using conventional melt-quenching technique. The transition temperature, T_g, crystallization temperature, T_c and melting temperature, T_m were determined using Differential Thermal Analysis (DTA). The glass with nano-crystalline particle was prepared by heating the as-cast glass at temperature 15 - 20 ^oC above the T_c. The X-Ray Diffraction (XRD) technique with the Scherrer equation was used to determine the size of nano- crystalline particle of the samples while the Scanning Electron Microscopy (SEM) technique was used to identify the formation of nano-crystalline phase. The crystallization kinetics was investigated by using DTA at various heating rate while the glass density and hardness was determined by Precisa Densitometer and Vickers microhardness, respectively. Meanwhile, the emission characteristic was evaluated using the Photoluminescence Spectroscopy (PL). The thermal analysis showed that the T_g and T_c increased with the increasing of Eu^{3+} content while T_m showed a break in linearity as the Eu^{3+} increased. The result also showed that the glass stability up to 109.29. XRD spectra confirmed the presence of nano particles with an average diameter around 68.7 nm. Meanwhile the SEM studies revealed the existence nano-crystalline morphology which was associated with the existence of crystallized phase. The influence of Eu^{3+} content on crystallization kinetics showed a shift of crystallization temperature peak towards a higher temperature with the increasing of heating rate as described by Kissinger and Ozawa method. The activation energy (E_a) was found to decrease from 306.9 eV to 48.9 eV with an increasing of dopant concentration. The density was found to be in the range of 5.234 to 5.334 g cm^{-3} while the Vickers microhardness was found to vary from 2.59 to 2.84 GPa depending on the dopant concentration. A detailed study on the luminescence spectra showed that all emission peaks for $^5D_0 \rightarrow {}^7F_0$, $^5D_0 \rightarrow {}^7F_1$, $^5D_0 \rightarrow {}^7F_2$, $^5D_0 \rightarrow {}^7F_3$ and $^5D_0 \rightarrow {}^7F_4$ transitions were found to be around 568 nm, 600 nm, 628 nm, 664 nm and 712 nm, respectively. It was also found that the heat-treated glass and the inverse quality factor decreased with increasing Eu^{3+} dopant concentration.

ABSTRAK

Kaca tellurite dengan komposisi (80-x) TeO_2 – 5 Na_2O – 15 MgO – (x) Eu_2O_3, mempunyai kepekatan $0 \leq x \leq 2.5$ mol% telah disediakan menggunakan teknik pelindapan leburan konvensional. Suhu peralihan, T_g, suhu penghabluran, T_c dan suhu lebur, T_m ditentukan menggunakan analisis kebezaan terma (DTA). Kaca dengan hablur nano diperolehi dengan memanaskan kaca pada suhu 15 - 20 ^oC di atas suhu penghabluran, T_c. Teknik pembelauan sinar-X (XRD) dan persamaan Scherrer digunakan bagi menentukan saiz zarah nano hablur manakala teknik pengimbas mikroskopi elektron (SEM) digunakan bagi mengenal pasti pembentukan fasa nano hablur. Kinetik penghabluran dikaji dengan menggunakan DTA pada kadar pemanasan yang berbeza manakala ketumpatan dan kekerasan kaca masing-masing ditentukan dengan menggunakan Precisa Densitometer dan kekerasan mikro Vickers. Sementara itu, pancaran dinilai dengan menggunakan spektroskopi kefotopendarcayaan (PL). Untuk analisis terma, T_g dan T_c meningkat dengan peningkatan Eu^{3+} sementara T_m tidak menunjukkan peningkatan linear denagn penambahan kepekatan Eu^{3+}. Keputusan juga menunjukkan bahawa julat kestabilan kaca sehingga 109.29 ^oC boleh dicapai. Spektrum XRD membuktikan kewujudan zarah hablur nano dengan saiz purata diameter 68.7 nm. Sementara itu, kajian SEM membuktikan morfologi hablur nano yang boleh dikaitkan dengan kewujudan fasa hablur. Pengaruh kandungan Eu^{3+} pada kinetik penghabluran menunjukkan peralihan suhu puncak penghabluran ke arah yang lebih tinggi dengan peningkatan kadar pemanasan seperti diterangkan menggunakan kaedah Kissinger dan Ozawa. Tenaga pengaktifan (E_a) didapati menurun daripada 306.9 eV hingga 48.9 eV dengan peningkatan kepekatan pendopan. Ketumpatan kaca didapati berada dalam julat 5.234 hingga 5.334 g cm^{-3} manakala kekerasan mikro Vickers dari 2.59 hingga 2.84 GPa bertambah mengikut kepekatan pendop. Kajian yang teliti ke atas spektrum pendarcayaan menunjukkan bahawa semua pancaran bagi transisi $^5D_0 \rightarrow {}^7F_0$ $_5D_0 \rightarrow {}^7F_1$, $_5D_0 \rightarrow {}^7F_2$, $_5D_0 \rightarrow {}_7F_3$ dan $^5D_0 \rightarrow {}_7F_4$ masing-masing didapati berada di sekitar 568 nm, 600 nm, 628 nm , 664 nm dan 712 nm. Didapati juga bagi kaca yang dirawat haba, faktor kualiti songsang menurun dengan pertambahan kepekatan Eu^{3+}.

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LIST OF ABBREVIATIONS

bp	-	Bipyramid
BO	-	Bridging oxygen
DTA	-	Differential Thermal Analysis
FWHM	-	Full width half maximum
g	-	gram
IR	-	Infrared
NBO	-	Non-bridging oxygen
NIR	-	Near infrared
nm	-	nanometer
PL	-	Photoluminescence
SEM	-	Scanning electron microscopy
tbp	-	Trigonal bipyramid
XRD	-	X-ray diffraction

LIST OF SYMBOLS

α	-	Crystallization fraction
b	-	Heating rate
d	-	Atomic spacing
β	-	Full width half maximum
d	-	Sample thickness
E_a	-	Activation energy
ΔE	-	Free energy difference per unit volume
ΔJ	-	Electric dipole
k	-	Planck's constant
K_g	-	Glass forming tendency
m	-	Mass
M	-	Molecular weight
n	-	Avrami parameter
ρ	-	Density
ρ_a	-	Air density
ρ_l	-	Liquid density
Q	-	Quality factor
Q^{-1}	-	Inverse quality factor
R	-	Universal gas constant
S	-	Thermal stability
ΔS	-	Spin forbidden emission band
t	-	Time
T	-	Temperature
T_c	-	Crystallization temperature
T_d	-	Developed temperature

T_g	-	Glass transition temperature
T_m	-	Melting temperature
T_n	-	Nucleated temperature
U	-	Growth rate
V_m	-	Molar volume
W_a	-	Weight of sample in air
W_l	-	Weight of sample in immersion fluid
v_o	-	Frequency
θ	-	Angle
λ	-	Wavelength
λ_{exci}	-	Excitation wavelength

CHAPTER 1

INTRODUCTION

1.1 Background of the Study

Every definition of glass defines it as an amorphous solid. Meanwhile, as a non-crystalline solid, glass can be defined as inorganic material that produced by melting process then annealed it to solid state or to a rigid condition without crystallization process (Rawson, 1980). Glasses can also be described as supercooled liquid or as a solid and therefore have a finite viscosity of ambient temperatures (Zachariasen, 1932). In addition, as a rigid material, glass does not flow when it subjected to moderate forces (Doremus, 1973). The difference between glass and crystal is that it possesses lack of long-range order therefore random in the arrangement of its molecular structure while crystal is otherwise. According to Bengisu (2001), glass are built up of random clustered of ordered atoms which contributes to some aspects of a crystalline solid but its random atomic structure and short range orders is actually a characteristic of a liquid.

The viscosity of glass is in the range of 10^{14} Poise (P) and always presented the short-range order when imposing the X-Ray beam (Sahar, 1998; Santos *et.al*, 2001). It flows at an incredibly slow rate. It has been said that glass could only flow if some considerations are ignored as it forms without any continuous where a viscosity of the material is increased (Clare *et.al*, 1991; Holloway, 1973). Some of them are of commercially useful compositions and in fact is a rigid solid at ordinary temperatures (Uhlman *et.al*, 1980). Since then, glasses are technologically important as they arise much interest. If compared with other materials, glass exhibit various behaviour as they have their own distinguishable properties. They have no reaction or difficult to be influenced with atmospheric agent and environment. Besides, according to Sahar (1998), glass is not easily corroded. Some workers emphasized glass stability base on the crystallization rate during cooling or reheating (Gabrielson, 1957).

As glass are widely been used in many high technology devices, the development of glass properties is an important research area. The uses of tellurite glasses as a host material have incredibly increases as their applicable potentials in laser and fiber applications (Tanabe *et.al*, 1990). They have low melting temperature, low glass transition temperature and high thermal stability (Tanabe *et.al*, 1990). Furthermore, tellurite shows good transmission in the visible and the near – IR up to 4.5 µm which gave high contribution of optical devices (Burger *et.al*, 1992; Kowada, 1996; Sabadel *et.al*, 1997; Ravi *et.al*, 2001). Besides, the understanding of their microscopic mechanism gave much technical applications and fundamental interests for both academic and the industry (El-Mallawany, 2002). Thus, they have become increasingly important as a new optical functional material because of the low phonon energy and fast response time (Hao Liu *et.al*, 2006). The absence of hygroscopic properties makes tellurite glass is more applicable than phosphate and borate glasses (Sidek *et.al*, 2006). Hence, their corrosion resistance in various environments is of considerable technological importance (Singh *et.al*, 2007; Linda Lee *et.al*, 2007). Modifier such as Na_2O or Li_2O is necessary to add to TeO_2 because the melted material quickly recrystalized. Previous researcher reported that the modifying elements such as Mg, Zn, and Ba were chosen for their ability to aid in the formation of stable tellurite glasses (Nishida *et. al*, 1990). The concentration of about 10 mol% of sodium is necessary to increase the glass forming stability of the mixtures was proven by McLaughlin in 2000.

As tellurite glass are resistant to atmospheric moisture, this allows the incorporations of large concentration of rare-earth ions into the matrix (Nageno *et.al,* 1993). The rare earth doping into tellurium based glass has great implications on the property of the glass. Interest in tellurite glass containing rare earth element are expected for nonlinear optical devices as for their large third-order nonlinear optical susceptibility (Wang, 1994). Moreover, the assimilation of rare earth ions can stabilize the metastable crystalline phase which leads to a development of optical devices (Matthew, *et.al,* 2002; K. Hirano *et.al,* 2002).). Among all glasses, the Eu^{3+} doped tellurite glass has arrised great attention as they can perform persistent spectral hole burning in the $^7F_0 \rightarrow {}^5D_0$ transition (P. Giridhar *et.al,* 2000). The Eu^{3+} ion is also used as a probe for finding the local structure around the rare earth ion in a crystal or a glass due to relative simplicity of its energy level structure with non-degenerate ground $^7F_0$ and emitting $^5D_0$ states (P. Babu *et.al,* 2000). Hence, their synthesis and dynamic is of considerable technological importance especially for domestic applications (Singh *et.al*, 2007). The integration of rare-earth ions into some kinds of glasses elucidated the significance of rare earth ion-glass host interaction for engineering waveguide devices (Animesh *et.al,* 2007). All of these considerations comprise a powerful control feature for the fabrication of rare-earth doped tellurite glass.

Interestingly, these properties of tellurite glasses are easily influenced by structural effects and kinetics of crystallization. Understanding the crystallization behavior is very important in order to determine the ideal conditions to fabricate glasses for optical applications and for manufacturing glass ceramics (Animesh *et.al,* 2007). This contributes to determination of the stability of glass where the formation of crystal must be avoided (Ray and Delbert, 1990). By using the non-isothermal method, the physical parameters of temperature is studied in detail. In most cases the sample is heated at a fix rate rather than by using isothermal method (Mehta *et.al*, 2004). Moreover, the application of isothermal process is rarely used and usually proceeds under non-isothermal conditions (Zhang *et.al,* 1990). Thus, the determination of thermal characteristics is of current attention to this study. Reheating the glass produces a characteristic DTA curve (Pye *et.al,* 1972). The simple and yet useful method of using DTA for thermal analysis techniques may contribute to obtain the information about the nucleation and the crystallization mechanisms in such glass system (Elliot, 1983). This systematic study allows this study to understand the mechanism of crystallization kinetics in tellurite glass.

1.2 Statement of Problem

The synthesis of nano-crystalline Eu^{3+} doped TeO_2 based glass has been less extensive been reported and the growth dynamic of the glass system is yet unclear (Ravi *et.al*, 2001). The rare-earth doping into nano-crystalline glass has great implications over the properties of glass which expected to be used in nonlinear optical devices because of their large third-order nonlinear optical susceptibility (Holand *et.al,* 2002). Hence, their synthesis and dynamic understanding is of considerable technological importance (Hirano, *et.al,* 2002). However, According to the previous reported phase diagrams, the tellurite glasses can easily be obtained with a high concentration of europium (Watanabe *et.al,* 2000). Moreover, properties of Eu^{3+} doped TeO_2 glasses which undergo heat treatment process are studied only recently. Therefore, the aims of this study are to prepare the tellurite glass doped with europium (Eu^{3+}) via melt quenching technique. The nano-crystalline glass will be prepared by the heat-treatment process. The effect of dopant throughout all samples towards the physical, thermal, structural and optical properties will be investigated. This study will focus on the behaviors of crystallization kinetics of Eu^{3+} doped TeO_2 - Na_2O - MgO. The activation energy, E_a of crystal growth is deduced through Kissinger and Ozawa analysis under the non-isothermal condition. The kinetic study on crystallization of glass is further complemented by using Avrami exponent method. In addition, the optical properties are investigated using spectroscopy photoluminescence.

1.3 Objectives of the Study

The objectives of this study are:

i. To prepare the glass and heat-treated nano-glass based (80-x) TeO_2 - 5 Na_2O – 15 MgO - (x) Eu_2O_3.

ii. To determine the thermal properties of Eu^{3+} doped tellurite glass using Differential Thermal Analysis (DTA).

iii. To calculate the crystallizations kinetic of Eu^{3+} doped tellurite glass using DTA.

iv. To identify the crystallization phases and occurance of nano particles in Eu^{3+} doped tellurite glass using X-Ray Diffraction (XRD) and Scanning Electron Microscopy (SEM).

v. To study the physical properties of Eu^{3+} doped tellurite glass in terms of density and hardness.

vi. To investigate the optical properties of Eu^{3+} doped tellurite glass in terms of Photoluminescence (PL).

1.4 Scope of the Study

This study has been divided into several scopes as follows:

i. Preparation of tellurite glass and heat-treated tellurite glass of composition (80-x) TeO_2 - 5Na_2O - 15MgO - (x) Eu_2O_3 system which ($0.0 \leq x \leq 2.5$ mol %).

ii. Determination of thermal properties and calculation of the crystallization kinetics by implemented Kissinger, Ozawa and Avrami method using DTA.

iii. Identification of the crystalline nature of the occurrence of nano-particles in glass matrix using XRD and SEM.

iv. Determination of physical properties from density and hardness.

v. Investigation of the luminescence spectra of Eu^{3+} doped tellurite glass using Photoluminescence sspectroscopy.

1.5 Significance of Study

Among several compositions of glasses which were tested in this research, the glass which had the highest stable thermally phase will be determined. This result will imply that the crystallographic stability is enhanced in rare earth doped samples which is expected to give improvement in poor mechanical properties of tellurite glasses (Sidek *et.al*, 2006; Linda Lee *et.al*, 2007). Thus, this research is done to create more understanding on the nano-glass and to develop nano-structured amorphous in parallel with the development of nano technology. A clearer understanding of growth dynamic of the glass system has be employed. This is very important in order to understand the synthesis and crystallization behavior of the nanocrystalline glass system. Of particular interest is the Eu^{3+} luminescence in the yellow–red region that is frequently exploited for polychromatic displays. This luminescence, may be influenced by presence of nano-crystallized particle in the sample and shows the luminescence enhancement.

1.6 Thesis Plan

The thesis describes the preparation and characterization of europium doped tellurite based nano glasses prepared by melt-quenching technique. This thesis has been divided to five main chapters. Chapter 1 outlines the introduction part which are including general background of study and some reviews from previous work and describes the problem statements which led to this research, objectives, scope and the significance of study. Chapter 2 will describe all the literature reviews, current knowledge and related theories regarding the glass especially for tellurite based glass. The theories describes including the physical properties which are the density and hardness, thermal properties, structural properties, crystallization kinetics and optical properties in terms of absorption spectra and luminescence spectra. Next, Chapter 3 which is research methodology will explain all the experimental procedure including the sample preparation and the experimental techniques. In Chapter 4, the experimental results and discussion throughout this research will be explained in detail. Finally, Chapter 5 summarize all the experimental findings and conclusions.

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