

**THE HARDNESS AND THERMAL EXPANSION OF ERBIUM DOPED
TELLURITE GLASS**

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**In the name of Allah, Most Gracious Most Merciful
All praise and thanks are due to Allah Almighty and
peace and blessing be upon His Messenger
To my *Ayah, Umie, siblings and friends* for their prayers,
encouragement, sacrifice and especially for their love.
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ABSTRACT

A series of glass based on $(80-x) \text{TeO}_2 - 20 \text{ZnO} - x \text{Er}_2\text{O}_3$ where $0 < x \leq 2.5$ mol % was prepared using melt quenching technique. The amorphosity of the glass was determined by X-ray diffraction (XRD) and the physical properties such as density and hardness was determined by means of Archimedes and Vickers Hardness method respectively. The thermal expansion was measured using dilatometric method. The fine glass rods were prepared using rotary ultrasonic machine (RUM) with diamond rods tools of 4 mm in diameter. Meanwhile, the surface roughness of the rods was measured using profilometry technique. The result of XRD patterns confirmed that the glass was in amorphous phase. It was found that, the density of the glass increased with increasing Er_2O_3 content. The range of density was found to be between 5.541g/cm^3 and 5.663g/cm^3 . It was also found that as the amount of Er_2O_3 content increased, the hardness of glass decreased from 3.21 GPa to 2.277 GPa. Meanwhile, the thermal expansion coefficient of the glass was found to decrease from $15.8 \times 10^{-6} \text{ }^\circ\text{C}^{-1}$ to $13.3 \times 10^{-6} \text{ }^\circ\text{C}^{-1}$. It was found that the composition of $78.5 \text{TeO}_2 - 20 \text{ZnO} - 1.5 \text{Er}_2\text{O}_3$ exhibited the smallest thermal expansion coefficient and thus suitable for the fabrication of the glass rod. The surface roughness of the glass rods in the range of $2.15 \text{ } \mu\text{m}$ to $1.18 \text{ } \mu\text{m}$ was found to be dependent on the composition of the glass. In this work, the feed rate of 0.5, 1.0, 1.5 and 2.0 mm/min with spindle speed at 3000 rpm was the most suitable parameter to produce the desired glass rod.

ABSTRAK

Sampel kaca menggunakan siri sistem $(80-x) \text{TeO}_2 - 20 \text{ZnO} - x \text{Er}_2\text{O}_3$ dimana $0 < x \leq 2.5$ mol % telah disediakan menggunakan teknik pelindapan leburan. Fasa amorfokaca dikenalpasti melalui teknik pembelauan Sinar-X (XRD) dan sifat-sifat fizikal seperti ketumpatan dan kekerasan masing-masing telah diukur menggunakan kaedah *Archimedes* dan *Vickers Hardness*. Pengembangan terma kaca pula diukur melalui kaedah dilatometer. Mesin *rotary ultrasonic* (RUM) dengan mata berlian berdiameter 4 mm telah digunakan untuk menyediakan rod kaca. Sementara itu, kekasaran permukaan kaca ditentukan melalui teknik profilometri. Hasil yang diperolehi daripada XRD telah mengesahkan sampel kaca adalah amorfos. Didapati bahawa ketumpatan kaca meningkat dengan bertambahnya kandungan Er_2O_3 . Julat Ketumpatan kaca yang diperolehi berada diantara 5.541 g/cm^3 dan 5.663 g/cm^3 . Selain itu, dengan peningkatan kandungan Er_2O_3 , kekerasan kaca berkurang dari 3.215 GPa sehingga 2.277 GPa. Manakala, pekali pengembangan terma kaca didapati menurun dari $15.8 \times 10^{-6} \text{ }^\circ\text{C}^{-1}$ sehingga $13.3 \times 10^{-6} \text{ }^\circ\text{C}^{-1}$. Didapati, komposisi kaca $78.5 \text{TeO}_2 - 20 \text{ZnO} - 1.5 \text{Er}_2\text{O}_3$ mempunyai pekali pengembangan terma yang paling rendah dan sesuai untuk dijadikan rod kaca. Kekasaran permukaan di sepanjang rod kaca berada pada julat $2.15 \text{ }\mu\text{m}$ hingga $1.18 \text{ }\mu\text{m}$ dan sangat bergantung kepada komposisi kaca tersebut. Dalam kajian ini, kadar suapan 0.5, 1.0, 1.5 and 2.0 mm/min dengan kelajuan pengumpar 3000 rpm merupakan parameter yang paling sesuai untuk menyediakan rod kaca.

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LIST OF SYMBOLS

λ	Wavelength
E_v	Interatomic Potential
r	Interatomic Distance
T_g	Transformation Temperature
T_u	Upper Annealing Limit
M_g	Point of Incipient Deformation
T_d	Dilatometric Softening Point
A, B, n, m	Integer Number
ρ	Density
m_u	Weight of Sample In Air
m_t	Weight In Immersion Fluid
ρ_0	The Density of The Immersion Fluid
Hv	Vickers Hardness
L	Applied Load
Θ	Angle.
D	The Mean Diagonal
l	Length
l_0	Initial Length
T_0	Initial Temperature
α	The Linear Thermal Expansion Coefficient
ε	Relative Elongation
Δ	Changes
T	Temperature

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CHAPTER 1

INTRODUCTION

1.1 General Introduction

Laser an acronym for light amplification by stimulated emission of radiation, is certainly one of the greatest innovation of twentieth century. Its persisted growth and development has been an exciting division in the history of science, technology and engineering. Laser is distinguished from other electromagnetic radiation mainly in terms of its coherence, spectral purity and ability to propagate in a straight line.

Erbium is a well known element used in laser applications especially in an eye-safe rare-earth solid state laser. A significant characteristic of erbium doped host glass is the Er^{3+} concentration which is located in the eye-safe spectral region due to wavelength around $1.5\mu\text{m}$ (Jiang *et al.*, 1998; Ding *et al.*, 2000; Nandi *et al.*, 2006). The absorption in the region of greater than $1.4\mu\text{m}$ laser wavelength spectrum by liquid water in the eye provides enhanced eye-safety. Mierczyk *et al.* (2000) also collected that the wavelength of around $1.55\mu\text{m}$ is in the eye-safe regime where significantly higher pulse energies can be used without damaging human eyes. Laser action in erbium has been demonstrated in a variety of material such as garnets, fluorides, and glasses. Erbium laser performance is not very impressive in terms of efficiency or energy output. However, erbium has attracted attention because of two

particular wavelength of interest. A crystal, such as YAG with highly doped erbium produces an output at around 2.9 μm (Yaqin *et al.*, 2000) and erbium doped phosphate glass generates an output at 1.54 μm (Li *et al.*, 2009). Both of these wavelengths are absorbed by water, which leads to interesting medical applications. The wavelength 1.54 μm arises from a transition between the $^4\text{I}_{13/2}$ state and the $^4\text{I}_{15/2}$ ground state of Er^{3+} (Snoeks *et al.*, 1995). In fact, owing to the laser properties, thermal conductivity of glass is considerably lower than that of most crystal hosts, the emission lines of the ions in glasses are inherently broader than in crystals. A broader line increases the laser threshold value of amplification. Nevertheless, this broadening has an advantage. It offers the possibility of obtaining and amplifying medium for the same linear amplification coefficient. Thus, glass and crystalline lasers complement each other. For continuous or very high repetition rate of operation, crystalline materials provide higher gain and greater thermal conductivity. Glasses are more suitable for high-energy pulsed operation because of their large size, flexibility in their physical parameters and the broadened fluorescent line (Jaba *et al.*, 2009; Koechner *et al.*, 2003; Edward *et al.*, 1974).

Notwithstanding the above, erbium doped with tellurite as a host also has extensively being studied in recent years owing to the excellent properties exhibit in tellurite glass. Tellurite glass has been demonstrated to have excellent rare-earth (RE) ion solubility and high quality specialty glass requirement for potential insulating in solid state laser material. The emission spectrum from erbium in tellurite glasses is almost as broad as the corresponding spectrum in silica (Marjanovic *et al.*, 2003). Meanwhile, tellurite as a host material posses to have various excellent properties such as the lowest phonon energy among oxide glasses, high dielectric constant, strength and corrosion resistance over other host such as fluoride glass. Whereas, rare-earth elements in tellurite glass generally have lower non-radiative decay rates, larger values of the radiative cross section (or transition strength), shorter fluorescent lifetimes and a red shift of the radiative transition (Wang *et al.*, 1994; Rolli *et al.*, 2002; El Mallawany, 2002). Furthermore, tellurium oxide is covalently bonded, stable and has a highly deformed octahedral in its structure because of the valence characteristics of Te results in two sets of Te-O distance. Since, each oxygen atom must be shared with three tellurium atoms, symmetry requirements force a distortion of octahedral to accommodate them into a

regularly repeating lattice, thus there are six different Te-O distances, four Te-Te distances and twelve O-O distances. It is possible that this distortion produces a structure energetically similar to the vitreous state in which there is only short range order (El Mallawany, 2002).

This research concentrates on the system of $(80-x)\text{TeO}_2 - 20\text{ZnO} - x\text{Er}_2\text{O}_3$ with $0 \leq x \leq 2.5$ mol % glasses because of its wide and stable glass formation range (Sulhadi, 2007). The glass system is prepared through conventional melt quenching technique. This research contributes to fabricate rod glass with desired diameter and length when operating with end pumping laser cavity system.

1.2 Problem Statement

The study of erbium-doped tellurite glass which emphasized on the optical, physical, chemical durability and mechanical properties has previously been done by many researchers (Sulhadi, 2007; Jaba *et al.*, 2000). However for some reasons, the property that effected on the fabrication process especially in machining the laser material has not very well mention in literature. So it is the aim of this project to study an appropriate required physical and some thermal parameters to fabricate the glass rod (active medium) during machining procedure. The optimization of these physical and thermal parameters is very important to get the accuracy in rod design especially their geometrical parameters. From this point of view, this research is engaged towards the performance improvement of the fabrication in machining the optical glasses and to characterize the sufficient minimum surface roughness that can be considered as confidential to many manufactures. Thus, research should be accelerated to find the hardness and thermal properties especially to measure the strengthening of the samples in order to get the most sufficient condition when operate during machining process.

1.3 Research Objective

The objectives of this project are as follow:

- i) To prepare Er^{3+} doped Tellurite glass of the $(80-x) \text{TeO}_2 - 20 \text{ZnO} - x \text{Er}_2\text{O}_3$ system with $(x \leq 2.5 \text{mol } \%)$
- ii) To determine the amorphousity of these glass.
- iii) To study the density, hardness and thermal expansion of the glasses.
- iv) To estimate the process parameter (spindle speed and feed rate) to fabricate the fine glass rods.
- v) To determine the optimum surface roughness along the glass rods

1.4 Scope of Study

In this study, the glass based on $(80-x) \text{TeO}_2 - 20 \text{ZnO} - x \text{Er}_2\text{O}_3$ system ($x \leq 2.5 \text{ mol } \%$) is prepared using melt quenching method. The system is choose due to the wide and stable glass formation range, and have high quality specialty in structural and optical properties as reported by Sulhadi (2007). The sample is characterized in term of their amorphosity, density, hardness, and thermal expansion. X-ray diffraction method is used to reveal the amorphosity of the glasses. Density measurement is performed by Archimedes method in toluene. The hardness of the glass is measured using Vickers Hardness. The dilatometry method is used to estimate the thermal expansion of the glass system. The rod of erbium doped tellurite glass is fabricated using Rotary Ultrasonic machining (RUM) with appropriate machining parameters such as spindle speed and feed rate. In addition, Mitutoyo Formtracer CS-5000 is also used to determine the surface roughness of the glass rod after machining procedure.

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APPENDIX A

Batch Calculation

The calculation start with determine the weight fraction of each component required to produce the desired molar composition. These begin by multiplying the mole fraction and molecular weight for each component. Then, total up all these contribution to determine the molecular weight of the glass, and divide each individual contribution by the molecular weight of the glass to determine the weight fraction of each component. Lastly, multiply the weight fraction of each component by the amount of glass to be produced ((80-x) TeO₂-20 ZnO-x Er₂O₃ system). Fortunately, all the batch calculations follow the same procedure.

1. (80-x) TeO₂-20ZnO-x Er₂O₃ system.

Glass composition: (80-0) TeO₂-20ZnO-(0) Er₂O₃

Molecular weight of component (in gmol⁻¹):

$$\text{TeO}_2 = 159.60 \quad \text{ZnO} = 81.37 \quad \text{Er}_2\text{O}_3 = 382.52$$

$$\text{Molecular weight of glass: } (0.80 \times 159.60) + (0.20 \times 81.37) + (0 \times 382.52) = 143.95$$

$$\text{TeO}_2 = (0.80 \times 159.60) / 143.95 = 0.8734$$

$$\text{ZnO} = (0.20 \times 81.37) / 143.95 = 0.1131$$

$$\text{Er}_2\text{O}_3 = (0 \times 382.52) / 143.95 = 0.0000$$

For 100gm of glass,

$$\text{TeO}_2 = 0.8734 \times 100 = 87.34\text{g}, \text{ZnO} = 0.1131 \times 100 = 11.31\text{g}, \text{Er}_2\text{O}_3 = 0.0000 \times 100 = 00.00\text{g}$$