



**UNIVERSITI TEKNIKAL MALAYSIA MELAKA**

**THE EFFECT OF SINTERING TEMPERATURE AND CLAY  
ADDITION TOWARDS GLASS CERAMIC PRODUCED FROM  
RECYCLE GLASS BY USING  
PRESSING METHOD**

This report submitted in accordance with requirement of the Universiti Teknikal Malaysia Melaka (UTeM) for the Bachelor Degree of Manufacturing Engineering (Engineering Materials) with Honours.

By

**NUR FARIZAN BINTI AYOOB**

FACULTY OF MANUFACTURING ENGINEERING

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Saya NUR FARIZAN BINTI AYOOB

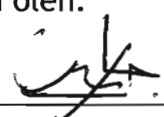
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PERMATANG TOK MAHAT  
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Cop Rasmi:  
**DR. JARIAH BINTI MOHAMAD JUOI**  
Pensyarah: Kanan  
Fakulti Kejuruteraan Pembuatan  
Universiti Teknikal Malaysia Melaka

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Author's Name : NUR FARIZAN BINTI AYOOB  
Date : 19 April 2010

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Signature :  
Author's Name : NUR FARIZAN BINTI AYOOB  
Date : 19 April 2010

## **APPROVAL**

This report is submitted to the Faculty of Manufacturing Engineering of UTeM as a partial fulfillment of the requirements for the degree of Bachelor of Manufacturing Engineering (Engineering Materials) with Honours. The members of the supervisory committee is as follow

.....

**Dr. Jariah Mohamad Juoi**

(Main Supervisor)

## ABSTRACT

This research aims to study and investigate the effect of sintering temperature and clay addition towards glass ceramic produced from recycle glass by using pressing method. Soda lime silicate glass is used as main raw material and ball clays act as filler for glass ceramic fabrication. Firstly, the recycle glass bottles were crushed by using hammer until they passed through a sieve of less than 75 $\mu$ m. The glass powder was then mixed with ball clays with weight percent ratio of 95:05, 90:10 and 85:15. Then, green ceramic article is formed using uniaxial dry pressing method. Green is referred to the unfired ceramic. In this study, the sintering temperature are varied at 750°C, 850°C and 950°C. The microstructure and phase present in glass ceramic is analyzed using scanning electron microscope (SEM) and x-ray diffraction (XRD). The physical properties are determined using porosity, density measurement and water absorption test. Mechanical analysis is carried out using microhardness test to determine its mechanical properties. The results indicated that with 10 weight percentage of clay addition and sintering temperature of 850°C, glass ceramic produced with good and suitable physical and mechanical properties. It is showed that the increasing of sintering temperature and percentage of clay addition had increasing hardness and density of sintered glass ceramic. Hence, it will decrease the water absorption and porosity of the glass ceramic. Due to the results obtained, the optimum sintered temperature and percentage clay addition was found at 850°C with 10 weight percent clay addition. This is because at this point, the mechanical and physical properties gives the less percentage amount of porosity and water absorption and higher on bulk density and microhardness compare than the others. Through the study, the enhancement of fabricated glass ceramic could be applied to the structural applications through the advantage of mechanical and physical properties performance of hardness, water absorption, porosity and density.

## ABSTRAK

Projek ini adalah bertujuan untuk mengkaji dan menganalisis kesan suhu pembakaran dan peratusan penambahan ketulan tanah liat terhadap penghasilan seramik kaca dengan menggunakan kaedah penekanan. Botol kaca kitar semula digunakan sebagai bahan utama dan ketulan tanah liat pula digunakan sebagai bahan pemangkin bagi menghasilkan seramik kaca. Seramik kaca diaplikasikan didalam struktur binaan (contohnya, jubin, batu-bata, pekakas ketuhar dan pekakas meja). Proses ini bermula dengan menghancurkan botol kaca tersebut sehingga ianya boleh melalui penapis yang bersaiz  $75\mu\text{m}$ . Serbuk kaca ini kemudiannya dicampurkan dan digaulkan dengan tanah liat dengan peratusan nisbah berdasarkan berat sebanyak 95:05, 90:10 dan 85:15. Jasad hijau dihasilkan apabila penekanan pengeringan dikenakan keatasnya. Jasad hijau merujuk kepada seramik yang belum melalui proses pembakaran. Dalam projek ini, suhu pembakaran adalah berubah-ubah pada suhu  $750^{\circ}\text{C}$ ,  $850^{\circ}\text{C}$  dan  $950^{\circ}\text{C}$ . Pada setiap suhu pembakaran, masa digunakan untuk pembakaran sekata adalah sejam. Sifat-sifat struktur mikro dan fasa seramik kaca dianalisis dengan menggunakan scanning electron microscope (SEM) dan powder x-ray diffraction (XRD). Sifat-sifat fizikal seramik kaca dianalisis dengan melakukan ujian penyerapan, pengukuran kepadatan dan penyerapan air keatas seramik kaca. Sifat-sifat mekanikal pula ditentukan melalui ujian mikroketahanan. Keputusan kajian menunjukkan bahawa dengan peratusan penambahan tanah liat dan suhu pembakaran yang tepat, sifat fizikal dan mekanikal yang sesuai dengan aplikasinya boleh diperolehi. Hal ini menunjukkan bahawa peningkatan suhu pembakaran dan peratusan penambahan tanah liat dapat meningkatkan kekerasan dan ketumpatan pembakaran seramik kaca. Oleh kerana itu juga, ia akan menurunkan peratusan penyerapan air dan porositi dari kaca seramik yang dihasilkan. Dengan keputusan yang diperolehi, suhu pembakaran optimum dapat diperolehi pada suhu

850°C dengan penambahan peratusan berat ketulan tanah liat kepada 10 peratus. Ini kerana pada suhu ini, keputusan menunjukkan bahawa sifat fizikal dan mekanikal adalah yang terbaik diperolehi iaitu peratusan porositi dan penyerapan air adalah kurang dan peratusan ketumpatan keseluruhan dan kekuatan meningkat berbanding yang lain. Melalui kajian ini, peningkatan penghasilan kaca seramik dapat diterapkan pada aplikasi struktur melalui keistimewaan dari sifat mekanik dan fizik prestasi kekerasan, penyerapan air, porositi dan ketumpatan.



# DEDICATION

*For my beloved family, friends and UTeM*

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# LIST OF ABBREVIATIONS, SYMBOL, SPECIALIZED NOMENCLATURE

1000x	-	1000 times magnification
2000x	-	2000 times magnification
3000x	-	3000 times magnification
5000x	-	5000 times magnification
%	-	Percent
°C	-	Degrees Celsius
$\rho$	-	Density
Al <sub>2</sub> O <sub>3</sub>	-	Aluminum oxide/ Alumina
ASTM	-	American Society for Testing and Materials
CaO	-	Calcium oxide
cm <sup>3</sup>	-	Centimeter cube
e.g.	-	Example
<i>et al.</i>	-	and others
g	-	gram
GPa	-	Giga Pascal
kg	-	Kilogram
kV	-	Kilo Volt
min	-	Minutes
mm	-	Millimeter
MPa	-	Mega Pascal
$\mu\text{m}$	-	Micrometer
rms	-	root mean square
SEM	-	Scanning electron microscope
SiO <sub>2</sub>	-	Silicon dioxide/ Silica
wt. %	-	weight percentage
XRD	-	Xray diffractometer

# CHAPTER 1

## INTRODUCTION

### 1.1 Background of study

Nowadays, there is a great concern regarding to the increasing amount of industrial wastes such as plastics, glasses, and grog (fired clays). The disposal of these wastes is one of the issues that have received a lot of attention and a high demand for the safety of the environment. One technique used to reduce such wastes is by recycling, which is not only benefits to the environment but also to the economy (Loryuenyong *et al.*, 2009).

Brown and Mackenzie, 1982 stated that different kinds of wastes have already been recycled in ceramic industry. Example includes waste glasses, which are readily incorporated as an alternative ceramic raw material or as a fluxing agent in stoneware, tiles, bricks and concrete. However, among these, many thousands tones of glass are discarded, much in the form of non-returnable bottles and containers. In the developed countries, a growing interest in conservation of resources and ecological preservation has led to an increase in the recovery of solid wastes including glass, some of which is re-melted. Glass is an ideal material for recycling. The use of recycled glass in new container helps save of energy. It helps in brick and ceramic manufacture, and it conserves raw materials, reduces energy consumption, and the volume of waste sent to landfill (Turgut and Yahlizadec, 2009).

Glass-ceramics from the sintering of powdered glasses is one of the most interesting applications (Bernardo, 2008). It is known that in glass-ceramics, the crystals of the size of 0.1-20  $\mu\text{m}$  are precipitated in the glasses. There are two methods for obtaining glass-ceramics; one is the surface crystallization, and the other the bulk crystallization (Tanaka *et al.*, 2005). The glass-ceramics in which the crystals are precipitated in the whole of the glass by the bulk crystallization have the properties of high mechanical function and high chemical durability. The glass ceramics in which the prismatic crystals of Wallastonite ( $\text{CaO}\cdot\text{SiO}_2$ ) and Anorthite ( $\text{CaO}\cdot\text{Al}_2\text{O}_3\cdot 2\text{SiO}_2$ ) are precipitated have the properties suitable for the construction materials. Therefore, these have been called the glass-ceramics for the construction materials. There are many studies on these materials and a part of them have been commercialized (Tanaka *et al.*, 2005). Commercial examples of this approach are the Wallastonite-based 'Neoparies', developed in Japan since 1970s. Sintered pyroxene-based glass ceramics from cheaper and more accessible raw materials has been developed in Bulgaria and in Italy, since the early 1990s. The parent glass being provide in powdered form, therefore long fining times are not needed and drastically reducing the costs of preliminary glass making. In addition, very limited processing times may be advantageous in avoiding the volatilization of dangerous oxides (like those of heavy metals) which may be dissolved in glasses when obtained from wastes (Bernardo *et al.*, 2007).

## 1.2 Problem statement

Today, solid waste management is a critical national issue. One of the issues regarding landfills in Malaysia is an abbreviate life span due to the intensifying amount of the solid waste generation and human population as well. Due to rapidly changing lifestyle, waste productions are always increasingly expected to be great. Therefore, the production of glass ceramic materials made by recycling industrial waste is introduced as well-known technology. Many researchers have paid much attention to produce glass, glass ceramic and sintered materials from industrial wastes in order to make them reasonable safe for the environment. Besides, glass ceramics obtained from industrial wastes did have several desirable properties to fulfil many applications such as wall covering panel, floors and roofs in industrial and public buildings, interior facing of containers for the chemical industry and as road surfacing.

Glass ceramic materials are generally produced by a traditional glass forming technique starting from the melted glass, followed by controlled nucleation and crystallization heat treatment processes. In recent years, glass ceramics were developed using the technique of sinter-crystallization of glass powders (Erol *et al.*, 2009). The advantages of this method are essentially that it does not require high investment and is suitable for the production of small quantities of articles of complicated shapes. More recently, sintered glass ceramic materials were produced using natural raw materials. The present study aims to investigate the effect of sintering temperature and clay addition towards glass ceramic produced from recycle glass by using dry pressing method.

### 1.3 Objectives

There are several aims that need to be achieved in this project, which are

- i) To study the effect of sintering temperature and clay addition towards glass ceramic produced from recycled glass by using dry pressing method.
- ii) To characterize the microstructure and phases present in the produced glass ceramic samples.
- iii) To investigate the physical and mechanical properties of the produced glass ceramic samples.

### 1.4 Scope of study

In order to investigate the effect of sintering temperature and clay addition towards glass ceramic produced from recycle glass, the scope of the study is tailored to produce glass ceramic samples for structural application. The fabrication process is by common ceramic techniques (e.g. uniaxial pressing method). The study starts by preparing recycle glass as raw materials to be used in fabrication of glass ceramic samples. The glass powders were prepared by crushing bottles made from glass using hammer until they passed through a sieve of less than  $75\mu\text{m}$  to obtain fine particles. The stage of process will be followed by mixing the glass powder and ball clays with ratio of 95:5, 90:10 and 85:15. Ball clays acts as filler which is to improve the bonding properties between particles and helps to obtain dense materials by sticking glass powders together during pressing method (Rozenstrauha *et al.*, 2006).

The next stage of process involves the forming process which is to form the mixing glass powder into green ceramic article. Green is referred to the unfired ceramic. Preferably uniaxial dry pressing is used. One gram of mixed powder is pressed with 3.5 tons of uniaxial dry pressing in a cylindrical shape with diameter of 13mm. The pressed article is then removed from the die and sintered in a furnace. The sintering temperature of this

study will be varied at 750°C, 850°C and 950°C. For each sintering, the duration of holding time will be one hour.

At the end of this study, the sintered glass ceramic samples were then subjected to characterization, physical analyses and mechanical testing. The microstructure analysis of the fractured surface of the fabricated of glass ceramic is observed using the scanning electron microscope (SEM) and powder x-ray diffraction (XRD). The physical properties are analysed using porosity, density measurement and water absorption test, while the mechanical properties will be determined using microhardness test. All of these tests are carried out according specific ASTM.

## **CHAPTER 2**

### **LITERATURE REVIEW**

#### **2.1 Introduction**

This chapter discuss review related study on the glass ceramic. The content are focusing on the effect of sintering temperature and time towards glass ceramic produced from recycle glass using pressing method.

#### **2.2 Waste**

Diaz *et al.*, (1993) stated that waste is unwanted and unusable materials that are expelled from a resource because apparently it is of no further use to the processor. There are many types of waste including municipal solid waste, construction waste, medical waste, hazardous waste and biodegradable waste. Solid waste is a common term used to describe the things we throw away which embrace things commonly describe as garbage, refuse and trash (Davis and Cornwell, 2008). There is some possible classification of waste. The classification are by physical state (solid, liquid, gaseous), and within solid waste by original use (packaging), material (e.g., paper, glass and plastic), physical properties (e.g., recyclable, combustible and compostable), origin (e.g., domestic, commercial and industrial) or safety level (hazardous and non hazardous) (McDougall *et al.*, 2000).

## **2.3 Recycling Waste**

Recycling is the selection, classification and reemployment of waste as a raw material to produce the same, or very similar product, to the parent material. It involves processing used materials into new products to prevent waste of potentially useful materials, reduce the consumption of fresh raw materials, the energy usage, air pollution from incineration and water pollution from land filling by reducing the need for conventional waste disposal, and lower greenhouse gas emissions as compared to virgin production (Dhir, 2001). Recycling waste is a material that includes many kinds of glass, paper and plastic.

### **2.3.1 Recycling glass**

According to Callister (2005), glass is a hard, brittle, transparent material produced by melting a mixture of materials which is used for containers, lenses, windows and other applications. It is an amorphous solid that has been around since 12,000 BCE. They are noncrystalline silicates and most consist mainly of silicon, sodium, and calcium oxides (referred to as soda-lime glass) with other minor components, such as aluminium and magnesium oxides (Haun and Joseph, 2002). Glass is also defined as a supercooled liquid that rigid and static but does not change molecularly between melting and solidification into desired shape. It is one of versatile substances widely used in our lives through manufactured products such as sheet glass, bottles, glassware, and vacuum tubing. In addition, glass is also an ideal material for recycling (Turgut and Yahlizadec, 2009).

Recycling glass is a part of a simple but hugely beneficial process. It is a process of converting waste glass into usable products. Depending on the end use, this commonly includes separating it into different colors because glass colors are achieved through the addition of different ions to the glass mixture. Recycling glass structure does not deteriorate when reprocessed. For bottles and jars, up to 80% of the total mixture can be made from reclaimed scrap glass, called cullet. Cullet from a factory has a known