UNIVERSITI TEKNIKAL MALAYSIA MELAKA

INVESTIGATION ON PHYSICO-MECHANICAL PROPERTIES OF GLASS-CERAMIC COMPOSITE FROM ECO-WASTE MATERIALS

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

by

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Date : 25 JANUARY 2022

APPROVAL

This report is submitted to the Faculty of Manufacturing Engineering of Universiti Teknikal Malaysia Melaka as a partial fulfilment of the requirement for Degree of Manufacturing Engineering (Hons). The member of the supervisory committee is as follow:

....

(DR. ZURINA BINTI SHAMSUDIN)

ABSTRAK

Tujuan kajian ini adalah untuk menyiasat kesan pemuatan pengisi daripada bahan sisa mesra alam terhadap sifat fiziko-mekanikal komposit kaca-seramik. Kulit kerang (KK) dengan saiz zarah sekitar 75μm telah digabungkan sebagai pengisi dalam komposit kaca-seramik sepanjang kajian ini. Sementara itu, kaca soda kapur silikat (*SLSG*) berfungsi sebagai matriks yang mempunyai saiz keseluruhan sekitar 75 μm. Pengisi KK telah dikeringkan dan dikalsinkan pada suhu 1000°C dengan kadar pemanasan malar 10°C/min selama empat jam sebelum didedahkan kepada proses pensinteran terus. Empat kelompok rumusan dibuat dengan nisbah SLSG: CS 50:50, 60:40, 70:30 dan 100:0 wt.%. Untuk mencapai penyebaran seragam campuran, mesin pengisar bola telah digunakan untuk menggabungkan zarah. Bahagian segi empat sama hijau dibentuk dengan memampatkannya menggunakan penekan akapaksi pada 10 tan selama dua minit. Pada suhu 700, 750, 800 dan 850°C, sampel telah disinter dengan kadar pemanasan malar 2°C/min dan tempoh tinggal selama satu jam. Untuk menentukan kehadiran ikatan hidrogen, *Fourier transformasi infrared spectroscopy* (FTIR) telah digunakan untuk mencirikan unsur KK, manakala X-ray Difraksi (XRD) dilakukan untuk mengenal pasti fasa komposit kaca SLS yang dikitar-semula. Sifat fizikal diukur menggunakan ASTM C373 sebelum keliangan lutsinar bagi komposit dianalisis. Kekerasan komposit SLSG kitar-semula dinilai menggunakan ujian kekerasan Vickers (ASTM C1327-99) dan ukuran akustik (ASTM E494-95), dengan peratusan ralat direkodkan untuk memerhatikan struktur mikro pada permukaan yang retak dan untuk membuat kesimpulan hubungan antara struktur mikro dan sifat fizikal dan mekanikal komposit SLSG. Dapatan menunjukkan bahawa sampel dengan 30wt.% pengisi KK mempunyai pengecutan linear yang tinggi dan keliangan ketara yang rendah, kedua-duanya menyumbang kepada ketumpatan pukal 2.09g/cm^3. Corak XRD menunjukkan kuarza, kristalit, silikon oksida (SiO_2). kalsium oksida (CaO), dan natrium oksida (Na_2O) selepas proses pensinteran. Dalam ujian mikrokekerasan, komposisi ini mempunyai bacaan mikrokekerasan purata tertinggi iaitu 1004.86Hv.

ABSTRACT

The purpose of this study was to investigate the effect of filler loading from eco-waste material on physico-mechanical properties of the glass-ceramic composite. The cockle shell (CS) with a particle size of around 75μm was incorporated as a filler in a glass-ceramic composite throughout this study. Meanwhile, soda-lime silica glass (SLSG) served as a matrix having an overall size of around 75 μm. The CS filler was dried and calcined at temperatures of 1000°C with a constant heating rate of 10°C/min for four hours before being exposed to the direct sintering process. Four batches of formulation were made with SLSG: CS ratios of 50:50, 60:40, 70:30, and 100:0 wt.%. To achieve uniform dispersion of the mixture, a planetary ball mill was employed to combine the particles. The green square part was formed by compacting it using uniaxial pressing at 10 tons for two minutes. At temperatures of 700, 750, 800 and 850°C, the samples were sintered with a constant heating rate of 2°C/min and a dwell duration of an hour. To determine the presence of a hydrogen bond, Fourier transforms infrared spectroscopy (FTIR) was utilised to characterise the element of CS, while X-ray Diffraction (XRD) was performed to identify the phase of the recycled SLS glass composite. Before analysing the apparent porosity of the composite, the physical characteristics were measured using ASTM C373. The hardness of recycled SLSG composite was evaluated using the Vickers hardness test (ASTM C1327-99) and acoustic measurement (ASTM E494-95) where the percentage of the error will be recorded as comparison data. Scanning electron microscopy (SEM) was used to observe the microstructure of the surface fracture and concluded the relationship between microstructure and physico-mechanical properties of the SLSG composite. The findings revealed that the sample with 30wt. % CS filler had outstanding physical and mechanical qualities at 850°C. This formulation has a high linear shrinkage and a low apparent porosity, both of which contribute to its greater bulk density of 2.09g/cm^3. XRD patterns indicated quartz, cristobalite, silicon oxide (SiO_2), calcium oxide (CaO), and sodium oxide (Na_2O) following the sintering process. The composition had the highest average microhardness reading, 1004.86Hv, in the microhardness test.

DEDICATION

To my beloved family,

for their compassion.

To exclusively my supervisor,

for her guidance and ideas.

To my lecturer and friends,

for your emotional support, assistance, advice, and kindness.

Thank You So Much.

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Ultimately, I would like to thank everyone who helped with the preparation of this Final Year Project and deeply apologize for not being able to show my appreciation individually.

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LIST OF ABBREVIATIONS

ACS	-	Ark Clamshell
Al_2O_3	-	Aluminium Oxide
ASTM	-	American society for testing and materials
$CaCo_3$	-	Calcium Carbonate
CaF_2	-	Calcium Fluoride
CaO	-	Calcium Oxide
CS	-	Cockle shell
DT	-	Destructive Testing
ES	-	Eggshell
Etc.	-	Et cetera
Fe_2O_3	-	Iron Oxide
FTIR	-	Fourier Transform Infrared Spectroscopy
GCC	-	Glass-ceramic Composite
GIC	-	Glass Ionomer Cement
Hv	-	Hardness Vickers
K_2O	-	Potassium Oxide
MgO	-	Magnesium Oxide
MnO	-	Manganese Oxide
Na_2O	-	Sodium Oxide
NDE	-	Non-destructive Evaluation
NDE	-	Non-destructive Examination
NDI	-	Non-destructive Inspection
NDT	-	Non-destructive Testing
P_2O_5	-	Phosphorus Pentoxide
PE	-	Pulse-Echo
RM	-	Ringgit Malaysia
SEM	-	Scanning Electron Microscopy
SiO_2	-	Silicon Dioxide
SLS	-	Soda-lime Silicate
SLSG	-	Soda-lime Silicate Glass

SO_3	-	Sulphur Trioxide
SrO	-	Strontium Dioxide
TiO_2	-	Titanium Oxide
TT	-	Through Transmission
TOFD	-	Time of Light Diffraction
UT	-	Ultrasonic Testing
WGC	-	Wollastonite glass-ceramic
XRD	-	X-ray Diffraction
ZnO	-	Zinc Oxide

LIST OF EQUATIONS

LIST OF SYMBOLS

%	-	Percentage
%AP	-	Apparent Porosity
θ	-	Angle
°	-	Degree
°C	-	Degree Celsius
°C/min	-	Degree Celsius per minute
μm	-	Micron metre
Kg/m^3	-	Kilogram per metre cube
g	-	gram
g/cm^3	-	Gram per centimetre cube
mm	-	Millimetre
min	-	min
m/s	-	Metre per second
N/m^2	-	Newton per metre squared
wt.%	-	Weight Percentage

CHAPTER 1

INTRODUCTION

1.1 Background of Study

These days, urbanization and rapid industrial and lifestyle development contribute to an increase in the consumption of natural resources and a decrease in their availability (Jassim, 2017). On the contrary, humans have always produced trash and disposed of it in some fashion, which has an impact on the environment. As a result, the increased waste generated by industrial factories and human activities should be monitored. As a response, scientists have found new types of engineering, such as sustainable engineering and green engineering, to reduce energy and natural resource usage (Jassim, 2017). One of many ways in saving the environment is to reuse the waste from sustainable or eco-materials.

Soda-lime silicate glass (SLSG) as eco-material wastes generated from rapid industrial development are intensively used as alternative materials in the production of the glass-ceramic composite (Hossain et al., 2018). SLSG contributed smooth and non-reactive surfaces (Hasanuzzaman et al., 2016). It has greater physico-mechanical properties such as strength and fracture toughness than the parent glass if formed by melting and casting (Ingole et al., 2018) and heat treatment controlled. In this study, the glass-ceramic composite was formed using a sintering process. Eco-material waste such as cockle shells (CS) was used as the filler load in recycled soda lime silicate (SLS) glass-ceramic composite. Cockle Shells (CS) has shown potential to increase the strength of composite due to the presence of calcium oxide (CaO.)

Polycrystalline materials that were formed by the controlled crystallization of glass are defined as glass-ceramics (Pinckney, 2001). The glass-ceramic composite has better physical and mechanical properties (e.g., strength; fracture toughness) than the parent glass produced by melting and casting since it has a composite structure consisting usually of a fine crystalline phase scattered in a matrix of glass (Rahaman, 2014). Because of their strong mechanical, chemical, and abrasion resistance, high hardness, variable thermal expansion depending on chemical composition, and sinterability to relatively high densities (92–98%) at temperatures usually less than 1000 °C, glass–ceramics can be a solution in many applications (Arcaro et al., 2017). To improve the strength properties of the glass-ceramic composite, filler material was added during the manufacturing of the glass-ceramic composite.

This study aims to investigate the effect of the addition of eco-waste materials on the properties of the glass-ceramic composite. The physical properties were tested using conventional measurement and the mechanical properties were evaluated using two different methods which are microhardness testing and acoustic method.

1.2 Problem Statement

The materials with a less hazardous substance, materials with a green environmental profile, or materials of higher recyclability can be classified as eco-materials. These eco-materials from wastes such as carbon fibres, glasses or from natural sources, for example, spent bleach earth, and cockle shells have potential that were explored as a filler to improve the performance of the product glass-ceramic composite using sintering process (Shamsudin et al., 2018). The glass-ceramic sample will be sintered at different temperatures ranging from 700 to 850°C (Ayoob et al., 2011; Gualberto et al., 2019).

A study by Jusoh et al., (2019a) has discussed that sintering temperature will affect the porosity, linear shrinkage, density, water adsorption and microstructure of the glass-ceramic composite. Influence the outcome of strength properties if poor densification causes high porosity. As the glass-ceramic is defined as polycrystalline materials, the strength is enhanced by decreasing their grain size. Hence, the formulation for the filler loading (wt.%) to improve the performance of the product glass-ceramic composite was investigated in this paper.

To measure one of the strength properties which is Young's Modulus, *E* for brittle materials, the common destructive test (flexural test) was used (Cattell et al., 2020). Regrettably, these features may not be dependable enough because the samples are performed on coupons, with the idea that the coupon is a true example of the component that would be used in services. That is why the results of the destructive test (flexural test) on coupons may not apply to the component to be used in service, as it might be affected by the handling process.

As the result of the problem, this project focused on the fabrication of glass-ceramic composite from eco-waste materials. The mechanical property was evaluated via microhardness test and acoustic method. Non-Destructive Test (acoustic method) aided in predicting the failure probability of a component given the crack size and fracture toughness parameters of the materials (Sohn & Olivas-Martinez, 2014). The acoustic technique is used because it can recognize the thickness and the longitudinal velocity.

1.3 Objective

The following are the study's objectives:

(a) To characterise the effect filler loading (wt.%) of the cockle shells (CS) in the physical properties in the recycled soda-lime silicate (SLS) glass at different sintering temperatures.

(b) To evaluate the mechanical properties of SLSG/CS glass-ceramic composite using microhardness testing and acoustic method.

(c) To correlate the effect of filler loading on physico-mechanical to morphology from the literature review.

1.4 Scope of Study

The following are the study's scope of the study:

a) The formulations of the filler loading (wt.%) of cockle shells (CS) are 30wt.%, 40wt.% and 50wt.% in the recycled soda-lime silicate (SLS) glass and cockle shells (CS) glass-ceramic composite sintered at different sintering temperatures from 700°C, 750°C, 800°C and 850°C at a constant heating rate at 2°C/min and dwelling time for 1 hour.

b) Calcination process of CS at 1000°C, characterisation of material via X-ray Diffraction (XRD) for phase identification and Fourier Transform Infrared Spectroscopy (FTIR) for detecting the hydrogen bond. Physical analysis of the recycled SLS glass and CS composite is determined using ASTM C373.

c) Evaluate the mechanical properties on four (4) batches of the recycled soda lime silicate (SLS) glass and cockle shells (CS) composite using microhardness test according to ASTM C1327-99 and acoustic method (ASTM E494-95).

d) Correlate the microstructure of cockle shells/recycled soda lime silicate (SLS) glass-ceramic composite with physico-mechanical properties using the literature review.

1.5 Organization of Report

This study is divided into five chapters: introduction, literature review, methodology, result and discussion, and conclusion. The first chapter discussed the background of the study, problem statement, objectives, and scopes of the study. The second chapter's literature review includes past research or study on glass-ceramic composite, SLSG, CS, sintering process and parameters affecting sintering, microhardness testing and acoustic testing. The third chapter covers the entire flow of the study, methodologies, and procedures that will be used to finish this research. In the fourth chapter, the information collected after completing several selected tests such as the Vickers hardness test, bulk density, linear shrinkage, and apparent porosity were examined. Conclusions and recommendations from this study are presented in chapter five.

CHAPTER 2

LITERATURE REVIEW

This chapter discussed reviews study based on earlier research by other scholars. The goals of this chapter are to learn more about prior studies that were relevant to this paper and can be used to support this study to reach the best conclusion for this paper. This chapter discussed the studies about eco-material waste as filler for recycled soda lime silicate (SLS) glass-ceramic composite, destructive test (Flexural test) and non-destructive testing (ultrasonic technique). From the methodological step through the completion of PSM 2, this chapter guided the planning process of the entire project.

2.1 Glass-Ceramic Composite (GCC)

Glass-ceramics are ceramic materials made from the nucleation and crystallization of glass under controlled conditions. Glass-ceramics could also offer substantial advantages over standard glass or ceramic materials by combining the flexibility of glass forming and inspection with better and frequently unique glass-ceramic characteristics (Pinckney, 2001). Deubener *et al.*, (2018) proposed the updated definition of glass-ceramics, which glass-ceramics are inorganic, non-metallic materials prepared by controlled crystallization of glasses via different processing method.

Efficient nucleation, which allows the formation of small, randomly oriented grains without voids, micro-cracks, or other porosity, is the core of regulated internal crystallization (Höland & Beall, 2019). Pinckney (2001) stated that the important variables in the creation of a glass-ceramic are glass composition, glass ceramic phase assembly, and microstructure crystalline. Due to the speedy development of the glass ceramics, researchers aiming to improve the mechanical properties of glass ceramic by integrating recycled glass with natural waste material (Chinnam et al., 2013). There are various types of glass-ceramic applications such as technical applications, consumer applications, optical applications, medical and dental applications, electrical and electronic applications, architectural applications, coatings and solders, and glass-ceramics for energy applications (Höland & Beall, 2019).

2.2 Eco-material Waste as Raw Material in Processing Glass-Ceramic Composite

Sustainable materials are those employed in the consumer and industrial economies that can be produced in sufficient quantities without depleting non-renewable sources or upsetting the environments and critical natural resource systems established steady-state balance. The eco-material wastes such as soda-lime silicate glass and cockle shell are used in this study.

2.1.1 Soda-lime silicate (SLS) glass

For hundreds of years, soda-lime glass, also called soda lime silicate (SLS) glass has been manufactured throughout most of Europe. Silica, mostly in the shape of sand, and limestone were plentiful almost everywhere. Crown glass can be another term for a high-silica variety of soda lime which had been used for window. The SLS glass is made up of silicone-oxygen tetrahedron (SiO_4) connected at the oxygen atoms (Karazi et al., 2017). As seen in Figure 2.1, the chemical ordering is quite strong; each silicon atom is coupled to four oxygen atoms, and each oxygen atom is shared by two silicon atoms.

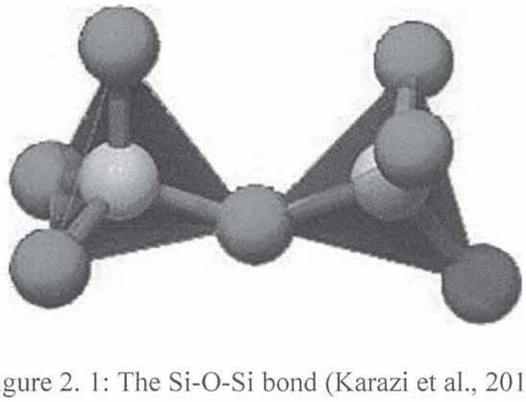

Figure 2. 1: The Si-O-Si bond (Karazi et al., 2017)