UNIVERSITI TEKNIKAL MALAYSIA MELAKA

A STUDY ON HARDNESS OF PORCELAIN BODY SINTERED AT VARIOUS TEMPERATURES

Thesis submitted in accordance with the partial requirements of the
Universiti Teknikal Malaysia Melaka for the Bachelor
of Manufacturing Engineering (Engineering Material) with Honors

By

Siti Hajar Binti Abd Rahman

Faculty of Manufacturing Engineering

May 2008

UNIVERSITI TEKNIKAL MALAYSIA MELAKA

Karung Berkunci 1200, Ayer Keroh, 75450 Melaka
Tel : 06-233 2421, Faks : 06 233 2414

FAKULTI KEJURUTERAAN PEMBUATAN

Rujukan Kami (Our Ref) :
Rujukan Tuan (Your Ref):

5 May 2008

Pustakawan
Perpustakawan Universiti Teknikal Malaysia Melaka
UTeM, No 1, Jalan TU 43,
Taman Tasik Utama, Hang Tuah Jaya,
Ayer Keroh, 75450, Melaka

Saudara,

PENGKELASAN TESIS SEBAGAI SULIT/TERHAD
- TESIS SARJANA MUDA KEJURUTERAAN PEMBUATAN (Department of Engineering Material): Siti Hajar binti Abd. Rahman
TAJUK: "A Study on Hardness of Porcelain Body Sintered at Various Temperatures"

Sukacita dimaklumkan bahawa tesis yang tersebut di atas bertajuk **"A Study on Hardness of Porcelain Body Sintered at Various Temperatures"** mohon dikelaskan sebagai terhad untuk tempoh lima (5) tahun dari tarikh surat ini memandangkan ia mempunyai nilai dan potensi untuk dikomersialkan di masa hadapan.

Sekian dimaklumkan. Terima kasih.

"BERKHIDMAT UNTUK NEGARA KERANA ALLAH"

Yang benar,

....

DR. AZIZAH BINTI SHAABAN
Pensyarah,
Fakulti Kejuruteraan Pembuatan

UNIVERSITI TEKNIKAL MALAYSIA MELAKA

BORANG PENGESAHAN STATUS LAPORAN*

JUDUL: **A Study on Hardness of Porcelain Body Sintered at Various Temperatures**

SESI PENGAJIAN: **2004/2008**

Saya **Siti Hajar binti Abd. Rahman**

mengaku membenarkan tesis (PSM/Sarjana/Doktor Falsafah) ini disimpan di Perpustakaan Universiti Teknikal Malaysia Melaka (UTeM) dengan syarat-syarat kegunaan seperti berikut:

5. Tesis adalah hak milik Universiti Teknikal Malaysia Melaka dan penulis.
6. Perpustakaan Universiti Teknikal Malaysia Melaka dibenarkan membuat salinan untuk tujuan pengajian sahaja dengan izin penulis.
7. Perpustakaan dibenarkan membuat salinan tesis ini sebagai bahan pertukaran antara institusi pengajian tinggi.
8. **Sila tandakan (√)

☐ SULIT (Mengandungi maklumat yang berdarjah keselamatan atau kepentingan Malaysia yang termaktub di dalam AKTA RAHSIA RASMI 1972)

☒ TERHAD

☐ TIDAK TERHAD

Disahkan oleh:

Alamat Tetap:
2299 Jln. Undang, Kg. Mak Kemas,
Bkt. Payong, 21400 K.Terengganu,
Terengganu

Tarikh: 12 MAY 2008

Cop Rasmi: DR. AZIZAH BINTI SHAABAN
Pensyarah
Fakulti Kejuruteraan Pembuatan
Universiti Teknikal Malaysia Melaka
Karung Berkunci 1200, Ayer Keroh
75450 Melaka

Tarikh: 12 MAY 2008

* Tesis dimaksudkan sebagai tesis bagi Ijazah Doktor Falsafah dan Sarjana secara penyelidikan, atau disertasi bagi pengajian secara kerja kursus dan penyelidikan, atau Laporan Projek Sarjana Muda (PSM).

** Jika tesis ini SULIT atau TERHAD, sila lampirkan surat daripada pihak berkuasa/organisasi berkenaan

UNIVERSITI TEKNIKAL MALAYSIA MELAKA

Karung Berkunci 1200, Ayer Keroh, 75450 Melaka
Tel : 06-233 2421, Faks : 06 233 2414
Email : fkp@kutkm.edu.my

FAKULTI KEJURUTERAAN PEMBUATAN

Rujukan Kami (Our Ref) :
Rujukan Tuan (Your Ref):

05 Mei 2008

Pustakawan
Perpustakawan Universiti Teknikal Malaysia Melaka
UTeM, No 1, Jalan TU 43,
Taman Tasik Utama, Hang Tuah Jaya,
Ayer Keroh, 75450, Melaka

Saudara,

PENGKELASAN TESIS SEBAGAI SULIT/TERHAD
- TESIS SARJANA MUDA KEJURUTERAAN PEMBUATAN (Department of Materials Engineering): Siti Hajar binti Abd. Rahman
TAJUK: A Study on Hardness of Porcelain Body Sintered at Various Temperatures

Sukacita dimaklumkan bahawa tesis yang tersebut di atas bertajuk **"A Study on Hardness of Porcelain Body Sintered at Various Temperatures"** mohon dikelaskan sebagai terhad untuk tempoh lima (5) tahun dari tarikh surat ini memandangkan ia mempunyai nilai dan potensi untuk dikomersialkan di masa hadapan.

Sekian dimaklumkan. Terima kasih.

"BERKHIDMAT UNTUK NEGARA KERANA ALLAH"

Yang benar,

Dr. Azizah binti Shaaban
Pensyarah,
Fakulti Kejuruteraan Pembuatan
☎06-2332573/019-6555707
Email: azizahs@utem.edu.my

UNIVERSITI TEKNIKAL MALAYSIA MELAKA

BORANG PENGESAHAN STATUS TESIS*

JUDUL: A STUDY ON HARDNESS OF PORCELAIN BODY SINTERED AT VARIOUS TEMPERATURES

SESI PENGAJIAN : 2004/2008

Saya SITI HAJAR BINTI ABD. RAHMAN

mengaku membenarkan tesis (PSM/Sarjana/Doktor Falsafah) ini disimpan di Perpustakaan Universiti Teknikal Malaysia Melaka (UTeM) dengan syarat-syarat kegunaan seperti berikut:

1. Tesis adalah hak milik Universiti Teknikal Malaysia Melaka .
2. Perpustakaan Universiti Teknikal Malaysia Melaka dibenarkan membuat salinan untuk tujuan pengajian sahaja.
3. Perpustakaan dibenarkan membuat salinan tesis ini sebagai bahan pertukaran antara institusi pengajian tinggi.
4. **Sila tandakan (√)

☐	SULIT	(Mengandungi maklumat yang berdarjah keselamatan atau kepentingan Malaysia yang termaktub di dalam AKTA RAHSIA RASMI 1972)
☐	TERHAD	(Mengandungi maklumat TERHAD yang telah ditentukan oleh organisasi/badan di mana penyelidikan dijalankan)
☐	TIDAK TERHAD	

Disahkan oleh:

(TANDATANGAN PENULIS)

(TANDATANGAN PENYELIA)

Alamat Tetap:
2299 Jln. Undang, Kg. Mak Kemas,
Bkt. Payong, 21400 K. Terengganu,
Terengganu.

Cop Rasmi: DR. AZIZAH BINTI SHAABAN
Pensyarah
Fakulti Kejuruteraan Pembuatan
Universiti Teknikal Malaysia Melaka
Karung Berkunci 1200, Ayer Keroh
75450 Melaka

Tarikh: 12 MAY 2008

Tarikh: 12 MAY 2008

** Jika tesis ini SULIT atau TERHAD, sila lampirkan surat daripada pihak berkuasa/organisasi berkenaan dengan menyatakan sekali sebab dan tempoh tesis ini perlu dikelaskan sebagai SULIT atau TERHAD.

APPROVAL

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

....

Dr. Azizah Binti Shaaban
(Project Supervisor)

DR. AZIZAH BINTI SHAABAN
Pensyarah
Fakulti Kejuruteraan Pembuatan
Universiti Teknikal Malaysia Melaka
Karung Berkunci 1200, Ayer Keroh
75450 Melaka

DECLARATION

I hereby declare that this report entitled **"A STUDY ON HARDNESS OF PORCELAIN BODY SINTERED AT VARIOUS TEMPERATURES"** is the result of my own research except as cited in the references.

Signature	:	....
Author's Name	:	**Siti Hajar Binti Abd Rahman**
Date	:	12 MAY 2008

ABSTRACT

This project is carried out to study the hardness of porcelain body sintered at temperature range between 1230°C to 1350°C. Beside hardness and density, the microstructure of the porcelain body is also observed. Starting material used is commercial porcelain powder that is prepared trough spray drying process. Commercial porcelain contains feldspar, kaolin and quartz and binders such as bentonite clay. Characterization of starting material is done using Scanning Electron Microscope (SEM).Powder characterization is also important to determine the particle size of porcelain powder. The powder is then compacted and sintered at 1230°C to 1350°C temperature range. Hardness test, density measurement and microstructure observation is carried out to determine the effect of various sintering temperature. Energy Dispersive X-Ray (EDX) analysis and X-Ray Diffraction (XRD) analysis is done to determine the chemical and phase composition of porcelain body. Results indicate that the optimum sintering temperature is at 1250°C where maximum hardness and density are achieved at this temperature. The hardness value at this sintering temperature is HV707.36, density 2.461g/cm^3 and linear shrinkage of 9.36%. The microstructural analysis revealed that the ideal firing temperature occurs when the glassy phase covers mostly the entire sample surface with minimum pores appearance. X-ray diffractograms of samples fired in the optimum firing temperature show that the samples are composed by quartz, mullite and glassy phase. It is found out that this commercial porcelain body from spray dried porcelain powder produced by this particular manufacturer posses the optimum hardness, density and microstructure at even lower temperature compared to the other traditional porcelain's.

ABSTRAK

Projek ini dijalankan bagi mengkaji kekerasan jasad porselin yang disinter pada julat suhu 1230°C sehingga 1350°C. Selain kekerasan dan ketumpatan struktur mikro jasad porselin turut dikaji. Bahan awal yang digunakan dalam kajian ini ialah serbuk porselin komersial yang disediakan melalui proses semburan kering. Porselin komersial mengandungi feldspar, kaolin dan kuarza beserta bahan pengikat seperti tanah liat bentonite. Penentuan karakteristik bahan asal dijalankan dengan menggunakan *Scanning Electron Microscope* (SEM). Penentuan karakteristik adalah penting terutamanya dalam menetukan saiz setiap partikal serbuk porselin. Serbuk ini kemudiannya akan dimampatkan dan disinter pada julat suhu 1230°C sehingga 1350°C. kekerasan, ketumpatan, dan pemerhatian terhadap struktur mikro dijalankan bagi menentukan kesan pelbagai suhu persinteran. Analisis Energy Dispersive X-Ray (EDX) dan X-Ray Diffraction (XRD) dijalankan bagi menetukan kandungan kimia dan fasa jasad porselin. Keputusan menunjukkan suhu persinteran optimum ialah pada 1250°C di mana kekerasan dan ketumpatan maksumim dicapai pada suhu ini. Nilai kekeran pada suhu ini ialah HV707.36, ketumpatan 2.461g/cm^3 dan pengecutan linear ialah 9.36%. Analisis struktur mikro mendedahkan bahawa suhu persinteran yang ideal terjadi apabila fasa berkaca melitupi hamper keseluruhan permukaan sampel dengan kehadiran liang yang minimum. Analisis XRD pada suhu optimum menunjukkan sampel mengandungi kuarza, mullite dan fasa berkaca. Penemuan telah mendapati bahawa jasad porselin daripada serbuk porselin semburan kering yang dihasilkan oleh syarikat pembuatan ini memiliki kekerasan dan ketumpatan bersama dengan keadaan struktur mikro yang optimum pada suhu yang lebih rendah berbanding dengan porselin tradisional yang lain.

ACKNOWLEDGEMENT

I would like to express my gratitude to all those who gave me the possibility to complete this PSM report. I want to thank the Faculty of Manufacturing Engineering and Department of Material for giving me permission to commence this Projek Sarjana Muda in the first instance, provide the necessary facilities and to use departmental data.

I extend my sincere gratitude to all staffs in this department especially all the technicians for shepherding and never hesitated to help me whenever I step into the laboratory. The helpfulness has made my tasks become easier.

I have furthermore to thank the PSM Committee Members for the good arrangement and caring of student's problem along the PSM period. I am deeply indebted to my PSM supervisor Dr. Azizah Shaaban whose help, stimulating suggestions and encouragement helped me in all the time of tasks regarding the PSM and writing of this report.

I am bound to lecturers in Department of Material and FKP theirs wise counsel, advice, and stimulating support. My former colleagues who are in the same journey as me supported me in tasks and projects. I want to thank them for all their help, support, interest, and valuable hints. Especially I am obliged to my beloved mother, Fatimah Abd. Ghani who is always be there giving me support, strength and great help in difficult times. Last but not least, I wish to acknowledge to all persons who give supporting, advice, and assistance that are directly or indirectly involved to the success of my PSM. Thank you so much.

TABLE OF CONTENT

LIST OF FIGURE

LIST OF TABLE

LIST OF ABBREVIATION

SEM	-	Scanning Electron Microscope
EDX	-	Energy Dispersive X-Ray
XRD	-	X-Ray Diffraction
HV	-	Vickers Hardness

CHAPTER 1

INTRODUCTION

1.1 Introduction to Porcelain

Porcelain literally brings the meaning of a hard, white, translucent ceramic made by firing pure clay and then glazing it with variously colored materials. Porcelain is a vitrified product of mixtures of clay, quartz and feldspar. Porcelain microstructures are grain and bond type with large particles of filler (usually quartz) held together by a finer matrix, which is almost fully dense, composed by mullite crystals and a glassy phase (Marquez J. M. et al. 2007). Because of the complex interplay between raw materials, processing routes and approaches, and the kinetics of the firing process, porcelains represent some of the most complicate ceramic systems (Kamseu *et al*. 2005).

Although porcelain is frequently used as a synonym for china, the two are not identical. They resemble one another in that both are vitreous wares of extremely low porosity, and both can be glazed or unglazed. However, china, also known as soft-paste or tender porcelain, is softer: it can be cut with a file, while porcelain cannot. This difference is due to the higher temperatures at which true porcelain is fired, 2,650 degrees Fahrenheit (1,454 degrees Celsius) compared to 2,200 degrees Fahrenheit (1,204 degrees Celsius) for china. Due to its greater hardness, porcelain has some medical and industrial applications while china is limited to domestic and artistic use. Moreover, porcelain is always translucent, while china is opaque.

In engineering field, porcelain is a ceramic material made by heating selected and refined materials, often including clay in the form of kaolinite, to high temperatures. The raw materials for porcelain, when mixed with water, form a plastic body that can be worked to a required shape before firing in a kiln at temperatures between 1200 and 1400 degrees Celsius. The toughness, strength, and translucence of porcelain arise mainly from the formation at high temperatures of glass and the mineral mullite within the fired body. Properties associated with porcelain beside as mention above include low permeability, hardness, glassiness, high durability, whiteness, resonance, brittleness, high resistance to the passage of electricity, high resistance to chemical attack, high resistance to thermal shock and high elasticity.

Porcelain is widely applied in manufacturing field to produce table, kitchen, decorative and sanitary wares, objects of fine art and tiles. Porcelain also widely used for high voltage insulators because of its high resistance to the passage of electricity. It is also used in dentistry. Porcelain veneers for example have been successfully used to mask teeth with discolored and poorly shaped (Figure 1.1).

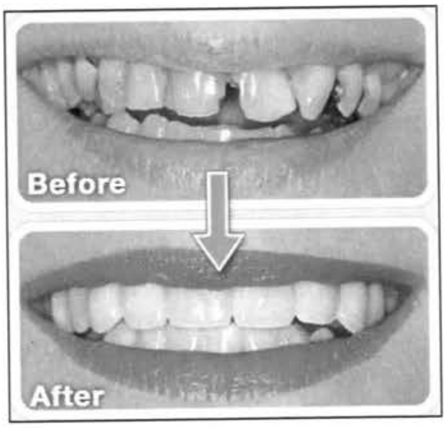

Figure 1.1. Porcelain veneers used to mask teeth

The primary components of porcelain are clays, feldspar or flint, and silica, all characterized by small particle size. To create different types of porcelain, craftspeople combine these raw materials in varying proportions until they obtain the desired green (unfired) and fired properties.

Although the composition of clay varies depending upon where it is extracted and how it is treated, all clays vitrify (develop glassy qualities), only at extremely high temperatures unless they are mixed with materials whose verification threshold is lower. Unlike glass, however, clay is refractory, meaning that it holds its shape when it is heated. In effect, porcelain combines glass's low porosity with clay's ability to retain its shape when heated, making it both easy to form and ideal for domestic use.

The principal clays used to make porcelain are china clay and ball clay, which consist mostly of kaolinate, a hydrous aluminum silicate.

Feldspar, a mineral comprising mostly aluminum silicate, and flint, a type of hard quartz, function as fluxes in the porcelain body or mixture. Fluxes reduce the temperature at which liquid glass forms during firing to between 1,835 and 2,375 degrees Fahrenheit (1,000 and 1,300 degrees Celsius). This liquid phase binds the grains of the body together.

Silica is a compound of oxygen and silicon, the two most abundant elements in the earth's crust. Its resemblance to glass is visible in quartz (its crystalline form), opal (its amorphous form), and sand (its impure form). Silica is the most common filler used to facilitate forming and firing of the body, as well as to improve the properties of the finished product. Porcelain may also contain alumina, a compound of aluminum and oxygen, or low-alkali containing bodies, such as steatite, better known as soapstone.

1.2 Introduction to Project

Porcelain powder prepared trough spray-drying process in the other hand consist of selected clays, kaolinitic minerals, quartz and feldspar, shaped by dry pressing to form a ceramic material that is then fired at temperatures up to 1250°C. Porcelain is resistant to wear, deep abrasion, and frost. It is a practically non-absorbent material with great flexural strength. Quality porcelain will remain constant over time. Porcelain is ideal for interior and exterior floors and walls and even facades of buildings. It is available in natural, smooth, polished and structured finishes (Sanchez E. *et al.* 2006).

The high technological properties of porcelain stoneware, especially as regards water absorption, chemical and frost resistance, bending strength and abrasion resistance, cause that porcelain mold was actually the material with the highest increase in application especially in latex glove mold (Figure 1.2). Typical composition of commercial spray dried porcelain is 40–50 wt% kaolin clays, 35–45 wt% feldspars; 10-15wt% quartz (Sanchez E. *et al.* 2006).

However, the commercial interest in porcelain is accompanied by a scarcity of high quality research and although it is possible to find in the literature several papers on this material, they are mainly focused on processing (Chen C.Y. & tuan W.H 2001, Marquez J.M. et al. 2007) feasibility of different raw materials to produce porcelain stoneware tiles (Braganca S.R. & Bergmann C.P 2002) and general information.

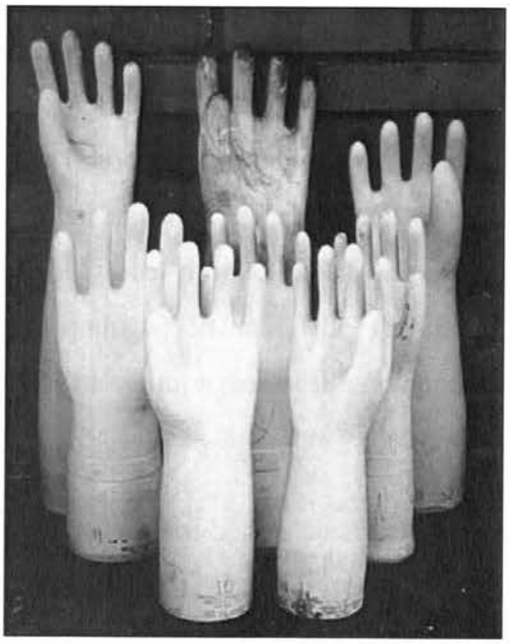

Figure 1.2. Porcelain mold for gloves

The present work is part of a more extensive investigation with focus more on the commercial spray dried porcelain powder that is used to produce latex glove mold with their sintered porcelain body properties. The first is to establish the effect of sintering temperature (with sintering range of 1230-1350°C) on physical and mechanical properties of porcelain body and secondly, to study the effect of microstructure gain from the optimum sintering temperature. For this purpose, it is very important to have a previous knowledge of the behaviour of a porcelain body during the firing process, being this the objective of the present study. With this aim, a porcelain sample has been produced with following criteria:

- As receive powder was a commercial composition, similar to that used in production of latex glove mold, but without additive supplement, such as synthetic inorganic pigments synthetic inorganic pigments or pressing additives as binders or plasticizers, and,

- Sample was sintered at various sintering temperature to determine the optimum sintering temperature that employ maximum value of properties.

1.3 Problem Statement

Porcelain stoneware is a glass-bonded material with excellent technical performances for wide application, such as mechanical, wear, frost and chemical resistance. In the last decade, the growth rate of the global production of porcelain stoneware tiles increased more than other ceramic products; in fact, the technical properties of porcelain stoneware, coupled with even more improved aesthetic appearance, gave it an important role on market.

Commercial porcelain stoneware bodies consist mainly of a mixture of ball clays (30-40%), feldspars (45-50%), quartz-feldspathic sands (10-15%) and several additives such as glass-ceramics frits and pigments. The tiles are obtained by wet grinding, spray-drying, dry pressing, fast drying, and are sintered by fast single firing (<60 minutes cold-to-cold, 1180-1200°C of maximum temperature and 5 minutes soaking times). Finished products such as used in outdoor application have strict requirements in terms of water absorption (<0.5%) and planarity and this only can be achieved with porcelain instead of the other ceramic materials. The ceramic material is made up of crystalline phases, both new formed (i.e. mullite) and residual ones (quartz, feldspars) embedded in an abundant glassy matrix.

The main limit in further development of porcelain stoneware is the presence of a closed porosity (usually 2-8%).The industrial sintering process is not able to remove completely the residual porosity, consisting of both small spheroidal, gas-filled pores and irregularly shaped, large pores probably resulting from coalescence of smaller ones. The industrial attention has been especially paid to the intermediate stage of sintering, where densification occurs with a linear shrinkage around 7-9%; the final stage has been less concerned, notwithstanding the closed pores happened to be to a large extent unsinterable, since prolonged firing resulted in swelling by coarsening.

Studies on the mechanisms active during the whole sintering path of porcelain, in order to achieve the phenomenological model of the process have been done by the several previous studies. The different in sintering temperature used to sinter the commercial will then affect on the dense of porcelain body that is developed during sintering process. This study is then also look at the hardness effect and microstructure study of sintered commercial porcelain with this variety of sintering temperature.

1.4 Objectives

The purposes of this project are:

1) To study the microstructure, mechanical properties, and hardness of porcelain body sintered at 1230°C to 1350°C
2) To evaluate the most optimum sintering temperature for commercial porcelain.

1.5 Scope of Study

Pre-receive material is commercial porcelain powder that is prepared trough spray drying process. Dry pressing will be was carried out to form green body. Dense porcelain body is developed trough sintering technique. The sample microstructure will be observed using SEM. Hardness test and density of porcelain is then will be carried out to determine the mechanical properties of commercial porcelain sintered at 1230°C to 1350°C. Chemical analyses involved are EDX and XRD to determine the element and phase exist in the porcelain that is sintered at optimum sintering temperature