UNIVERSITI TEKNIKAL MALAYSIA MELAKA

Mechanical Properties of Mullite-Bonded Silicon Carbide Sintered at Various Heating Temperature

Thesis submitted in accordance with the requirements of the Universiti Teknikal Malaysia Melaka for the Degree of Bachelor of Engineering (Honours) Manufacturing Engineering (Engineering Materials)

By

Lim Yee Kai

Faculty of Manufacturing Engineering

March 2008

UNIVERSITI TEKNIKAL MALAYSIA MELAKA

BORANG PENGESAHAN STATUS LAPORAN PSM

JUDUL:

Mechanical Properties of Mullite bonded Silicon Carbide Sintered At Various Heating Temperature

SESI PENGAJIAN: Semester 2 2007/2008

Saya LIM YEE KAI

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

1. Laporan PSM / tesis adalah hak milik Universiti Teknikal Malaysia Melaka dan penulis.
2. Perpustakaan Universiti Teknikal Malaysia Melaka dibenarkan membuat salinan untuk tujuan pengajian sahaja dengan izin penulis.
3. Perpustakaan dibenarkan membuat salinan laporan PSM / 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	

(LIM YEE KAI)
Alamat Tetap:
No.2, JLN 5/2,
Taman Daya, Kepong,
52100 Kuala Lumpur

(DR. MOHD WARIKH BIN ABD RASHID)

Cop Rasmi:

Tarikh: 4/5/2008

Tarikh: ______________

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

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):

01 Mei 2008

Pustakawan
Perpustakaan Universiti Teknikal Malaysia Melaka (UTeM)
Taman Tasik Utama, Hang Tuah Jaya,
Ayer Keroh, 75450, Melaka

Saudara,

PENGKELASAN LAPORAN PSM SEBAGAI SULIT/TERHAD
- LAPORAN PSM SARJANA MUDA KEJURUTERAAN PEMBUATAN (BAHAN KEJURUTERAAN): LIM YEE KAI
TAJUK: Mechanical Properties of Mullite bonded Silicon Carbide Sintered At Various Heating Temperature

Sukacita dimaklumkan bahawa tesis yang tersebut di atas bertajuk " **Mechanical Properties of Mullite bonded Silicon Carbide Sintered At Various Heating Temperature**" 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. MOHD WARIKH BIN ABD RASHID
Pensyarah,
Fakulti Kejuruteraan Pembuatan

DECLARATION

I hereby declare that this report entitled "Mechanical Properties of Mullite-Bonded Silicon Carbide Sintered at Various Heating Temperature" is the result of my own research except as cited in the references.

Signature		
Author's Name	:	**LIM YEE KAI**
Date	:	30/4/2008

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). The members of the supervisory committee are as follow:

DR. MOHD WARIKH BIN ABD RASHID

(PSM Supervisor)

ABSTRACT

Mullite-bonded porous silicon carbide samples are produced by the reaction sintering in air from silicon carbide (SiC), alumina (Al_2O_3), and using graphite as the pore former. The sintering process is based on the oxidation of SiC and the mullitization between Al_2O_3 and oxidation derived SiO_2 (Cristobalite). At mullitization temperature (1450^0C), SiC particles are bonded by the mullite ($3Al_2O_3.2SiO_2$) and SiO_2. Sintered samples will be characterized by density determination and three-point bend tests. Morphology and microstructure analysis study will be done by scanning electron microscopy, while the phase composition will be investigated by X-ray diffraction. Mullite phase transformation start at 1450^0C which was the optimum sintering temperature for mullite transformation. The mullite content increase with sintering temperature concurrent with decrease of Al_2O_3. Porous SiC ceramics were bonded by the mullite (needle like) and oxidation-derived SiO_2. The densities was increase while the open porosity was decreases with increasing of sintering temperature. Flexural strength was direct proportion to the sintering temperature but inverse proportional to the percentage of open porosity. Highest content of mullite and mechanical properties achieved at 1500^0C,

ABSTRAK

Poros Mullite-bonded silikon karbida dihasilkan oleh reaksi pensinteran dalam udara dari silikon karbida (SiC), alumina (Al_2O_3), dan menggunakan grafit sebagai pembentuk liang. Proses sinter adalah berasaskan pengoksidaan SiC dan mullitization antara Al_2O_3 dan pengoksidaan terbitan SiO_2 (Kristobalit). Di mullitization suhu, zarah-zarah SiC adalah diikat oleh mulit ($3Al_2O_3.2SiO_2$) dan SiO_2. Sampel yang tersinter akan dijalankan ujian ketumpatannya and kelenturan tiga mata. Morfologi dan mikrostruktur kajian analisis akan dibuat oleh mikroskop elektron pengimbasan (SEM), manakala komposisi fasa akan disiasat oleh belauan sinar X (XRD). Mulit permulaan penjelmaan fasa pada 1450^0C merupakan optimum pensinteran suhu untuk mulit tranformasi. Mulit peningkatan dengan pensinteran suhu serentak dengan pengurangan Al_2O_3. SiC yang poros seramik adalah diikat oleh mulit (bentuk jejarum) dan pengoksidaan terbitan SiO_2. Ketumpatan adalah meningkat manakala keporosan terbuka adalah berkurangan dengan penambahan pensinteran suhu. Lenturan kekuatan hádala berkadar terus dengan pensinteran suhu tetapi berkadar songsangan dengan peratusan keporosan. Kandungan mulit dan ciri-ciri mekanikal tertinggi dicapai pada 1500^0C,

ACKNOWLEDGEMENTS

I would like to thanks and deepest gratitude from bottom of my heart for all the support, encouragement and inspiration that I obtain to finish these thesis.

My special thanks to my supervising lecturer, Dr. Mohd Warikh, which we had a good working relationship and who offered help, guidance and encouragement during prepare this thesis.

I also thank to my family for their constant encouragement and support. To all of my lecturers, technician, and friends who help, guide and encourage me.

TABLE OF CONTENTS

APPENDICES

LIST OF FIGURES

LIST OF TABLES

LIST OF ABBREVIATIONS, SYMBOLS, SPECIALIZED NOMENCLATURE

Al_2O_3	-	Aluminium Oxide
$3Al_2O_3.2SiO_2$	-	Mullite
SiC	-	Silicon Carbide
PVA	-	Polyvinyl Alcohol
EDX	-	Energy Dispersion X-ray
SEM	-	Scanning Electron Microscope
XRD	-	X-ray diffraction
ϕ	-	Percentage of porosity
V_V	-	volume of void-space
V_T	-	total or bulk volume of material
σ_o	-	strength of a nonporous structure
σ	-	strength of the pore characteristic
P	-	porosity
b	-	constant depending on the pore characteristics

CHAPTER 1

INTRODUCTION

1.1 Overview

Ceramic are inorganic, nonmetallic materials and can be crystalline or amorphous. Ceramic are compounded with metallic and non-metallic element, such as aluminum or calcium, oxygen or silicon and nitrogen. Normally, sintered ceramics are required to have minimum porosity. However, there are cases where porosity is desirable for example in humidity and gas sensors and where there thermal shock resistance is of overriding importance. Porous silicon carbide ceramic is one of the potential materials to be used as filters, membranes, catalytic substrates, thermal insulation, gas burner media, and refractory materials due to of their excellent mechanical and chemical stability, such as low bulk density, high permeability, high temperature stability, erosion and corrosion resistance, and excellent catalytic activity.

The high degree of covalence in SiC ceramic makes it difficult to sinter SiC by heating of powder compacts at moderate temperature. Hence, many different sintering techniques have been developed. Among those techniques, hot-pressing with a sintering aid, pressureless sintering with a sintering aid and reaction sintering are the important one. In this study, in situ reaction bonding technique is use in low temperature fabrication of SiC ceramic.

In reaction bonding technique, a secondary phase may add and utilize the oxidation derived silica to bond SiC particles with mullite bonding. Mullite ($3Al_2O_3$ $.2SiO_2$) is the intermediate phase in the SiO_2-Al_2O_3 system. It has excellent high temperature properties with improved thermal shock and thermal stress owing to the low thermal expansion, good strength and interlocking grain structure. It is believed that mullite bonding will possesses better high temperature stability and oxidation resistance, matching thermal expansion and good chemical compatibility between mullite and SiC.

The effect of charge composition, characteristic of the starting material (silicon carbide, SiC and alumina, Al_2O_3) and pore-forming agent (graphite) on the formation of highly porous SiC-based by in situ reaction bonding will be studied. Pores in porous SiC ceramics can be made by burning out the graphite during sintering process. These can keep the skeleton of the green bodies intact before the bonding phase formed at higher temperature.

The fabrication process of synthetic mullite porous ceramic involves precision mixing, forming and sintering. The preparation of SiC green body is using reaction sintering below the melting point. Then sample is test for its mechanical properties; microstructure and phase composition analysis. The effect of sintering temperature on properties of porous SiC ceramics such as porosity and flexural strength will be study.

1.2 Statements of problem

Ceramic materials face a stiff competition from metal alloy and composites in the extreme environment applications. In order to compete well, new and low cost processing method with high performance ceramic is needed. In the meantime, porous silicon carbide ceramic have wide application in filtration, separation, catalysis and high temperature structural material. As diesel particulate filter, SiC ceramic has a higher melting temperature (2700^{o}C), better thermal shock resistance and high chemical stability but with lower mechanical properties compare to metal alloy. Further, the SiC ceramic also facing major problem in fabrication process due to the natural high degree of covalency in SiC makes it difficult to sinter to high densification by normal heating of powder compacts. As a result, various low temperature sintering techniques have been developed. This reaction bonded silicon carbide (RBSC) technique required no machining and low cost manufacturing technique. This technique had solved the problem of high cost techniques (hot press and hot isostatic press) of sintering and high cost of machining after sintering because it able to produce very low shrinkage of sintered ceramic. The reaction sintering in pressureless environment further reduces the fabrication cost of the material. The low mechanical strength of the porous SiC ceramic can be improved by mullite bonding during reaction sintering. Therefore, in this study of alumina and graphite will be added for reaction sintering in order to produce mullite bonded which exhibit excellent high temperature strength. According to Ding et al., (2007), the flexure strength of porous ceramic is highly dependent on the sintering temperature. The strength of porous ceramics is exponential increase with the decrease of the porosity and the porosity also inverse proportional to raising sintering temperature. Subsequently, it is important to determine the optimum sintering temperature in the fabrication of mullite bonded porous silicon carbide with improved mechanical properties.

1.3 Objectives

The purposes of this project are:

i. To identify the optimum sintering heating temperature for mullite-bonded porous SiC ceramic with highest mechanical properties.
ii. To examine the morphology and microstructure of porous SiC ceramic at elevated temperature.
iii. To investigate the phase composition evolution at elevated temperature

1.4 Scopes of the project

This project studies the effect of elevated sintering temperatures on porous SiC ceramic by in-situ reaction bonding technique. Mullite formed during the sintering process will act as the bonding phase between silicon carbide. The effect of the bonding material on the porous microstructure will be examined under SEM. Phase evolution was investigated using X-ray diffraction. The mechanical properties of porous SiC ceramic will be studied in the aspect of percentage of porosity and flexural strength.

CHAPTER 2

LITERATURE REVIEW

This chapter reviewed the basic theory on properties and mechanical testing on mullite bonded porous silicon carbide. It also cover various study and analysis results and finding from previous researches. Most of the review base on fabrication and sintering of silicon carbide with the effect of temperature, material composition, porosity and material characteristic.

2.1 Overview of Ceramic material

Ceramic (Munz *et al.*, 2001) can be classified into different group by considering their chemical composition, microstructure, or application. We can distinguish the traditional ceramics and advanced based on their application. The traditional ceramics include tableware, pottery, sanitary ware, tiles, bricks and clinker. The advance ceramic can be divided into electronic ceramics (insulators, substrates, capacitors, varistors, actuators, and sensors), optical ceramics (window, laser), magnetic ceramics and structural ceramics. The structural ceramic was applied in mechanical engineering, chemical engineering, high temperature technology, and in biomedical technology. Special ceramic which were not directly related to the categories mentioned above were reactor ceramics (absorber materials, breeder materials, nuclear fuels) and refractory product.

Classification of ceramic was to distinguish between

- Silicate ceramics
- Oxide ceramics
- Non-oxide ceramics

The classification was a mixture of composition (oxide, non-oxide) and atomic structure (glassy-amorphic, crystalline). The main feature of silicate ceramics was the glass-amorpic phase with a pronounced pore structure. The main content was SiO_2 with additions of Al_2O_3, MgO, BeO, ZrO_2 and other oxides. The further subdivision was between clay-ceramic with mullite ($3Al_2O_3.2SiO_2$) as the main constituent and other silica ceramics, e.g. cordierite ($2MgO.2Al_2O_3.5SiO_2$). Clay ceramics were subdivided into those with coarse grain structures. Earthenware, tableware, porcelain and tiles belong to the first category. Bricks, clay pipes, and clinker belong to the latter.

Oxide ceramics were different from silica ceramic due to it have major crystalline phase with only a small content of glassy phase.
Example of oxides:

Al_2O_3, BeO, MgO, ThO_2, TiO_2, UO_2, ZrO_2

The properties of single oxide can be modified by additives. Al_2O_3 ceramic was the dispersion toughened ceramic with fine dispersion of particle of ZrO_2 or TiC. The system Al_2O_3- ZrO_2 was name ZTA (zirconia-toughened aluminium oxide).

2.2 Silicon Carbide

According to Munro (1997) sintered silicon carbide ceramics was produced by using submicrometer powders that extracted from an Acheson furnace and ground to a fine particle size. Acheson furnace (Saddow *et al.*, 2004) was invented by

Eugene and Alfred Cowles and this furnace was adopted by Acheson to produce suitable materials that could substitute diamond as an abrasive and cutting material. Acheson mixed coke and silica in the furnace and discover a crystalline product which have great hardness, refractability, and infusibility, which was the compound of carbon and silicon. The product was given by name "carborundum" with formula SiC (Figure 2.1). SiC was sinter at temperature 2500 °C with boron and carbon as sintering aids to achieve improved densification during sintering. However, Evans et al. had realized that silicon carbide was very difficult to manufacture and the fully dense sintered SiC raw material was very expensive.

Riedel (2000) had stated that SiC was the most important carbide due to its extreme properties and potential commercial applications and its belongs to the group of nonmetallic hard materials (Figure 2.2) which had great hardness and high melting point result from a high fraction of covalent bonding. Superhard compounds were obviously formed by combination of the four low atomic number elements: boron, carbon, silicon, and nitrogen to form a quaternary system; carbon as diamond, boron nitrogen as cubic boron nitride, boron-carbon as boron carbide, and silicon-carbon as silicon carbide belong to the hardest materials.

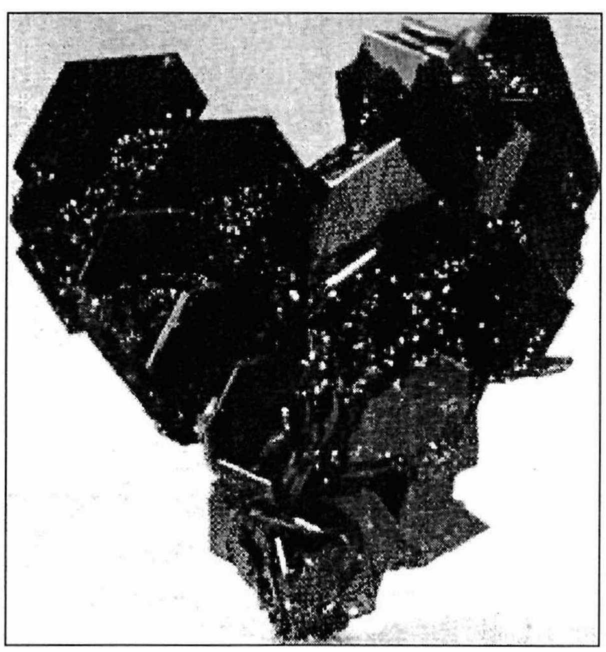

Figure 2.1: Crystalline SiC grain from the Acheson process