



**UNIVERSITI TEKNIKAL MALAYSIA MELAKA**

# **Flexural Strength and Analysis of Mullite Bonded Silicon Carbide Sintered at Various Heating Period**

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

By

**Mohd Fahmi Bin Shafie**

Faculty of Manufacturing Engineering

May 2008



**UNIVERSITI TEKNIKAL MALAYSIA MELAKA**

**BORANG PENGESAHAN STATUS TESIS\***

**JUDUL: FLEXURAL STRENGTH AND ANALYSIS OF MULLITE-BONDED SILICON  
CARBIDE SINTERED AT VARIOUS HEATING PERIOD**

SESI PENGAJIAN: 2/2007-2008

Saya MOHD FAHMI BIN SHAFIE

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:  
1236 Kg Belukar Jambu,  
21080 K. Terengganu,  
Terengganu.

Cop Rasmi:

Tarikh:

Tarikh:

\* 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 dengan menyatakan sekali sebab dan tempoh tesis ini perlu dikelaskan sebagai SULIT atau TERHAD.

## **APPROVAL**

This thesis submitted to the senate of UTeM and has been accepted as partial fulfillment of the requirements for the degree of Bachelor of Manufacturing Engineering (Engineering Material). The members of the supervisory committee are as follow:

.....

Main Supervisor  
(Official Stamp and Date)

## DECLARATION

I hereby, declared this thesis entitled “Flexural Strength and Analysis of Mullite-Bonded Silicon Carbide Sintered at Various Heating Period” is the results of my own research except as cited in references

Signature : .....

Author's Name : .....

Date : .....

## **ABSTRACT**

Mullite bonded silicon carbide porous ceramic has been fabricated by differentiate the sintering period to investigate the effect to the flexural strength and also the other properties such as water absorption, apparent porosity, apparent specific gravity and bulk density. In this case, three main materials have been used which are silicon carbide and alumina while graphite has been used as pore former. Apart of that, the phase of the mullite bonded silicon carbide porous ceramic has been analyzed and its microstructure also has been observed to confirm the existing of the mullite bonding at temperature 1450 °C. From the Archimedes test that has been conducted, the percentage of water absorption and apparent porosity have been decreased with the increasing of the sintering period while the apparent specific gravity and bulk density have been increased with the increasing of the sintering period. The flexural strength which is done by three point bending test found that the flexural strength has been increased with the increasing of the sintering period. From the phase analysis and the microstructure observation, it has been proved that the mullite bonding was existed. The optimum sintering time that have been chosen is 12 hours sintering period base on the flexural strength result.

## ABSTRAK

Seramik berliang daripada silika karbida ikatan mullite telah dihasilkan dengan masa pembakaran yang berbeza untuk menyiasat kesan terhadap kekuatan lenturan dan juga terhadap sifat-sifat yang lain seperti daya penyerapan air, keliangan graviti spesifik dan juga ketumpatan pukal. Dalam kes ini, tiga bahan utama telah digunakan iaitu silika karbida dan alumina manakala grafit bertindak sebagai penghasil liang. Selain daripada itu, fasa seramik berliang daripada silika karbida ikatan mullite telah dianalisis dan struktur mikro telah diteliti untuk memastikan kewujudan ikatan mullite pada suhu 1450 °C. Dari kaedah Archimedes yang telah dijalankan, peratusan serapan air dan keliangan telah berkurang dengan peningkatan masa pembakaran manakala graviti spesifik dan ketumpatan pukal telah meningkat dengan peningkatan masa pembakaran. Ujian kekuatan lenturan dilakukan dengan menggunakan kaedah pembengkokan tiga titik mendapati kekuatan lenturan meningkat dengan peningkatan masa pembakaran. Daripada analisis fasa dan penelitian struktur mikro, ikatan mullite telah terbukti wujud. Masa optimum yang telah dipilih ialah 12 jam berdasarkan kekuatan leturan yang paling tinggi.

## **DEDICATION**

*For my beloved mother and father, family and also the whole members of UTeM.*

## **ACKNOWLEDGEMENT**

I want to express my gratitude to the Almighty Allah s.w.t who have giving me strength from time to time to finish this final report. To the family members especially my mother, Mrs Ziamah Che Embong and my father, Mr Shafie bin Bakar who never felt tired to support me from behind in moral and financial aspects. Special Thanks to my supervisor, Dr. Mohd Warikh bin Abd Rashid and my former supervisor, Mr Lau Kok Tee who gave a lot of advises and encouragement in completing this report. To all of the technicians that involved in completing this project, Mr Sarman, Mr Ismail, Mr Hisyam, Mr Rashdan and Mr Azhar Shah, thanks a lot for the help. Also to my team members, Hanafi bin Suhaimi, Ahmad Fitri bin Ab Halim, Muhammad Affendi bin Mustafa and Lim Yee Kai who have assisted me a lot along the accomplishment of this project. Last but not least, to all of the housemate and friends, thanks a lot for the support.



# TABLE OF CONTENTS

Abstract	i
Abstrak	ii
Dedication	iii
Acknowledgement	iv
Table of Contents	v
<b>1. INTRODUCTION</b>	<b>1</b>
1.1 Background	1
1.2 Problem Statement	2
1.3 Hypothesis	2
1.4 Objectives	2
1.5 Scope of Study	3
<b>2. LITERATURE REVIEW</b>	<b>4</b>
2.1 Overview of Raw Material	4
2.1.1 Aluminum Oxide	4
2.1.1.1 Properties of Aluminum Oxide	5
2.1.1.2 Application of Aluminum Oxide	5
2.1.2 Silicon Carbide	6
2.1.2.1 Properties of Silicon Carbide	7
2.1.2.2 Application of Silicon Carbide	7
2.1.3 Graphite	7
2.1.3.1 Properties of Graphite	8
2.1.3.2 Application of Graphite	9
2.2 Mechanical Properties of Ceramics	9
2.2.1 Flexural Strength	9
2.2.2 Effect of Porosity on the Mechanical Properties	10
2.3 Reaction Sintering for Silicon Carbide	11
2.4 Mullite as Bonding Material	11
2.5 Effect of Sintering Period	12

<b>3. METHODOLOGY</b>	<b>16</b>
3.1 Sample Preparation	16
3.1.1 Material Mixture	17
3.1.2 Ball Mill Mixing	17
3.1.2.1 Ball Mill Procedure	18
3.1.3 Powder Compaction	18
3.1.3.1 Powder Compaction Procedure	19
3.1.4 Sintering Process	20
3.1.4.1 Sintering Procedure	21
3.1.5 Dimensioning Sample	22
3.1.5.1 Dimensioning the Thickness	23
3.1.5.2 Dimensioning the Width and Length	23
3.2 Testing and Analysis	24
3.2.1 Three Point Bending Test	25
3.2.1.1 Three Point Bending Procedure	25
3.2.2 Archimedes Test	26
3.2.2.1 Archimedes Test Procedure	26
3.2.3 Phase Analysis	27
3.2.4 SEM Observation	28
<b>4. RESULTS</b>	<b>30</b>
4.1 Temperature Consistency	30
4.2 Weight Loss	31
4.3 Archimedes Test	32
4.3.1 Apparent Porosity	33
4.3.2 Water Absorption	35
4.3.3 Apparent Specific Gravity	36
4.3.4 Bulk Density	37
4.4 XRD Analysis	39
4.5 Flexural Strength	41

<b>5. DISCUSSION</b>	<b>42</b>
5.1 In Situ Reaction Bonding Behavior	42
5.2 Flexural Strength	43
5.3 Effect of Sintering Period	43
5.4 Microstructural Fractography	44
<b>6. CONCLUSION AND RECOMMENDATIONS</b>	<b>46</b>
6.1 Conclusion	46
6.2 Recommendations	47
<b>REFERENCES</b>	<b>48</b>
<b>APPENDICES</b>	
A The Properties of Silicon Carbide	
B The Properties of Graphite	
C The Properties of Mullite	
D The Properties of Cordierite	
E Alumina phase diagram	

## LIST OF TABLES

2.1	Influence Sintering Period on Porosity, Bulk Density and Flexural Strength of Sintered SiC Porous Ceramic	15
3.1	The Composition of the Mixture	17
3.2	The Temperature, Sintering Period and Sintering Environment of the Samples	21
4.1	Temperature Consistency	30
4.2	Weight loss Percentage	31
4.3	Dry Weight, Suspended Weight, Saturated Weight and Exterior Volume	33
4.4	Percentage of Apparent Porosity	33
4.5	Percentage Water Absorption	35
4.6	Apparent Specific Gravity	36
4.7	Bulk Density	37
4.8	Flexural Strength Data	39

## LIST OF FIGURES

2.1	The Structure of Aluminum Oxide	5
2.2	The structure of Silicon Carbide	6
2.3	The Structure of Graphite	8
2.4	XRD Pattern for Sintering at 1300 °C with 1 hour Sintering Period	14
2.5	XRD Pattern for Sintering at 1300 °C with 3 hours Sintering Period	14
2.6	XRD Pattern for Sintering at 1300 °C with 5 hours Sintering Period	15
3.1	Flow Chart of Sample Preparation	16
3.2	Mini Ball Mill	18
3.3	Uniaxial Hydraulic Press Machine	19
3.4	Furnace for Sintering Process	20
3.5	Graph of Sintering Process	22
3.6	Referthermo	22
3.7	The Dimensioning Sample	23
3.8	The Flow Chart of Testing Process	24
3.9	Three Point Bending Test	25
3.10	Universal Testing Machine	26
3.11	X-Ray Diffraction (XRD)	28
3.12	Scanning Electron Microscope (SEM)	29
4.1	Graph of Temperature Consistency	30
4.2	Graph of Weight Loss Percentage	32
4.3	Graph of Apparent Porosity Percentage	34
4.4	Graph of Water Absorption Percentage	35
4.5	Graph of Specific Gravity versus Sintering Period	36
4.6	Bulk Density versus Sintering Period	38
4.7	Flexural Strength versus Sintering Period	40
4.8	XRD Patterns	41

5.1	SEM micrograph of mullite bonded silicon carbide sinter for 12 hours at temperature 1450 °C	44
5.2	The Mullite bonded silicon carbide sintered at 1450 C with 9 hour sintering period	45

# LIST OF ABBREVIATIONS, SYMBOLS, SPECIALIZED NOMENCLATURE

°C	–	Degree Celsius
μ	–	Micron
A %	–	Percentage of Water Absorption
Al <sub>2</sub> O <sub>3</sub>	–	Aluminum Oxide
ASTM	–	American Standard of Testing Materials
B	–	Bulk Density
C	–	Crystobalite
CO	–	Carbon Monoxide
CO <sub>2</sub>	–	Carbon Dioxide
D	–	Dry Weight
g	–	Gram
H	–	Hour
Min	–	Minute
Mm	–	Millimeter
PVA	–	Polyvinyl alcohol
P %	–	Percentage of Apparent Porosity
S	–	Suspended Weight
SEM	–	Scanning Electron Microscope
SiC	–	Silicon Carbide
SiO <sub>2</sub>	–	Silicon Dioxide
T	–	Apparent Specific Gravity
UTM	–	Universal Testing Machine
V	–	Exterior Volume
W	–	Saturated Weight
XRD	–	X-Ray Diffraction
α	–	Alpha aluminum Oxide
β	–	Beta

# CHAPTER 1

## INTRODUCTION

### 1.1 Background

Ceramics are inorganic, nonmetallic materials and are typically crystalline in nature. Ceramics are compounded with metallic and non-metallic elements, 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 ceramics is one of the potential materials to be used as filters, thermal expansion coefficient, high thermal conductivity and excellent mechanical property compared with alumina.

The fabrication process of porous ceramics involves precision mixing, forming, firing and machining. The effect of charge composition, characteristic of the starting silicon carbide powder and pore-forming agent on the formation of highly porous SiC-based will be studied. In order to realize the low-temperature fabrication of porous SiC ceramics, secondary phases may be added to bond SiC particles using reaction bonding process. In this study, we will explore the in situ mullite bonding on the SiC particles. It is believed that mullite possesses better high temperature stability and oxidation resistance, matching thermal expansion and good chemical compatibility between mullite and SiC. The graphite will be used as pore-former as it will keep the skeleton of the green bodies intact before the bonding phase formed at higher temperature.



## **1.2 Problem Statement**

The study of the project has the potential to be a new ceramic filter with the improving of the mechanical properties. The existing ceramic filter that has been widely used is cordierite ceramic filter. Compared to cordierite ceramic filter, melting point of silicon carbide is higher than cordierite which is 2700 °C compared to only 1200 °C. Besides that, the flexural strength of mullite also higher than cordierite which is 170 MPa compared to 117 MPa. The properties of the materials can be referred to Appendix A, B and C. So, with the combination of silicon carbide and mullite bonding can improve the durability of ceramic filter for used in the critical condition.

## **1.3 Hypothesis**

From the previous study, the increasing of the sintering period, the porosity will decrease and the bulk density and the flexural strength of the mullite bonded silicon carbide will increase. So, it is expected that the mechanical properties of the mullite bonded silicon carbide will increase with the increasing of the sintering period. Moreover, the optimum sintering period for the sintering process will be obtained in order to get the optimum mechanical properties of the mullite bonded silicon carbide ceramic.

## **1.4 Objectives**

The objectives of this project are as follows:

- (a) To study the flexural strength of Mullite-bonded Silicone Carbide sintered at various heating period.
- (b) To identify the optimum heating period of the prepared Mullite-bonded Silicone Carbide.

- (c) To observe and analyze the mullite structure in mullite bonded silicon carbide porous ceramic.

## **1.5 Scope of Study**

For this project, the scope of the study will focus for the flexural strength of the mullite bonded silicon carbide and also the physical properties which are apparent porosity, water absorption, apparent specific gravity, bulk density and the microstructure observation will be tested. The other properties of the mullite bonded silicon carbide such as thermal properties, electrical properties and other properties will not cover in this study. Moreover, the study also will focus on effect of sintering period to the mechanical properties of mullite bonded silicon carbide only and not other parameters such as the effect of temperature, grinding time, pore size and also composition of the mixture.

## **CHAPTER 2**

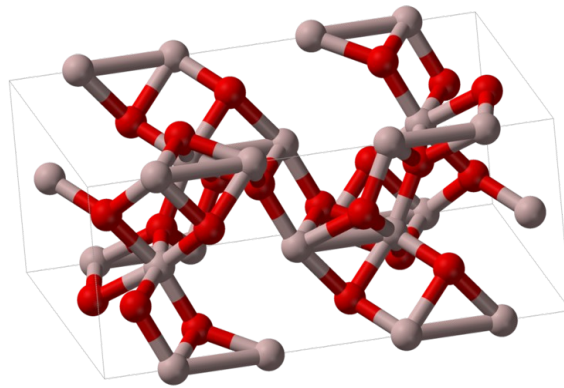
### **LITERATURE REVIEW**

#### **2.1 Overview of Raw Material**

For this project, there are three main raw materials to be used in the investigation of the mechanical properties of mullite bonded silicon carbide. The materials are aluminum oxide also known as alumina, silicon carbide and graphite. Here also will state the structure, the properties and also the application of these materials.

##### **2.1.1 Aluminum Oxide**

Aluminum oxide also known as alumina is an amphoteric oxide of aluminum with the chemical formula  $Al_2O_3$ . The structure of the alumina is given in Figure 2.1. The alumina is an important constituent abrasives, refractories, ceramics, and electrical insulation. Alumina is the most cost effective and widely used material in the family of engineering ceramics (Accuratus, 2002a). The raw materials from which this high performance technical grade ceramic is made are readily available and reasonably priced, resulting in good value for the cost in fabricated alumina shapes. With an excellent combination of properties and an attractive price, it is no surprise that fine grain technical grade alumina has a very wide range of applications.



**Figure 2.1: The Structure of Aluminum Oxide**

### **2.1.1.1 Properties of Aluminum Oxide**

Aluminum oxide,  $\text{Al}_2\text{O}_3$ , chemical compound with melting point about  $2000^\circ\text{C}$  and specific gravity about 4.0. It is insoluble in water and organic liquids and very slightly soluble in strong acids and alkalis. Alumina occurs in two crystalline forms. Alpha alumina is composed of colorless hexagonal crystals and gamma alumina is composed of minute colorless cubic crystals with specific gravity about 3.6 that are transformed to the alpha form at high temperatures. Aluminum oxide is a good thermal insulator and electrical insulator. In its most commonly occurring crystalline form, called corundum or  $\alpha$ -aluminum oxide, its hardness makes it suitable for use as an abrasive and as a component in cutting tool. Aluminum oxide is responsible for metallic aluminum's resistance to weathering. A number of alloys, such as aluminum bronzes, exploit this property by including a proportion of aluminum in the alloy to enhance corrosion resistance.

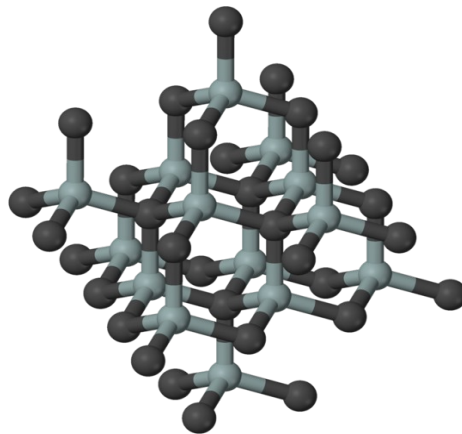
### **2.1.1.2 Application of Aluminum Oxide**

Major uses for aluminum oxide include the manufacture of water treatment chemicals such as aluminum sulphate, poly aluminum chloride and sodium aluminate. Aluminum oxide also used in coating Titania pigments and as a fire

retardants or smoke suppressant. The major uses of aluminum oxides are in refractories, ceramics, polishing and abrasive applications. Minor uses include use as a medium for chromatography. Aluminum oxide is also used in preparation of coating suspensions in compact fluorescent lamps.  $\text{Al}_2\text{O}_3$  is also used in fluoride water filters. It is one of the few methods available to filter water soluble fluorides out of water. Another used of aluminum oxide is for the laboratory instrument tubes and samples holders and also as a grinding media.

### 2.1.2 Silicon Carbide

Silicon Carbide is the only chemical compound of carbon and silicon. Silicon carbide or SiC is composed of tetrahedra of carbon and silicon atoms with strong bonds in the crystal lattice. This produces a very hard and strong material. The structure of the silicon carbide is given in Figure 2.2. It was originally produced by a high temperature electro-chemical reaction of sand and carbon. Silicon carbide is an excellent abrasive and has been produced and made into grinding wheels and other abrasive products for over one hundred years. Today the material has been developed into a high quality technical grade ceramic with very good mechanical properties.



**Figure 2.2: The structure of Silicon Carbide**

### **2.1.2.1 Properties of Silicon Carbide**

The superior properties of the silicon carbide have increased the interest of many manufactures to use it in the manufacturing field. The key properties of the silicon carbide are low density, high strength, low thermal expansion, high thermal conductivity, high hardness, excellent thermal shock resistance and superior chemical inertness. Silicon carbide is not attacked by any acids or alkalis or molten salts up to 800°C. In air, SiC forms a protective silicon oxide coating at 1200°C and is able to be used up to 1600°C. The high thermal conductivity coupled with low thermal expansion and high strength gives this material exceptional thermal shock resistant qualities. Silicon carbide ceramics with little or no grain boundary impurities maintain their strength to very high temperatures, approaching 1600°C with no strength loss (Accuratus, 2002b). Details of the properties of silicon carbide are given in Appendix A.

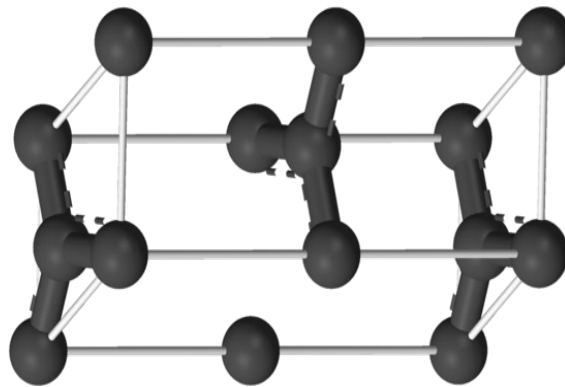
### **2.1.2.2 Application of Silicon Carbide**

The properties of the silicon carbide have made this material used for the numerous high performance applications. Today the material has been developed into a high quality technical grade ceramic with very good mechanical properties. It is also used in abrasives and refractories. The material can also be made an electrical conductor and has applications in resistance heating, flame igniters and electronic components. Structural and wear applications are constantly developing.

### **2.1.3 Graphite**

The material is generally grayish-black, opaque and has a radiant black shine. Graphite also is a crystalline form of carbon having a layered structure with basal planes or sheets of close-packed carbon atoms (Kalpakjian & Schmid, 2000). The structure of the graphite is given in Figure 2.3. It is unique in that it has properties of

both a metal and a non-metal. It is flexible but not elastic, has a high thermal and electrical conductivity, and is highly refractory and chemically inert. Graphite has a low adsorption of X-rays and neutrons making it a particularly useful material in nuclear applications. The unusual combination of properties is due its crystal structure. The carbon atoms are arranged hexagonally in a planar condensed ring system. The layers are stacked parallel to each other. The atoms within the rings are bonded covalently, whilst the layers are loosely bonded together by van der Waals forces. The high degree of anisotropy in graphite results from the two types of bonding acting in different crystallographic directions.



**Figure 2.3: The Structure of Graphite**

### **2.1.3.1 Properties of Graphite**

Consequently, graphite is weak when sheared along the layers. This characteristic gives graphite its low frictional properties of solid lubricant. However, its frictional properties are low only in an environment of air or moisture but in vacuum graphite is abrasive and is poor lubricant. Unlike other materials, strength and stiffness in graphite is increase with temperature. Although brittle, graphite has high electrical conductivity and good resistance to thermal shock and to high temperature although it is begins to oxidize at 500 °C. Details of the properties of graphite are given in Appendix B.

### **2.1.3.2 Application of Graphite**

Graphite is widely used in various industries such as chemical industries, nuclear industries, electrical applications and mechanical applications. There are many high temperature uses for graphite in the chemical industry such as in the production of phosphorus and calcium carbide in arc furnaces. Graphite is used as anodes in some aqueous electrolytic processes such as in the production of halogens (chlorine and fluorine.) High purity electro graphite is used in large amounts for the production of moderator rods and reflector components in nuclear reactors. Their suitability arises from their low absorption of neutrons, high thermal conductivity and their high strength at temperature. The main application for graphite as an electrical material is in the manufacture of carbon brushes in electric motors. In this application the performance and lifetime of the component is very dependent on grade and structure. Graphite is used widely as an engineering material over a variety of applications. Applications include piston rings, thrust bearings, journal bearings and vanes. Carbon based seals are used in the shafts and fuel pumps of many aircraft jet engines.

## **2.2 Mechanical Properties of Ceramics**

Ceramic materials are somewhat limited in applicability by their mechanical properties, which in many respects are poorer to those of metals. The principal drawback is a disposition to catastrophic fracture in a brittle manner with very little energy adsorption. For the mechanical properties of the ceramic, the flexural strength and the effect of the porosity on the mechanical properties of ceramic will be discussed.

### **2.2.1 Flexural Strength**

The stress-strain behaviour of brittle ceramic is not usually ascertained by a tensile test because of three base reasons: