FABRICATION OF DENSE ALUMINA THROUGH SLIP CASTING PROCESS

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UNIVERSITI TEKNIKAL MALAYSIA MELAKA

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

by

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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 member of the supervisory committee is as follow:

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DECLARATION

I hereby, declared this report entitled "Fabrication of Dense Alumina Through Slip Casting Process" is the results of my own research except as cited in references.

Signature	:	....
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ABSTRACT

Dense alumina products were prepared through slip casting method. Slips are prepared at different alumina and ball clay content. The slips were prepared by mixing the powders with water as solvent and dispersant at different composition of alumina and ball clay content in each batch. Three different body formulated were prepared named as batch A, B and C. The process sequence for ceramic forming by using slip casting are started by setting the composition, followed by mixing the starting raw material as well as slip preparation through roller milling to obtain the homogenous slip and casting by using the plaster of Paris mold, drying of slip cast, into bisque formation and lastly the specimen was sintered. Mechanical properties and microstructures of dense alumina product were evaluated, analysis methods used in this project research are measure degree of shrinkage, Scanning Electron Microscopy (SEM) for microstructural observation, X-Ray Diffraction (XRD) for phase observation, three point bending test for flexural strength measurement of the specimen. All the specimen from batch A with the 0.1% ball clay addition have fracture during the drying stages. Batch C have higher shrinkage and higher flexural strength compared other two batches. From the microstructural image less pores for batch C specimen are reveal.

ABSTRAK

Spesimen alumina terpadat dihasilkan menggunakan teknik pengacuanan tuangan.. Bancuhan disediakan dengan menngunakan tiga campuran alumina dan tanah liat yang berbeza. Bancuhan juga telah dicampur dengan air dan bahan serak. Terdapat tiga bancuhan iaitu bancuhan A, B, dan C. Urutan proses menggunakan teknik pengacuan tuangan adalah seperti berikut; komposisi bahan dicampur, bancuhan disediakn menggunakan 'roller milling'untuk mendapatkan bancuhan yang sebati, bancuhan dituangan ke dalam acuan, spesimen yang terbentuk dikeringkan dan akhir sekali specimen dibakar pada suhu tinggi ditetapkan. Sifat-sifat mekanikal dan mikrostruktur alumina terpadat dinilai. Sifat mekanikal dan mikrostruktur dinilai, kaedah yang digunakan untuk menganalisis adalah dengan mengukur darjah pengecutan, Mikroskop Imbasan Elektron iaitu untuk pemerhatian pada mikrostruktur, Pembelauan X-Ray iaitu untuk pemerhatian pada fasa, ujian pelenturan tiga titik untuk mengukur kekuatan pelenturan. Didapati semua spesimen daripada bancuhan A telah patah pada bahagian tengah pada peringkat pengeringan dimana bancuhan ini mengandungi 99.9% alumina dan 0.1% tanah liat. Bancuhan C mempunyai pegecutan dan kekuatan pelenturan yang paling tinggi berbanding dengan dua bancuhan yang lain. Daripada gambaran mikrostruktur spesimen C, ia menunjukkan kurang lubang.

DEDICATION

For my beloved family and friends.

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Al2O3	-	Alumina
MgO	-	Magnesium Oxide
NH_4^+	-	Ammonia
NH_4^+-PMA	-	Salt of poly (methacrylic acid)
SiC	-	Silicon carbides
Si_3N_4	-	Silicon nitride
CaSO4.2H2O	-	Gypsum
˚C	-	Degree Celsius
%	-	Percent
Wt%	-	Weight percent
h	-	Hour
m	-	Meter
MPa	-	Mega Pascal
µm	-	Micrometer
SEM	-	Scanning electron microscopy
XRD	-	X-ray diffraction
ASTM	-	American Standard Testing Material

CHAPTER 1

INTRODUCTION

1.1 Introduction to Ceramic

William (2003) state that the term 'ceramic' comes from the Greek word *keramikos* which means 'burnt stuff' indicating that desirable properties of these materials are normally achieve through a high temperature heat treatment process called firing. Chemically ceramics are nonmetallic, inorganic compounds which are formed by the action of heat.

Ceramic generally are characterized by high hardness, high compressive strength, corrosion-resistant, low thermal expansion, high temperature resistant, good chemical inertness, low density, and low thermal and electrical conductivity. On the other hand, they are brittle and have low toughness and ductility. The properties possessed by most ceramics due to differences of bonding. Most of ceramic materials have a mixed bonding structure with various ratios between ionic and covalent character. The percentage of ionic bonds can be estimated by using electronegativity determinations.

1.2 Type of Ceramic

Ceramics are classified in many ways. It is due to different in composition, properties and its applications. Based on their composition, ceramics can be classified into Oxides, Carbides, Nitrides, Sulfides, Fluorides and etc. Based on their specific applications, ceramics are classified as glasses, clay products, refractories, abrasives, cements, and advanced ceramics for special applications. According to Kalpakjian (2006) ceramic classification is based on their engineering applications, ceramics are classified into two groups as: traditional and advanced ceramics.

1.2.1 Traditional Ceramic

Traditional ceramics are most made-up of clay, silica and feldspar. Traditional ceramics include objects made of clay and cements that have been hardened by heating at high temperatures. Traditional ceramic products usually made in large quantities by efficient and inexpensive manufacturing methods. The examples of traditional ceramic are such as dinnerware, sanitary ware, pottery, tiles and refractory. Traditional ceramics are most made up of clay, feldspar and silica.

Clay has been used in a wide range of ceramics product for thousands of years and continues to be a major component in most ceramic bodies today. Clay is essential and predominant component in most ceramic product.

Feldspar is the most common igneous rock mineral and the most plentiful mineral in the earth's crust. Feldspar is a common raw material in the production of ceramics product. In the fabrication of ceramic material, feldspar also serves as a flux to form a glassy phase at low temperatures. It improves the strength, toughness, and durability of the ceramic body and cements the crystalline phase of other ingredients.

Silica is a major ingredient in refractories and whitewares. Silica occurs commonly in nature as sandstone, silica sand or quartzite. Silica is one of the most abundant oxide materials in the earth's crust. Silica is a precursor to the fabrication of other ceramic products and as a material on its own.

1.2.2 Advanced Ceramic

Advanced ceramic include ceramics for electrical, magnetic, electronic, and optical application (sometimes referred to as functional ceramics) and ceramics for structural applications at ambient as well as at elevated temperatures (structural ceramics). Advance ceramics include carbides, such as silicon carbide (SiC); oxides, such as aluminum oxide, (Al_2O_3) and zirconia (ZrO_2); nitrides, such as silicon nitride (Si_3N_4); silicates such as kaolinite ($Al_2Si_2O_5$ (OH) $_4$) and mullite ($Al_6Si_2O_{13}$) and many other materials, including the mixed oxide ceramics that can act as superconductors. Advanced ceramic requires modern processing techniques, and the development of these techniques has led to advances in industry.

Advanced ceramics materials possess unique properties in chemical resistance, corrosion resistance; wear resistance and dimensional stability at room temperature as well as at elevated temperatures. Ceramic matrix composites are at the forefront of advanced materials technology because of their light weight, high strength and toughness, high temperature capabilities, and graceful failure under loading. Ceramic composites are considered as enabling technology for advanced aero propulsion, space propulsion, space power, aerospace vehicles, and space structures.

1.2.3 Ceramic Based Composites

Ceramic composites are being actively developed in many research establishments primarily to improve either the toughness or hardness to be more suited to a particular application. This group can be composed of a combination of oxide ceramics and non-oxide ceramics (granular, platy, whiskers, etc.), oxide and oxide ceramics, non-oxide and non-oxide ceramics, ceramics and polymers, etc. an almost infinite number of combinations are possible. Fiber, whisker or particulate reinforced composites can be made tougher and stronger than traditional un-reinforced ceramics. Ceramic composites offer the mechanical and physical properties unavailable in the past and provide the opportunities to solve past material problems.

1.3 Application of Ceramic

The applications for ceramics have found for a long time ago. Every day new and different applications are being discovered. This truly makes ceramics have broad ranges of functions and applications. Ceramic materials display a wide range of properties which purpose in many different product areas to facilitate user.

Most people, when they hear the word of ceramics, they will think of art, dinnerware, pottery, tiles, and brick. The above mentioned products are commonly referred to as traditional ceramic that have been developed since the time of the earliest civilizations. These traditional products have been and continue to be important and still represent a major part of the ceramics industry although in recent year advanced material have been successfully developed . These advanced ceramics are being used for applications such as space shuttle tile, engine components, artificial bones and teeth, computers and other electronic components, and cutting tools (Callister, 2003).

Examples of the wide range application of ceramics can be seeing as mention in the following tables. The applications of ceramics can be simplifies into two major categories which are traditional and advanced material as in the Table 1.1 and 1.2 respectively. For the traditional ceramic application it can be divided into five major industries segment together with several examples. For the advanced ceramic it can be classified into four categories which consists structural, electrical, coatings and lastly chemical and environmental together with a few examples.

Table 1.1: The example application of the traditional ceramic

Industry Segment	Common Examples
Structural clay products	Brick, sewer pipe, roofing tile, clay floor and wall tile (i.e., quarry tile), flue linings
Whitewares	Dinnerware, floor and wall tile, sanitary ware, electrical porcelain, decorative ceramics
Refractories	Brick and monolithic products are used in iron and steel, non-ferrous metals, glass, cements, ceramics, energy conversion, petroleum, and chemicals industries
Glasses	Flat glass (windows), container glass (bottles), pressed and blown glass (dinnerware), glass fibers (home insulation), and advanced/specialty glass (optical fibers)
Abrasives	Natural (garnet, diamond, etc.) and synthetic (silicon carbide, diamond, fused alumina, etc.) abrasives are used for grinding, cutting, polishing, lapping, or pressure blasting of materials
Cements	Used to produce concrete roads, bridges, buildings, dams, and the like

Table 1.2: The example application of the advanced ceramic

Industry Segment	Common Examples
Structural	Wear parts, bioceramics, cutting tools, and engine components
Electrical	Capacitors, insulators, substrates, integrated circuit packages, piezoelectrics, magnets and superconductors
Coatings	Engine components, cutting tools, and industrial wear parts
Chemical and environmental	Filters, membranes, catalysts, and catalyst supports

1.4 Objective

The objectives of this project are listed as below:

i. To fabricate the bar specimens and crucibles of dense alumina by using slip casting technique.

ii. To analyze the effect of different raw materials composition on the final properties of dense alumina.

1.5 Scope

The project involved the theoretical study on the ceramic slip casting process and fabricates the dense alumina through the slip casting process. The emphases are on the preparation of slip with correct percentage of composition between alumina and ball clay. Water are used until slip casting obtain the desired specific gravity ranges between 1.4-1.6. For this experiment, the raw materials used were alumina and ball clay with different ratios of raw materials constituent. There are three different weight ratios of powder mixture, which are 1:1000; 5:1000 and 10:1000. The ratio refers to ball clay: alumina. The slip casting was prepared and was mixed using roll mill for three hour to make sure all the chemical composition agglomerate homogenously. The ratio of grinding media is ¼ raw materials. The slip is then casted into the plaster mold of Paris by using solid and drain casting technique for bar specimens and crucibles respectively. In this study the bar specimens will be done through the process drying at room temperature and bisque at 900°C by using the heating rate 10°C/min. Then the green body will be sintered at 1400°C. The samples dimensions were measured before and after sinter to get the percent of shrinkage of sintered specimen. The finished specimens were needed to prepare for testing and evaluations. The sintered specimens were tested by using X-ray diffraction (XRD) to identify phase composition in every sample. Scanning Electron Microstructure (SEM) is used to analyze the microstructures in each sample. Other testing for specimens includes flexural test and shrinkage.

1.6 Problem Statement

Alumina is widely known as advanced ceramic materials which are used in a variety of applications. High purity alumina (Al_2O_3 content > 99%) have high thermal conductivity, low thermal expansion, corrosion resistance and high compressive strength leads to good thermal shock resistance is ideal for high temperature applications such as crucibles. Advanced ceramic typically used non-traditional techniques such as hot pressing, vapor deposition, gel casting and injection molding to form ceramic materials into a recognizable shape or product.

However considering the importance homogeneous distribution of the particles in the production of ceramic crucibles is essential to obtain a high-density impervious product; therefore the slip casting technique was chosen to produce the crucibles. Homogeneous distribution of the particles can be obtained by using the slip casting technique although it quit conventional to the industries.

Rak (2000) stated the problem in the slip casting contains about 30 wt% water and about half of it must be absorbed by the plaster of Paris mould in order to reduce the liquid slip to an acceptably firm body at the mold surface. This forms a dense cast with long production time and the molds are easily damaged.

High purity alumina forming with slip casting technique produce parts that are fragile and cannot easily be machined in the green body state. The composition of the purity alumina ceramic body can be changed to enhance particular desirable material characteristics by adding little additive. In this project, properties of the final body ceramic alumina with the different composition of additive is then analyzed.