

# FABRICATION OF ALUMINA CRUCIBLE

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

By

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## ABSTRACT

Ceramic crucibles is fabricate from alumina material by using slip casting process. Two different batches are produced by the different amount of talc as the additive in each batch in order to determine the effect of talc to the alumina products that produced. The volume of talc that is used in batch B is higher compare to the volume that is used in batch A. The effect of the casting time to the crucible thickness was investigated by using various casting time. From the experimental that has done showing that, the longer casting produce higher crucible thickness. The shrinkage level of both batches was measured for drying process, bisques process and sintering process. The result was showing that, the shrinkage level is majorly happen during the sintering process. The mechanical properties of each batches is studied through flexural test and microstructure observation using SEM. The specimens that produced from batch B is defining have batter mechanical properties compare to specimens from batch A. The specimens are proven as alumina by analyze using XRD.

## ABSTRAK

Mangkuk pijar seramik dihasilkan daripada bahan alumina dengan menggunakan process pengacuan tuangan. Dua bancuhan yang berbeza dihasilkan dengan membezakan nilai bedak yang digunakan sebagai bahan tambahan bagi mengenal pasti kesan penggunaan talc pada produk alumina yang dihasilkan. Bancuhan B memiliki kandungan bedak yang lebih tinggi daripada bancuhan A. Kesan masa pengacuan dikaji dengan menggunakan masa pengacuan yang berbeza. Proses pengecutan yang berlaku terhadap spesimen diukur bagi proses pengeringan, proses pra- pemanasan , dan proses pemanasan melampau di bawah takat lebur. Sifat mekanikal bagi spesimen untuk setiap bancuhan di kaji dengan menjalankan ujian kekuatan menahan patah, ujian ketumpatan, dan juga menjalankan pemerhatian terhadap microstruktur bagi spesimen untuk setiap bancuhan dengan menggunakan mikroskop imbasan electron. Daripada ujikaji yang dijalankan specimen daripada bancuhan B mempunyai sifat mekanikal yang lebih baik daripada bancuhan A. Kesemua spesimen dibuktikan dibuat daripada bahan alumina dengan menggunakan belauan sinar-X.

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## LIST OF ABBREVIATIONS, SYMBOLS, SPECIALIZED NOMENCLATURE

$\text{Al}_2\text{O}_3$	-	Alumina
$\text{MgO}$	-	Magnesium Oxide
$\text{NH}_4^+$	-	Ammonia
$\text{NH}_4^+$ -PMA	-	Salt of poly(meth acrylic acid)
$\text{SiC}$	-	Silicon carbides
$\text{Si}_3\text{N}_4$	-	Silicon nitride
$\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$	-	Gypsum
$^\circ\text{C}$	-	Degree Celsius
%	-	Percent
Wt%	-	Weight percent
h	-	Hour
m	-	Meter
MPa	-	Mega Paschal
$\mu\text{m}$	-	Micrometer
$\zeta$	-	Zeta
SEM	-	Scanning electron microscopy
XRD	-	X-ray diffraction

# CHAPTER 1

## INTRODUCTION

### 1.1 Overview of Project.

A detail understanding in fabrication of ceramic product i.e. alumina crucibles through slip casting process will be achieved from the research work. Two types of casting, which are drain casting and solid casting are investigated. Talc powder was added into alumina based material. Microstructure phase analysis, flexural properties, and shrinkage properties are studied.

### 1.2 Objective of the project:

The objectives of this project are listed as below:

- To study the effect of casting time to the crucible thickness.
- To investigate the shrinkage level of the ceramic.
- To examine the effect of talc addition into alumina.

### 1.3 Scope of the project:

The scopes of the project are presented as below:

- Preparation of slip at two different compositions:
  - Batch A: 99% Al<sub>2</sub>O<sub>3</sub>, 0.1% Ball Clay, 0.1% Talc

○ Batch B: 99% Al<sub>2</sub>O<sub>3</sub>, 0.1% Ball Clay, 0.5% Talc

- Fabricate the crucible with various casting time :
  - 5 minutes casting time
  - 15 minutes casting time
  
- Measure the shrinkage percentage for both batches.
  
- The properties of both batches are determined by doing the experiment to the solid bar specimen through, Flexural Test, microstructure observation using SEM, and analyze the phase of the specimens through XRD analysis.

## **CHAPTER 2**

### **LITERATURE REVIEW**

#### **2.1 Introduction.**

Ceramic is a burn stuff that is compounds of metallic and non-metallic elements. The characteristics that owned by ceramic are high temperature stability, high hardness, brittle, high mechanical strength, low elongation under application of stress and also low thermal and electrical conductivities. Ceramic are classified in many ways. It is due to divergence in composition, properties and applications. Based to ceramic composition, they can be classified as Oxides, Carbides, Nitrides, Sulphides, Fluorides and etc. Meanwhile based on their specific applications, ceramics are classified as; Glasses, Clay products, Refractory, Abrasives, Cements, and advanced ceramic for the special application. Further more, based on ceramic engineering applications, ceramics are classified into two groups as traditional and engineering ceramics. (Kingery *et. al.* 1976)

## 2.2 Traditional Ceramics

Traditional ceramics was named for the ceramic that made from naturally occurring material such as clay minerals. Traditional ceramics application includes; white ware, heavy clay products, refractory, construction materials, abrasive products and glass. (Anonymous1, 2008)The example of traditional ceramics application is summarized in Table 2.1 below.

**Table 2.1:** Application of Traditional Ceramics (Anonymous 1, 2008)

<b>Category</b>	<b>Examples</b>
White wares	Dishes, plumbing material, enamels and tiles
Heavy clay products	Bricks, pottery, materials for the treatment and transport of sewage, water purification
Refractory	Brick, cement, crucibles, moulds
Construction	Brick, plaster, blocks, concrete, tile, glass, fibreglass

Based to (Anonymous1, 2008) clay minerals are produced from hydrated compounds of aluminium oxide and silica. Clay mineral has layered structures including Kaolinite, hallo site, pyrophyllite and montmorillonite. These elements are all formed by the weathering of igneous rocks under the influence of water, dissolved CO<sub>2</sub>, and organic acid. Anonymous 1 stated that, the main sources of clay minerals were formed when feldspar was eroded from granite and deposited in lake beds, where it becomes altered to clay.

Meanwhile, silica is a major ingredient in glass, glaze, enamels, refractory and white ware. Silica major sources include quartz. Another naturally occurring mineral is feldspar. Feldspar is used in producing glass, pottery, enamel, and other ceramics products. The other naturally occurring minerals used directly in



ceramic production include talc, asbestos, wollastonite and sillimanite. (Anonymous1, 2008)

### **2.3 Advance Ceramics**

According to (Geiger, 2001) advance ceramic is the ceramic materials that have the exhibit superior mechanical properties, corrosion or oxidation resistance as well as thermal, electrical, optical or magnetic properties. Advance ceramic were define more pure and offered more control over the composition, microstructure, and crystal structure over minerals-based ceramics compared to traditional ceramic.

The advance ceramic is also termed as engineering and technical ceramics. (Geiger, 2001) also stated that, advance ceramic are generally divided into several classes including structural ceramics, electrical and electronic ceramics, ceramic coating and the last but not least the chemical processing and environmental ceramic.

Based to the same reference, the structural ceramic is defined including the application such as industrial wear parts, cutting tools, engine component and also the bio ceramics. For the electronic application, advance ceramic has a bigger share, including capacitors, substrates, integrated circuit packages, piezoelectric, magnets and also superconductors. Meanwhile, ceramic coating application is also found in engine components, cutting tools, and industrial wear parts. (Geiger, 2001)

### **2.4 Alumina Ceramics.**

Alumina ( $\text{Al}_2\text{O}_3$ ) is one examples of advance ceramic. This material is one of the extensively utilized structural ceramics. This material is having great potential to be used in many spherical application that required low density, high hardness chemical inertness and good high temperature. Material based on alumina ( $\text{Al}_2\text{O}_3$ ) is widely used in ceramic industry. The range of alumina ( $\text{Al}_2\text{O}_3$ ) content that

used in the ceramic production is based to the final characteristic of the product that wants to produce. (Bolton, 1998)

Generally, the content of alumina that used in ceramic production is between 100% and 80%. For the example, ceramic with more than 99.7% of alumina is used for refractory purposes. Meanwhile, ceramic with 96.5% to 99.0% of Alumina content, with addition of magnesium oxide is the ideal composition for producing electrical and engineering component. Table 2.2 below shows the properties of alumina-based ceramics. (Bolton, 1998)

**Table 2.2:** Properties of alumina-based ceramics. (Bolton, 1998)

Material	Density Mg/m <sup>3</sup>	Coeff. of expansion 10 <sup>-6</sup> K <sup>-1</sup>	Elastic Modulus GPa	Short- Term Strength MPa	Max. Use Temp. °C	Resist- ivity Ωm	Dielectric Constant At 50Hz	Loss Tangent 10 <sup>-4</sup> *	Dielectric Strength kV/mm
Alumina 99%	3.7	5.9	380	**	1600	10 <sup>12</sup>	9	5	17
Alumina 95%	3.5	5.5-5.9	320	**	1400	10 <sup>12</sup>	9	5	15
Alumina 90%	34	5.0-6.0	260	**	1200	10 <sup>12</sup>	9	5	15

## 2.5 Ceramics Forming Processing

The very specific characteristic of ceramic which have high temperature stability, makes conventional fabrication routes being unsuitable for ceramic processing. Ceramic processing can be classified into traditional ceramic forming and non - traditional ceramic forming.

### 2.5.1 Slip Casting

Slip casting which is also known as drain casting is the most common ceramic casting process. A slip is a suspension of colloidal ceramics particles in an immiscible liquid (generally water) which is insoluble in each other. (Kalpakjian, 2008) Ceramic shaping process, such as slip casting requires the dispersion of ceramic powder in an aqueous solution. (Hotta *et.al*, 2003) In general, ceramic

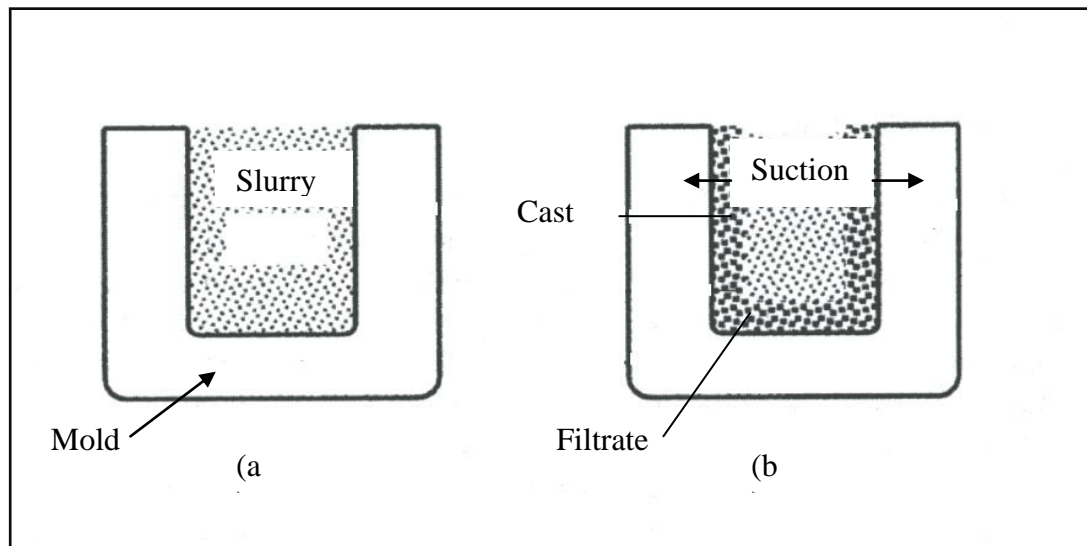
slurries must satisfy the following conditions to produce dense green bodies which including ; low viscosity, high solid content, and good dispersion. Good ceramic slurry is achieved by strong electrostatic, steric repulsion and weak attractive inter-particle van der Waals forces. (Kamiya and Yoshida, 2001)

According to (Rahman, 2007), the free flowing slurry of slip casting is poured into a permeable mold which commonly made from gypsum ( $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ ). The micro porous nature of the mold provides a capillary suction pressure, on the order of ~0.1 to 0.2 MPa, which draws the liquid from the slurry into the mold which known as filtrate. A consolidated layer of solid is forms on the walls of the mold. Figure 2.1, showing the forming of solid layer on the walls of the mold during the slip casting process .After the sufficient thickness of the cast has formed, the surplus slip is poured out and the cast are allowed to dry. This drying time will depend on the wetness of the mold and the thickness of the casting. (Burkett, 1993)

Normally the cast shrinks away from the mold during drying and can easily be removed. Once fully dried, the cast is heated to burn out any organic additives and then sintered to produce the final article. (Rahman, 2007) The examples of slip-casting composition are present in Table 2.3.

**Table 2.3:** Example of Slip-Casting Composition (Rahman, 2007)

Whiteware		Alumina	
Material	Concentration (vol. %)	Material	Concentration (vol. %)
Clay, silica, feldspar	45-50	Alumina	40-50
Water	50	Water	50-60
Sodium silicate, polyacrylate, or lignosulfate (dispersant)	<0.5	Ammonium polyacrylate (dispersant)	0.5-2
Calcium carbonate (flocculent, if required)	<0.1	Aluminum alginate or methylcellulose (binder)	0-0.5



**Figure2.1:** Schematic diagram of the slip-casting system: (a) initial system (b) after the formation of the thin cast.

In slip casting process, the slip should be poured slowly and smoothly into the cavity of the mould along the edge to remove all air from the cavity and prevent at the same time the formation of air pockets or bubbles in the clay. During pouring, the stream of slip should be moved around the mould, if the slip strikes only in one place it will cause the cast to pack at that place. This will result in warping of the casting. (Anonymous 2, 2008)

Excess slip need to be drained slowly and evenly from the mould after cast has formed to the proper thickness. If the slip is drained quickly, the suction of the outgoing slip may pull the soft clay away from contact with the plaster wall in the cavity, resulting in a warped casting. (Anonymous 2, 2008)

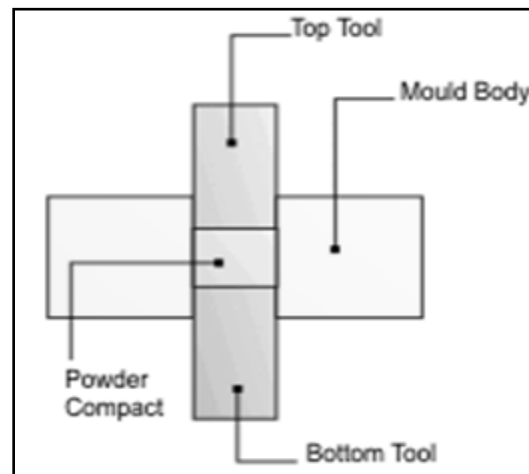
A small amount of slip is permitted to overflow on the shoulders of the mould when first pouring slip in the cavity. This is done, so that the cast will be held in the cavity, and will not drop out when the mould is first turned upside down. This overflowing permits an even trimming of the top of the casting and insures that the top will be exactly level. This overflowing on the shoulders of the mould is done only with flood moulds, and does not apply to two piece or multi-piece moulds. (Anonymous 2, 2008)

Each time a casting is made, the plaster of the mould absorbs water from the slip. It is necessary that this moisture dry out of the mould before using the mould again. "Leather hard" is the condition of the clay when the casting is ready to be separated from the mould. The clay in this condition is moist and somewhat plastic, but able to support itself without collapsing. It is a little less plastic than wedged plastic clay but plastic clay items or ornaments can be slip pasted to the leather hard casting with assurance. Anonymous 2 (2008)

### 2.5.2 Press Process

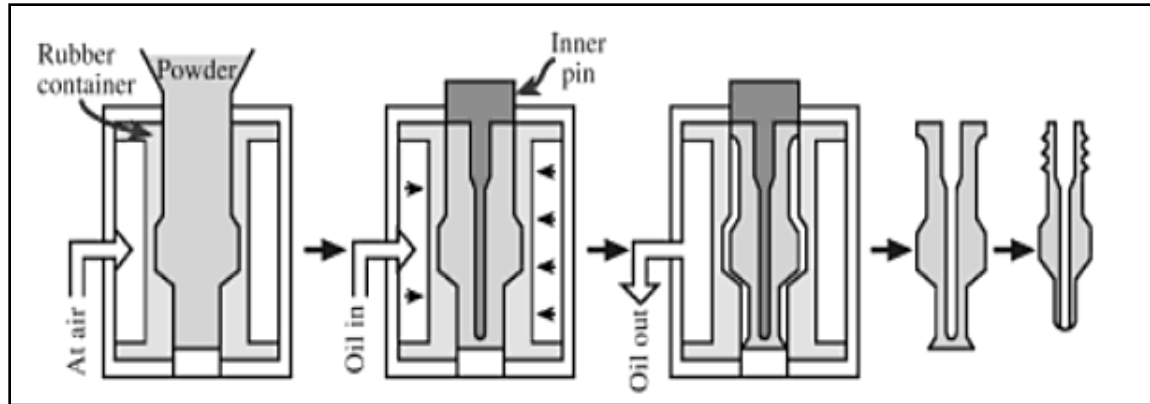
Pressing process is one example of non traditional ceramic process. The pressing process can be divided to dry and semidry pressing. For dry powder, the water that typically contain in the powder is less then 2% water .Meanwhile, for semidry powder is containing 5 to 20 % of water. Dry powder is typically use for uniaxial and isostatic pressing process. (Rahman, 2007)

Uniaxial pressing is as shown in figure 2.2, is one of the pressing processes that widely used operation in ceramic industry. For uniaxial pressing, the ceramic powder is compacted in a metal die by pressure is applied in a single direction. The formed piece takes on the configuration of die and platens through which the pressure is applied. (William D., 2003)



**Figure 2.2:** Schematic of the Uniaxial Die pressing process for ceramic powders

Even though, this process can be form with high flow rate and inexpensive process, this process is provided less packing density of the green body. With that reason, the uniaxial pressing is only suitable for forming simple shape product. Comparing with uniaxial pressing, isostatic pressing as shown in Figure 2.3 ,is define produce more homogenous pack density of green body. This forming process is suitable to produce more complex shape of green body. (Rahman, 2007)



**Figure 2.3:** Schematic of Isostatic Pressure

In preparing the raw material for pressing process, one of the important things is by controlling the flow behaviour of feed material in order to achieved efficient die filling, fast pressing rates and reproducible green body. Based to Rahman, fine powder do not flow very well and are difficult to compact homogeneously, so that spray dried is typically applied to the slurry to granulate them(Rahman, 2007)

So that, the initial step of the pressing process is beginning with forming of the slurry as the preparation for the slip casting. In preparing the slip the powder reduction of the grain size is also achieved by applying the mixing and grinding process. For the example is the ball milling process. (Mori *et. al.*, 2004) In their research they have investigate the ball mill simulation in wet mill process as the preparation of the pressing process.

In comparing the effectiveness of isostatic pressing and slip casting as the parior to Cold Isostatic Pressing (Mouzone *et. al.*, 2008) have overcome with a research. As the result, they were defined that, slip casting is able to produce more homogeneous in term of translucency and microstructure through out the sample body compare to isostatic pressing. Therefore, slip casting as a first consolidation stage prior to CIP appears to be more suitable than uniaxial pressing in order to prepare homogeneous optical ceramics.

The size of the granules is also important to be concern in order to achieving high density of green body. As was investigate by (Ching and Shun, 2008) they were defined that finer granules size can improve the density of the green body and also reducing the pressure time that needed for cold isostatic pressing.

## 2.6 Ceramic Sintering

Sintering process is the process where the green body is heated to produce the desired microstructure. The changes occurring during the stage may be fairly complex, depending on the complexity of the starting material. Two terms will be encountered for the heating stage including firing and sintering. Generally the term of *firing* is often used when the processes occurring during the heating stage are fairly complex, as in production of traditional ceramics. In less complex cases, the term *sintering* is often used. The simplest case is that for a pure single-phase material (e.g.,  $\text{Al}_2\text{O}_3$ ). (Rahman ,2007)

The system is heated to a temperature that is in the range of 0.5 to 0.75 of the melting temperature. (For  $\text{Al}_2\text{O}_3$  with the melting temperature of  $2073^\circ\text{C}$ , the sintering temperature is commonly  $1400^\circ\text{C}$  to  $1650^\circ\text{C}$ .) The powder does not melt; instead, the joining together of the particles and the reduction in the porosity or in other word can say as the densificationof the body, as required in the fabrication process, which occurs by atomic diffusion in the solid state. This type of sintering is usually referred to as *solid-state sintering*. Whereas solid-state sintering is the simplest case of sintering, the processes occurring and their interaction can be fairly complex. (Rahman,2007)

The driving forces for sintering are the reduction in surface free energy of the consolidated mass of particles. This reduction in energy can be accomplished by atom diffusion that lead to either densification of the body (by transport matter from inside the grain into the pores) or coarsening of the microstructure (by rearrangement of matter between different parts of the pores surfaces without actually leading to a decrease in the pore volume). The diffusion paths for