



## **FABRICATION OF ALUMINA-COPPER COMPOSITES BY USING RESISTIVE SINTERING METHOD**

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

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Date : 18 DECEMBER 2019

## **APPROVAL**

This report is submitted to the Faculty of Manufacturing Engineering of Universiti Teknikal Malaysia Melaka as a partial fulfilment of the requirement for Degree of Manufacturing Engineering (Hons). The member of the supervisory committee is as follow:

.....

**(Professor Madya Ir. Dr. Mohd Hadzley bin Abu Bakar)**

## ABSTRAK

Matlamat pengajian ini adalah untuk menilai sifat/ciri-ciri mekanikal dan fizikal komposisi Alumina-Kuprum sebagai pelbagai aplikasi yang boleh digunakan dalam industri. Bahan seramik mempunyai beberapa ciri-ciri yang memenuhi keperluan dalam industry secara meluas terutama dalam aplikasi mata alat. Walau bagaimanapun, terdapat beberapa kelemahan dikenal pasti yang menghad pengunaan bahan ini secara lebih meluas. Beberapa sampel dengan berbeza nisbah komposisi telah dihasilkan dengan alumina sebagai bahan asas. Dengan campuran nisbah kuprum, beberapa ujikaji akan dijalankan untuk menyiasat kesan perubahan nisbah terhadap ciri-ciri mekanikal dan fizikal komposisi tersebut. Bahagian paling kritikal adalah menghasilkan sampel asas menggunakan bahan dalam bentuk serbuk kerana ia memerlukan ketepatan dari segi aspek saiz partikel, berat, jenis dan gred. Setelah sampel asas siap, sampel ini akan melalui beberapa proses, seperti pra-pemadatan, tekanan isostatik sejuk dan yang terakhir adalah pendekatan baharu dalam rawatan haba iaitu rintangan rawatan haba atau nama komersialnya adalah pengaktifan elektrik rawatan haba. Terdapat beberapa perkara yang membezakan cara ini dengan cara tradisional/normal terutama dari segi mekanisma. Oleh itu, kajian ini adalah untuk mengetahui peratus kemungkinan berjaya dengan menggunakan tekanan dan elektrik mampu merawat haba (sintering) komposisi alumina-kuprum. Seperti yang kita tahu, kuprum bagus dalam pengaliran elektrik dan proses rawatan haba mempunyai peratus keberjayaan yang tinggi, namun adakah aplikasi baharu ini mampu mengurangkan masa proses dan penggunaan tenaga berbanding cara yang normal dimana masa diperlukan sangat lama dan penggunaan tenaga yang tinggi?

## ABSTRACT

The aim of this study is to evaluate the mechanical properties and physical properties of the compositions Alumina-Copper for various application in industries. The ceramic material have a few characteristics that made it widely used in the industries especially in the application of cutting tool. However, a few weaknesses have been detected which limits this application to go further. A few samples will be made with different compositions with alumina as the based ceramic materials. With different copper ratio, a few tests will be conduct to analyze the effect of the changes of ratio could affect the mechanical and physical properties of the composition. The crucial starts is to produce the green body of these powders as it require a lot of accuracy in terms of particle size, weight, types and grades. Once the green body is done, these samples will undergoes a few process such as pre-compaction, cold isostatic press and finally a new approach on sintering method which is resistive sintering or the commercial names called ECAS (Electrical activation sintering). There are a few things that differentiate ECAS with normal sintering especially the mechanisms. Thus, this study is to evaluate how the possibilities of success by applied current and pressure could sinter the compositions of alumina-copper. As we know, copper is a good electrical conductor and the process of sinter have high percentage to success, but does this application can reduce the time and energy compare to the normal sintering which took long soaking time and high energy required?

# **DEDICATION**

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Parents, Azlan bin Hamzah and Noreha binti Mohd Yusof

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## LIST OF ABBREVIATIONS

OFHC	-	Oxygen Free High Conductivity
XRD	-	X-Ray Powder Diffraction
EDS	-	Energy-Dispersive X-ray Spectroscopy
ECAS	-	Electrical Current Activation/Assisted Sintering
IACS	-	International Annealed Copper Standard
FAST	-	Field Activated/Assisted Sintering
ODS	-	Oxide Dispersion Strengthened

# CHAPTER 1

## INTRODUCTION

This chapter will explain and summarize the whole chapter in simple form by overview the background of study, problem statement, objective and scopes. The background of study will explain theoretically and based on current issues which only related aspects that will be put into considerations to ensure the research is always in the correct path. The problem statement will explain regarding the current issue, complaints, major problem that have being experienced by the industries or another researcher. The problem statement need to be a facts based on previous researches. The objective will clarify the mission or the goal of this research until the end in order to navigate towards the related topic only. Scopes will clarify the certain limitation that will affect the result of this research.

### 1.1 Background of Study

Cutting tool is one of the major factor that will affect the machining performances as well as the quality of the part produced. Maintaining the quality and minimization cost is not an easy tasks for every industrial sector as most of them need to invest more in terms of financial to make sure the quality aspects is always at the very good level which satisfied the customers.

The application of ceramic in cutting tool is widely used as it is very durable to be perform at high temperature and high speed machining. However, the limitation factor such as low thermal inferior shock resistance and low fracture toughness causes it to crack at certain points during machining. Compare to other material, ceramic cutting tool is the most dominant in the aspects of application in machining but due to the weakness, application of ceramic is stopped to a fix level. For this research, alumina ( $\text{Al}_2\text{O}_3$ ) and pure copper (OHFC) will be bond in order to improve the mechanical and physical properties suitable to be used as cutting tool. Theoretical on alumina production and alumina phase transition will be explain in chapter 2 as well as the grades of various type of pure copper.

A review of previous experiment on the bonding of alumina-copper using different methods will be a guideline for this research. The thing that difference is, a new approach on method to bond which is electrical current activation sintering/resistive sintering. The presence copper powder in the composition is to enable the flow of electrical current by using the concept of conduction and produce resistive heat. The parameter such time and temperature will be recorded to measure the production rate for sintering process as well as heating rate. For sintering characteristic, a few tests will be conduct such as hardness test, fracture toughness test for mechanical properties, and density, shrinkage, porosity for physical properties.

Analysis on the mechanical and physical properties of alumina-copper will be evaluate as well as the performance of resistive sintering method. For the process, thermal analysis will be the criteria to study the relation between time and temperature. In this experimental, oxidization factor will be neglected as the process is conduct in open space environment. The innovation of new composition will enhance the properties of the sample body for certain application.



## 1.2 Problem Statement

Even though ceramic such alumina has a few benefits that cannot be deny, but there is a few problems that limits the application of alumina. The first one is, low fracture toughness/low thermal inferior shock resistance which means the alumina cannot withstand the multiple points of sudden change in thermal gradient causes it to easily crack (E. Rocha-Rangel et.al (2010). This phenomenon due to the micro-structural of alumina has poor electrical conductivity. Thus, with great performance in cutting work piece at high temperatures and high speed machining, unfortunately, ceramic cutting tool has low tool life or high tool wear rate.

Another problem that should be focus on is, normal ceramic sintering requires high temperature and long soaking time. This means, to achieve bonding between nanostructures require high temperature and time. At the same time, the whole process normally consume numerous amount of energy especially in terms of electrical consumptions. Indirectly, the whole process could lead to waste in the aspects of costs and time.

Next, the application of electrical activation sintering (ECAS) or resistive sintering requires high electrical conductivity powders embedded in the structure, such as copper. This process cannot be done if the powders material is not a good conductor of electrical current since the concept of ECAS is by applied current and pressure through conduction contact. The heat resistive cannot occur if alumina only being process as alumina is poor electrical conductivity.

## 1.3 Objectives

- To analysis the effect of difference copper ratio in the compositions towards the properties
- To measure the capability and performance of resistive sintering method

## 1.4 Scopes of Study

In this research, the main purpose is to produce a composition of alumina-copper by using resistive sintering process. The research have three scopes that importance to put into considerations which is sintering characteristics and properties (e.g. density, hardness, shrinkage, etc.), and the evaluation of resistive sintering method in terms of capability and performance. The sintering characteristics and properties is defined based on the microstructure of the alumina-copper. A few tests will be conducts towards the alumina-copper powder compacts once the sintering process is done such as, hardness test, calculations of density and shrinkage. The after sinter samples will be evaluate based on hardness and comparison will be made towards the applied method and normal method. The approach of using resistive sintering method theoretically will reduce time and energy consumption, but in this experimental, the machine used is a pre-mature product came out from the conception idea as the availability of this method still need a lot of research in terms of parameter, design and material. Thus, there will be a few limitation that should be consider during the process of conducting the sintering towards the sample.

## **CHAPTER 2**

### **LITERATURE REVIEW**

#### **2.1 Introduction**

This chapter will discuss literature of alumina-copper sintering in the production of cutting tool, ratio composition of both material, type of sintering that will be used and the sintering characteristic. The effect of tool geometry and design towards machining performance will be reviewed. An overview of the sintering process will cover the theory of sintering, type of sintering, parameters, and the factors of consideration. A short review on the sintering process could lead to waste of electrical consumptions. Besides, this chapter will review the mechanical and physical properties of alumina-copper composition in terms of microstructure, hardness and density. A summary of phase transformations from transition alumina will affect the machining performance.

#### **2.2 Ceramic Material**

Ceramic material properties is famous as low density, high strength at high temperature, low thermal conductivity, low electrical conductivity, wear resistance, and corrosion resistance. However, the brittle nature of ceramic made it low fracture toughness which due to low thermal inferior shock resistance. In other words, ceramic material tends to crack or break when facing multiple point of thermal gradient at sudden changes. At room temperature, ceramic material has a low tensile strength and large scatter of strength. The best alternatives to improve ceramic properties is by added another material inside the composition. Below shows the table of mechanical properties of various type of ceramic.

<b>Material</b>	<b>Density (g/cm<sup>3</sup>)</b>	<b>Compressive Strength (ksi)</b>	<b>Tensile Strength (ksi)</b>	<b>Fracture Toughness (ksi √in)</b>	<b>Modulus of Elasticity (10<sup>6</sup> psi)</b>
Alumina	3.98	400	30	5	56
Siolan	3.25	500	60	9	45
SiC	3.1	560	25	4	60
Si <sub>3</sub> N <sub>4</sub>	3.2	500	80	5	45
ZrO <sub>2</sub> (partially stabilized)	5.8	270	65	10	30
ZrO <sub>2</sub> (transformation toughened)	5.8	250	50	11	29

Table 2. 1 Mechanical Properties of each ceramic materials

### 2.3 Alumina Material

In ceramic classes of engineering material, alumina is the most famous to be used. The characteristic and properties made alumina being used in various applications. It have the best properties and suitability to be used as cutting tool. To benefits its properties, alumina always involved in the application of cutting tool inserts as it can withstand elevated temperature at high speed machining. Below is the table that describe the material characteristics based on the different ratio or density of alumina. (Zhou & Snyder, 1991)

Due not only to their wide range of applications but also because the mystery posed with their typical properties, numerous non-organic nanostructures of oxides, both pure and doped, have been subject to extensive research activities.(Mubayi, Chatterji, Rai, & Watal, 2012)

Al<sub>2</sub>O<sub>3</sub>, B<sub>4</sub>C, SiC, and ceramic matrix composites (CMCs) such as Al<sub>2</sub>O<sub>3</sub>/ZrO<sub>2</sub> system are the major ceramic materials used commercially in the production of ballistic armors. Several disadvantages of ceramic armors are high costs, manufacturing obstacles and limitations to predict ballistic efficiency from the material's properties.(Karandikar, Evans, Wong, Aghajanian, & Sennett, 2009)

Alumina provides the best cost-benefit ratio for advanced ceramics, with high elasticity modulus, high refractoriness, high hardness, and relatively lower quality. However, ballistic

performance of alumina is lower than SiC and B4C due to its low fracture toughness and low flexural strength. (Tressler, 2001)

### **2.3.1 Alumina based ceramic cutting tool**

The great properties of alumina-based ceramic cutting tool such as high resistance to abrasion, high heat hardness, and chemistry inertness against to environments and the work piece make it a reliable choice as a tool for cutting tools. Chemically ceramic cutting tool based on alumina are more stable than high-speed steel and carbide, hence less tendency to adhere to metal during machining and less tendency to form build-up edge (Kumar et al., 2006).

Due to its properties, alumina based ceramic cutting tool has been chosen as the alternative way for carbide tool in the machining of hardened steel and produce good surface finish with dimensional accuracy in machine steel (Senthil Kumar et al.,2003).

### **2.3.2 Thermal conductivity of porous ceramic**

The thermal conductivity of solid phases at atmospheric pressure is always higher than that of gases. Thermal conductivity is still higher than that of air (at room temperature and normal pressure) by two orders of magnitude, even for yttria-doped zirconia, which is one of the strongest phases of insulating oxide. Dense solids are typically not very efficient thermal insulators for this purpose. On the other hand, the estimation of the thermal conductivity of porous materials is complicated by the fact that the lower micro-mechanical boundaries reach zero when it is possible to ignore the conductivity of the porous phase. Any forecast for porous materials must therefore be based primarily on the model.(Gregorová, Pabst, Sofer, Jankovský, & Matějček, 2012)

### **2.3.3 Alumina production-Bayer process**

Refining bauxite is quite popular in the industries of refining natural minerals, however most of us does not notice that by refining bauxite and proceed to a process called Bayer process, we can obtain alumina and precursor to aluminum. This action is done by extract the mix substances from the bauxite and smelting the grades of ( $\text{Al}_2\text{O}_3$ ). Grading of alumina can be done through annealing process which at difference temperature, the characteristics of alumina experiencing drastic changes. The Bayer process is first patented by Karl Josef Bayer which 110 years ago. Through the worldwide, Australia has the biggest sector of refining bauxite which conquer 30% of world production. (HIND, BHARGAVA, & GROCOTT, 1999).

#### **2.3.3.1 Process sequence of constructing alumina ( $\text{Al}_2\text{O}_3$ )**

The process started by crushing the bauxite into small particle and involves digestion process by using concentrated sodium hydroxide solution at temperatures up to  $270^\circ\text{C}$ . At these state, the variety substances inside the ore starts to dissolved and leave an insoluble residue. Aluminium trihydroxide is precipitated once the solid separation is done. The gibbsite will be removed and washed to calcination, which then the gibbsite is converted to alumina. Most of the extraction process is done by chemical processes either in solid/aqueous state since many of industrial in the worldwide apply physical processes as their important method. (HIND et al., 1999)

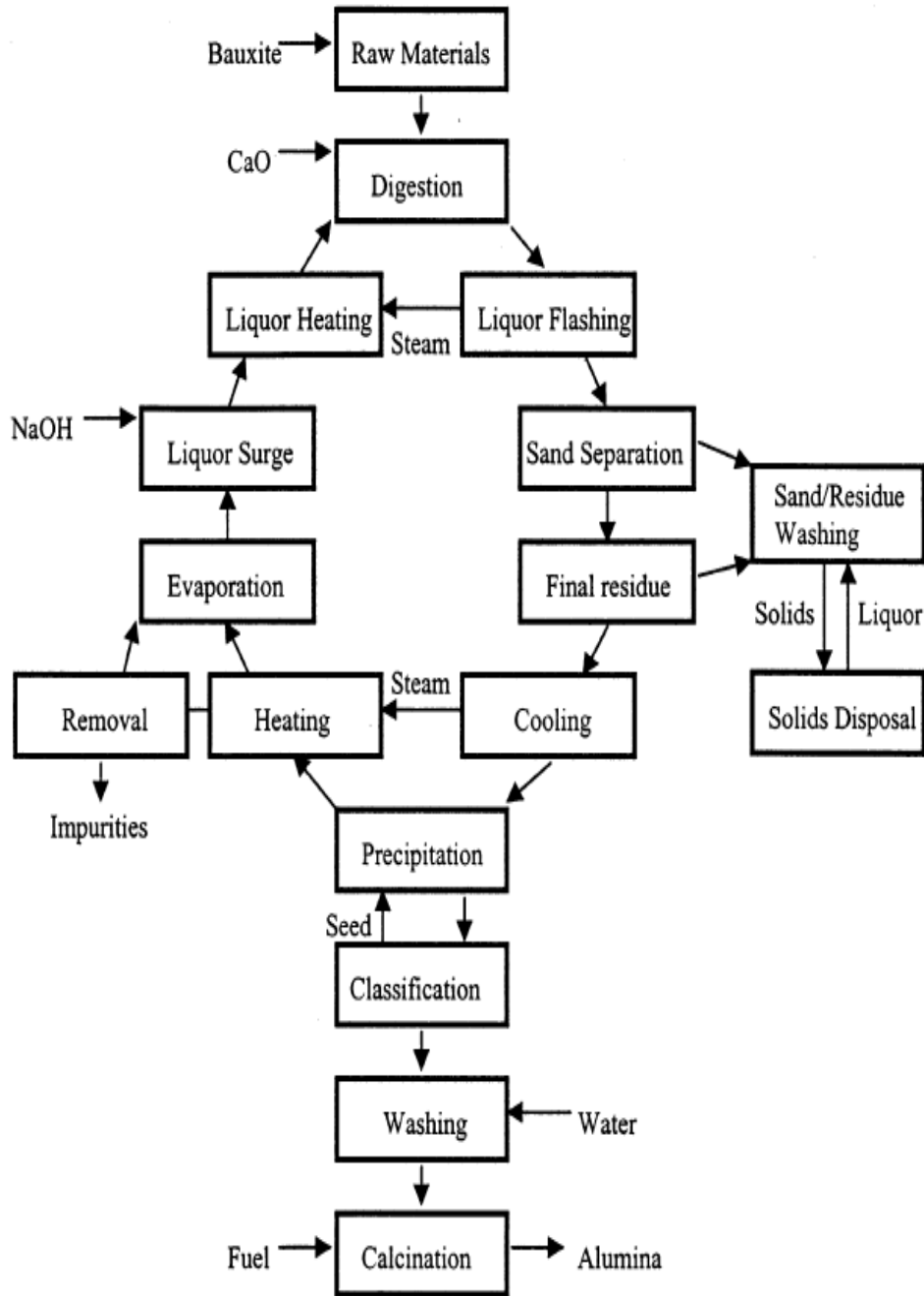
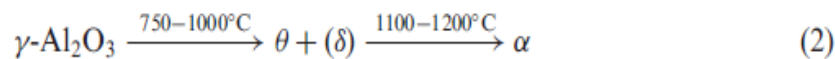
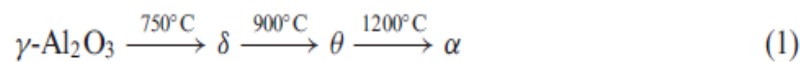


Figure 2. 1 Schematic representation of the Bayer Process depicting the cyclic nature

### 2.3.4 Alumina phase transitions

Alumina ( $\text{Al}_2\text{O}_3$ ) or mostly known as aluminum oxide has a wide variety of arrays in various application as its mechanical and physical properties fulfill the requirements of most manufacturer. It has the advantages which easy to pattern, smooth and high durability especially in the aspect of machining process. (Bodaghi, Mirhabibi, Zolfonun, Tahriri, & Karimi, 2008)

To be list, there are classes of alumina which exist in eight difference polymorphs and all of these eight phases were labeled with Greek alphabet. The most common that being used in the industries are  $\alpha$ -  $\text{Al}_2\text{O}_3$  and  $\gamma$ - $\text{Al}_2\text{O}_3$ . Among the eight difference phases alumina at  $\alpha$  phase is thermally stable while the other sevens are metastable. Thermally stable can be defined as the state where the molecular structure is stable at high temperatures and high resistance to deform. Metastable is the condition which the molecule is chemically unstable under certain conditions that would triggered that situations. (Bodaghi et al., 2008)



Equation 2. 1 Transition sequence

Research made by(Stierle et al., 2004), an experimental towards aluminium nitrate with the objective is to observe the formation of alumina during phase transition in nano-structure from  $\gamma$ -phase to  $\alpha$ -phase by using XRD (X-ray diffractometry) with single variables which is range of temperatures. The variable was decided to use three ranges of temperature which is  $400^\circ\text{C}$ ,  $800^\circ\text{C}$  and  $1000^\circ\text{C}$  and saturated aqueous solution of aluminum nitrate (gel form) undergoes annealing process in a furnace with duration of 2 hours.