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JUDUL: The Relationship Between Grain Sizes and Hardness of Sintered Alumina Product

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TAJUK: THE RELATIONSHIP BETWEEN GRAIN SIZES AND HARDNESS OF SINTERED ALUMINA PRODUCT

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....

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DECLARATION

I hereby, declared this thesis entitled "The Relationship Between Grain Sizes and Hardness of Sintered Alumina Product" is the results of my own research except as cited in references.

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APPROVAL

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

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ABSTRACT

Ceramic is a glazed or unglazed body of crystalline or partly crystalline structure, or of glass, which body is produced from essentially inorganic, non-metallic substances and either is formed from a molten mass which solidifies on cooling, or is formed and simultaneously or subsequently matured by the action of the heat. Alumina is one of the ceramic material formed of bodies with non-naturally occurring material is called new ceramic or modern ceramic. Alumina was given attention in this study of particles size and its hardness due to its most widely used as the synthetic raw materials for ceramics with excellent heat and corrosion resistance property. Thus, it has a variety of applications are being developed for machine part used in a lower temperature range, such as cutting tools, pumps and valves. Objectives of this study are to produce fine and narrow particles size distribution of alumina through planetary ball milling and, correlate between particle size and hardness of the sintered alumina, the changes in grain structure of alumina products will also be discussed. The main factor to study of particles sizes is correlated with the milling time, and the hardness of sintered alumina is corresponding to the grain size and microstructure. The seven alumina specimen with different milled time is fragmented on impart by mill ball. The milled powder is then compacted by the hydraulic pellet press and followed by sintering process. The changes in particles size and hardness of sintered alumina can be investigated via scanning electron microscope and hardness test respectively to identical the effect of grains size on hardness. As conclusion, increasing milling time resulted in a finer grain sizes and increase in hardness of sintered alumina. The Planetary ball mill cannot well-perform to fragment the particles sizes uniformly due to trapped powder between vial and ball milling. Finally, mechanical properties of milled particle size and hardness of sintered alumina will be affected the application and quality of the product.

ABSTRAK

Seramik merupakan bahan licau atau bukan licau dengan struktur kristalografi yang sepenuh dan separuh, dimana bahan ini dihasilkan daripada inorganik, bahan bukan logam atau membentuk daripada berat leburan dimana mengalami pemejawapan atau terbentuk pada masa yang sama.

Alumina adalah salah satu bahan seramik dihasilkan daripada bahan bukan semulajadi dan sebagai teknologi seramik maju atau bahan baru seramik. Alumina menitikberatkan dalam saiz partikel dan kekerasan disebabkan kegunaan yang meluas dengan sifat rintangan haba dan kakisan yang kuat daripada seramik.Dengan itu,kegunaan alumina dalam penghasilkan bahagian mesin yang memerlukan sifat suhu rendah, misalannya alat pemotong,pump dan valves amat diperlukan. Matlamat daripada projek bertujuan menghasilkan saiz dan taburan partikel yang halus dan kecil melalui bola pengisaran mesin, di mana berhubung kait dengan saiz partikel dan kekerasan sinter alumina.Akhirnya, memerhatikan perubahan alumina dalam tumbesaran butir. Faktor utama mempelajari projek ini berhubungkait dengan masa pengisaran dan kekerasan bagi sinter alumina yang kait dengan saiz butir dan mikrostruktur. Ketujuh-tujuh alumina spesimen ini mempunyai masa pengisaran yang berbeza dan dihancurkan oleh media pengisar. Selepas it, serbuk kisaran dimampatkan oleh mesin hidraulik pemampat dan diikut dengan persinteran. Perubahan dalam saiz partikel dan kekerasan sinter alumina akan dikaji dengan Mikroskop Elektron Imbasan (SEM) dan ujian kekerasan untuk mengenalpastikan kesan saiz butir partikel-kekerasan selepas activity pengisaran dan persinteran. Kesimpulannya, penambahan masa pengisaran akan mendapat saiz butir yang lebih halus dan penambahan kekerasanterhadap sinter alumina. Pengisar tidak dapat menyerata saiz butir dengan menyeluruh kerana terdapat partikel yang melekat pada media pengisar tidak dapat dikisarkan.

DEDICATION

To my beloved family

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LIST OF ABBREVIATIONS, SYMBOLS, SPECIALIZED NOMENCLATURE

α	-	Alpha
β	-	Beta
ε	-	fractional porosity
γ		Gamma/Specific surface energy
μ	-	Micro
ρ	-	Density
ρ_g	-	Green density
ρ_s	-	Density of sintered alumina
ρ_{th}		Theoretical alumina density
Δ		change
$\approx$		Approximately
$>$		More than
$<$		Less than
/	-	Or
%	-	Percent
^oC	-	Degree Celsius
A	-	total surface area of the compact
Al_2O_3		Alumina or Aluminum Oxide
$Al(OH)_3$	-	Hydrated alumina
$Al(OH)_4$	-	Sodium Aluminate
BPR	-	Ball to powder ratio
d	-	Particle size diameter
GSD		Geometric Standard deviation
h	-	Hour
ΔL	-	Change in length
$\overline{L}$	-	Mean intercept
L_L	-	The length of the line segments falling within the

		phase of interest per unit length of the test line
m		Meter
MgO	-	Magnesium Oxide
N_L	-	Number of points where the lines intercept the features of interest (grain) per unit length of test line
PDA	-	Particle distribution analysis
r_e	-	mean equivalent cylindrical pore radius
rpm	-	Rotational per minutes
RS	-	Rotational Speed
R_{sv}	-	surface-volume mean equivalent spherical particle radius
SEM	-	Scanning Electron Microscope
TiC	-	Titanium carbide
US.	-	United State
UTEM	-	Universiti Teknikal Malaysia Melaka
Vol	-	Volume

CHAPTER 1
INTRODUCTION

1.1 Introduction to Alumina Ceramic

Alumina (Al_2O_3) is the oxide of aluminum (Al) and consider as a fine ceramic. Alumina occurs in nature as the mineral bauxite, bayerite, corundum, diaspore and gibbsite. Alumina has an ionic bonding and there are numerous configurations for each combination of element. Aluminas have few type crystalline forms which are α- alumina, β- alumina and γ- alumina.

The production of alumina for ceramic is based on the Bayer process, which was for the refining aluminum. The process involved are mining, dissolution of the alumina at elevated temperature, addition of flocculants, precipitation of pure gibbsite, regeneration of the solutions for recycling and finally heating the gibbsite to 1100°C (calcinations) to give alumina.

Many different forms in which alumina manifests itself give the material a great versatility, with characteristics ranging from the hardness of sapphire to a softness similar to that of talc, from a high insolubility to ready solubility in acids or alkalies, from chemical inertness to high activity, from crystal sizes smaller than a micron to those of the single-crystal ruby gems.

The applications range from mechanical, chemical, and thermal resistance-demanding ones such as, refractory, cutting tools, abrasives, dental and bone implants, and space rockets protection to optical such as ruby lasers, or optical windows such as the radomes used to enclose the radar antenna equipment in high-speed aircrafts. Alumina used as a structural material processes properties such as having extremely high melting point (2,050°C), insoluble in water and organic liquid and very slightly soluble in strong acids and alkali, high hardness, electrical insulation and the ability to take on diverse shapes and functions.(Ichinose, 1987). The optical properties of ruby, which is α-Al2O3 with 0.5 to 2% Cr_2O_3, have been amply exploited by the jewel industry, but are also important in lasers, where rods of single-crystal ruby are used to generate the highly coherent laser light. Another important electronic application is electrical and electronic application are circuit substrates, spark plugs, magnetrons, where good insulators with low dielectric constant are desirable for the good performance of the circuits, As in the case of all phases of alumina, the number of applications is steadily growing.(Ruberto,1998).

1.2 Problem Statement

Alumina ceramics have been widely applied because of the high hardness, good wear resistance and outstanding mechanical properties. Due to its technological importance, sintering of ceramic powder compacts has been extensively investigated and many works have been developed for improving their mechanical properties. Smaller particles size and narrow size distribution have been reported by earlier researcher, Ma and Lim in their studies of effect of particle size distribution of agglomerate alumina powder compact that would promote sinterability and thus producing dense microstructure with higher hardness properties. However, the research resource of the effect of particle size of alumina on hardness of sintered alumina is scarce. Therefore, the present work attempts to study the relationship between grain size and hardness of sintered alumina and to have a comprehensive and overall understanding on the correlation between particle size, microstructure and hardness of sintered alumina. In other words, this study is to understand the effect of particle size on microstructure and the resultant microstructure on hardness of sintered alumina. Furthermore, the relationship between the hardness as a function of grain size will be determined.

Mechanical milling is known to be used to decrease the particles sizes. The planetary ball mill will be used in this study to reduce the particles size of alumina powder to some extent. Thus, the performance of the planetary ball mill in producing fine powder will be evaluated in term of microstructure and particle size distribution by using scanning electron microscope and laser particle size distribution respectively.

1.3 Objectives

The objectives of this study are to produce fine and narrow size distribution of alumina powder through planetary ball milling and to correlate between particle size and hardness of sintered alumina. The grain structures of alumina products will be compared as well.

1.4 Scope of Study

1.4.1 Selection of Milling Speed

Rotational speed (RS) of planetary ball mill is set at 300 revolutions per minutes (rpm). Rotational speed of 300rpm is selected due to some reasons. The operating rotational speed of the planetary ball mill is in the range of 40 rpm to 600 rpm. A trial run of planetary ball mill with 100, 200, 300, 400, 500, and 600 rpm was done. From the observation, the rotational speed for 100 and 200rpm is slow thus the milling process is inefficient. Then, strong vibration occurred in 400, 500 and 600 rotational speed. Dangerous environment is created to student during running time of the experiment. Furthermore, strong vibration ruins the reliability of the machine as well if the machine is operated in long period. Therefore, 300rpm is the optimum rotational speed for whole milling process.

The actual performance speed of planetary ball milled is different from the rotational speed setting because there have rotating speed for plate and rotating speed for bowl. Thus, setting of the machine controls the rotating speed of plate. The corresponding speed between plate and bowl is shown in Figure 1.1. From here, the milling speed is emphasized on the rotating speed of planetary milling plate.

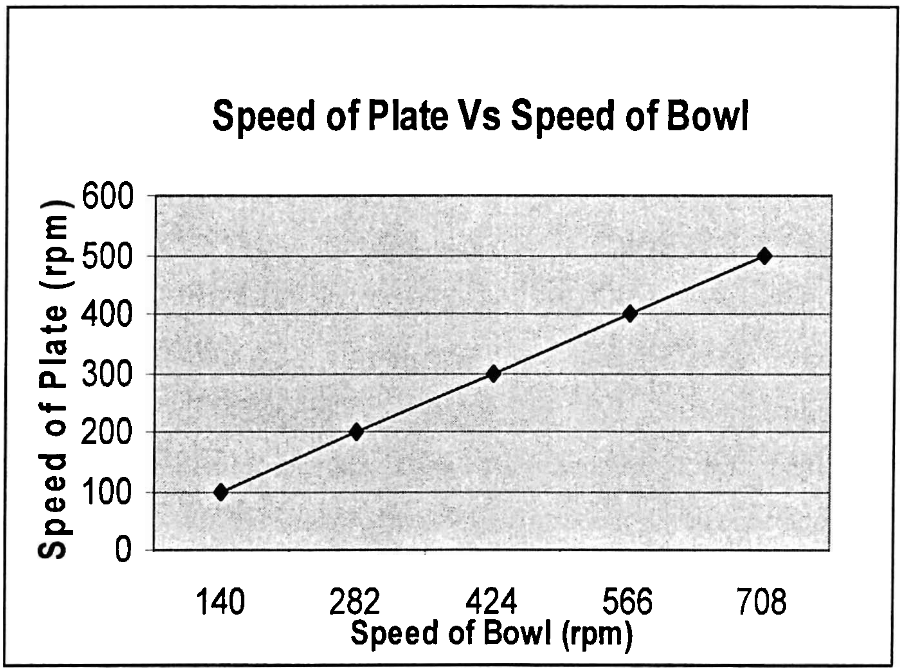

Figure 1.1: Rotating Speed of milling plate versus rotating speed of bowl

1.4.2 Selection of Milling Time

Alumina milling time is selected as 0.5, 1, 2, 3, 4, 5 and 6 hours in order to investigate the particles size distribution for each variable milling time. The selected milling time is not long since the time frame for the project is limited. Moreover, the millings activities need to coordinate with laboratory technician in order to avoid clashing with conducted class because operating noise of the machine is disturbing.

1.4.3 Sintering Profile

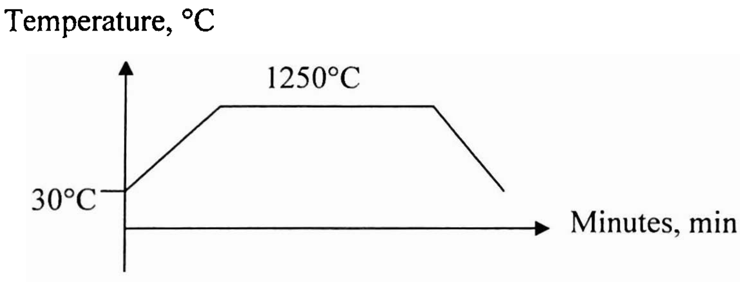

Figure 1.2: Sintering profile

The specimens consist of 2 hours, 4 hours and 6 hours milled alumina are set to 1250°C sintering temperature. These three specimens are selected for sintering process in order to investigate the different grain size and hardness after sintering process. Figure 1.2 shows the sintering profile for the sintering process. As room temperature is estimated around 30°C, then temperature is ramped up 10°C/min until 1250°C, hold for 1 hour and cooled down naturally in the furnace, finally final product is released and the properties are investigated.

1.5 Thesis Layout

The thesis is divided into five chapters. In this thesis, a review of literatures is presented in Chapter 2 which discusses about the alumina material, alumina processing, and the previous research work and theories development on the relation of grain size and hardness. In chapter 3, the flow of the methodology used in this experiment is discussed. Chapter 4 which is result and discussion and Chapter 5 are conclusion and suggestion.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

This chapter will be discussing studies from previous researchers to particular method. It consists of alumina, alumina manufacturing process, ceramic powder processing, particles size reduction, sieving, doping, compaction process, sintering process, particle size, microstructure, hardness relation, the effect of particle size and particle size distribution on sintering and microstructure and effect of grain size on hardness of sintered product.

2.2 Alumina (Al_2O_3)

Alumina, Al_2O_3 is the most widely used of synthetic raw material for ceramic. Many of the oxide ceramic have strong ionic bond. Of these, Al_2O_3 has the most stable physical properties, with the excellent heat and corrosion resistance. A loss of strength at high temperature is unavoidable, but a variety of applications are being developed for it as machine parts used in a lower temperature ranges, such as cutting tools, pumps, and valves.

Although alumina can be represented simply by the chemical formula Al_2O_3, its nature varied considerably depending upon, for instances, its crystalline form, the impurities present, and the particle diameter. Since the required physical properties also vary according to be intended use, many different type of crystalline form, such as α- alumina, β-alumina, γ-alumina and η-alumina. α-alumina composed of colourless hexagonal crystal with properties stated below. β-alumina is the compound of natrium oxide, $Na_2O.Al_2O_3$. The pure form obtained by calcination at high temperature. Pure α -Al_2O_3 has a very high melting point (2050 °C), extreme hardness (9 on the Mohs scale of hardness, which is next hardest to diamond, at 10), high electrical resistivity (1011 m at 500°C), low dielectric constant ($\varepsilon''_r \approx 10$ at room temperature), and high thermal conductivity (40 Wm−1K−1 at room temperature).The excellent of α -Al_2O_3 in all of these characteristic, together with its capacity of being efficiency joined to a metal, makes the material unequalled in a large variety of application.(Ruberto,1998) It is stable to about 1000°C and contains traces of water or hydroxyl ions. γ-Alumina composed of minute colorless hexagonal crystal with the sp.gr about 3.6 that are transformed to the α-form at the high temperature. (Somiya, 1987). Then, η-alumina is observed on heat treatment. The degree of crystallization is only 50-55% and the crystal size is small, around 6nm. Another noticeable feature of the microstructure is the large number of pores which gives a low effective density and the service temperature is 900°C. Continue heating results in the progression through the γ-alumina with a contaminant increase in strength and toughness. (Matthews et al, 1994). This different type of alumina form is chosen depend on intended use among all those commercially available.

2.2.1 The Alumina Manufacturing Process

Alumina, Al_2O_3, is produced by using Bayer process in ceramics industries as shown in Figure 2.1. The basic raw material used is bauxite. The principal operations in the Bayer process are physical beneficiation of the bauxite, digestion (in the presence of caustic soda, NaOH at an elevated temperature and pressure), clarification, precipitation and calcination, followed by crushing, milling, and sizing. During the digestion, most of the hydrated alumina goes into solution as sodium aluminate: