UNIVERSITI TEKNIKAL MALAYSIA MELAKA

The Use of MgO as Sintering Aid for Alumina Ceramics

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

By

SITI NOR ELYINA BINTI HAJI AB ZARIM

Faculty of Manufacturing Engineering

May 2008

© Universiti Teknikal Malaysia Melaka

UTeM Library (Pind.1/2007)

UNIVERSITI TEKNIKAL MALAYSIA MELAKA

BORANG PENGESAHAN STATUS LAPORAN PSM

JUDUL: **The use of MgO as sintering aid for Alumina ceramics**

SESI PENGAJIAN: **2007/2008**

Saya **SITI NOR ELYINA BINTI HAJI AB ZARIM**

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 dan penulis.
2. Perpustakaan Universiti Teknikal Malaysia Melaka dibenarkan membuat salinan untuk tujuan pengajian sahaja dengan izin penulis.
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)

Alamat Tetap:
22, Jalan 4, Selayang Pandang,
68100 Batu Caves, Selangor.

Tarikh: 7 May 2008

(TANDATANGAN PENYELIA)

Cop Rasmi:
DR. AZIZAH BINTI SHAABAN
Pensyarah
Fakulti Kejuruteraan Pembuatan
Universiti Teknikal Malaysia Melaka
Karung Berkunci 1200, Ayer Keroh
75450 Melaka

Tarikh: 7 May 2008

* 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.

© Universiti Teknikal Malaysia Melaka

DECLARATION

I hereby, declared this thesis entitled
"The Use of Magnesium Oxide (MgO) as Sintering Aid for Alumina Ceramics"
is the results of my own research
except as cited in references.

Signature	:	
Author's Name	:	**SITI NOR ELYINA BINTI HAJI AB ZARIM**
Date	:	**7 MAY 2008**

© Universiti Teknikal Malaysia Melaka

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 (Material Engineering).
The members of the supervisory committee are as follow:

………………………………
(Dr. Azizah Binti Shaaban)

(Official Stamp & Date)

© Universiti Teknikal Malaysia Melaka

ABSTRACT

The materials that involved in this experiment were Alumina ceramics and Magnesium Oxide (MgO). Alumina ceramics have high melting temperatures, hot and cold mechanical strength and are good for abrasion and corrosion resistant applications. The main objectives of this study are to determine the effectiveness of sintering aid for alumina where alumina is doping with Magnesium Oxide (MgO). Sintering of alumina products usually occurred at temperature above 1700˚C. Because grain growth of alumina is sensitive to sintering temperature, abnormal grain growth in alumina would occur in the final stage of densification if sintering temperature is too high, which has a great influence on flexural strength and wear resistance of pure alumina increase with decreasing grain size. The process that was conducted for this study was powder processing. The sintering aid, MgO which is added in smaller composition (1%) may enhance sintering process. This was because MgO is controlled the aspect ratio of grains and for better densification. The dopant powder is compacted and sintered at three temperature ranges which were 1300˚C, 1350˚C and 1400˚C. After sintering process, the micro-structural, physical and mechanical properties of sintered product were evaluated by using Scanning Electron Microscopy (SEM), Energy Disperse X-Ray (EDX), hardness testing and Archimedes principle. The result was found that the MgO is effective as sintering aid for alumina ceramics.

© Universiti Teknikal Malaysia Melaka

ABSTRAK

Kajian ini bertajuk Penggunaan Magnesium Oxide (MgO) sebagai Bahan Pemangkin dalam Pembakaran Alumina, Al_2O_3 Seramik. Seramik Alumina mempunyai ketahanan pada tinggi suhu, kekuatan mekanik panas dan sejuk. Objektif utama kajian ini adalah untuk menentukan keberkesanan MgO sebagai bahan pemangkin dalam proses pembakaran seramik. Pembakaran produk-produk alumina biasanya berlaku pada suhu lebih daripada 1700˚C. Oleh kerana pertumbuhan butiran alumina adalah peka kepada suhu, terdapat tumbesaran butiran yang luar biasa dalam alumina pada peringkat akhir penumpatan jika suhu pembakaran adalah terlalu tinggi dan mempunyai satu pengaruh yang besar terhadap kekuatan lenturan dan mempunyai sifat ketahanan terhadap kehausan apabila saiz butiran menurun. Proses yang telah dikendalikan untuk kajian ini ialah pemprosesan serbuk. MgO sebagai bahan pemangkin dalam proses pembakaran seramik yang ditambah dalam komposisi 1% mungkin dapat menigkatkan lagi proses pembakaran Alumina. Ini adalah kerana MgO adalah mempunyai nisbah aspek bijian yang terkawal dan lebih baik untuk penumpatan. Alumina seramik yang telah dicampur dengan MgO dipadatkan dan dibakar pada tiga suhu tempat yang berlainan iaitu 1300˚C, 1350˚C dan 1400˚C. Selepas proses pembakaran, mikro struktur, ciri-ciri fizikal dan mekanikal diuji dengan menggunakan Scanning Electron Microscopy (SEM), Energy Disperse X-Ray (EDX), ujian kekuatan Vickers dan prinsip Archimedes. Hasilnya, MgO adalah berkesan sebagai bahan pemangkin dalam membantu pembakaran Alumina seramik.

© Universiti Teknikal Malaysia Melaka

DEDICATION

To my mother and father, and my lovely family thank you for your undying love and support. To all my friend and lecturers, thank you for your support.

© Universiti Teknikal Malaysia Melaka

ACKNOWLEDGEMENTS

Alhamdulillah, praise to God, with the deepest sense of gratitude of the Almighty ALLAH who gives strength and ability to complete this project and thesis as it today.

First of all, I would like to express my unlimited gratitude to my family who has constantly been supportive throughout the project development. They have been the ones gave me strength working on this project until it is complete. I am very grateful having them in my life.

I would like to express my sincere appreciation to my project supervisor, Dr. Azizah Binti Shaaban for her encouragement, advices and guidance in completing this study.

© Universiti Teknikal Malaysia Melaka

Finally, I would like to thank all my friends and technician who have given me a lot of guidance and help to complete this project. Throughout the study of The Use of Magnesium Oxide (MgO) as Sintering Aid for Alumina Ceramics, their advices and assistance in various ways have been very helpful.

TABLE OF CONTENTS

© Universiti Teknikal Malaysia Melaka

© Universiti Teknikal Malaysia Melaka

LIST OF FIGURES

© Universiti Teknikal Malaysia Melaka

LIST OF TABLES

© Universiti Teknikal Malaysia Melaka

© Universiti Teknikal Malaysia Melaka

LIST OF ABBREVIATIONS, SYMBOLS, SPECIALIZED NOMENCLATURE

Al_2O_3	-	Aluminum Oxide
CIP	-	Cold Isostatic Pressing
CaO	-	Calsium Oxide
EDX	-	Energy Disperse X-Ray
EPA	-	Environmental Protection Agency
Fe_2O_3	-	Iron (III) Oxide
HCP	-	Hexagonal Close Packed
HV	-	Vickers Pyramid Number
K_2O	-	Potassium Oxide
MgO	-	Magnesium Hydroxide
Na_2O	-	Sodium Oxide
P_2O_5	-	Phosphorus Pentoxide
SEM	-	Scanning Electron Microscopy
SiO_2	-	Silicon Dioxide
TRI	-	Toxics Release Inventory
UKM	-	Universiti Kebangsaan Malaysia
UTeM	-	Universiti Teknikal Malaysia Melaka

© Universiti Teknikal Malaysia Melaka

CHAPTER 1

INTRODUCTION

1.1 Materials Background

Ceramics are diverse group of nonmetallic, inorganic solid compounds with a wide variety of compositions and properties. Ceramics are crystalline compounds consisting of metallic and nonmetallic elements whose properties differ from the constituents. Ceramics in general are hard, brittle and stiff. They are generally higher in compressive strength than tensile. They are totally elastic, meaning they exhibit no plasticity when a load is applied. There is little or no deformation prior to fracture and they have the highest melting points of any materials.

Magnesium oxide, or magnesia, is a white solid mineral that occurs naturally as periclase and is a source of magnesium (see also oxide). It has an empirical formula of MgO. It is formed by an ionic bond between 1 magnesium and 1 oxygen atom. Magnesium oxide is easily made by burning magnesium ribbon which oxidizes in a bright white light, resulting in a powder. It is hygroscopic in nature and care must be taken to protect it from moisture. Magnesium hydroxide forms in the presence of water ($MgO + H2O \rightarrow Mg(OH)_2$), but it can be reversed by heating it to separate moisture.

© Universiti Teknikal Malaysia Melaka

In medicine, magnesium oxide is used for relief for heartburn and sore stomach, as an antacid, magnesium supplement, and as a short-term laxative. It is also used to improve symptoms of indigestion. Side effects of magnesium oxide may include nausea and cramping. It is used also as a basic refractory material for lining crucibles.

Alumina which is also known as Aluminum Oxide is an amphoteric oxide of aluminum with the chemical formula Al_2O_3. Alumina was taken off the United States Environmental Protection Agency's chemical lists in 1988. The largest manufacturers in the world of Alumina are Alcoa, Alcan and Rusal which is specialize in the production of specialty Alumina.

Aluminum Oxide or Alumina is one of the most versatile of refractory ceramic oxides and finds use in a wide range of applications. Alumina is one of the important engineering ceramic materials because of its high-temperature stability and the retention of strength at high temperatures and cheap starting powder. Especially in recent years, high purity Alumina ceramics are more widely used for tribological applications such as grinding tools and grinding media.

1.1.1 Structure of Alumina

The most common form of crystalline Alumina, α-aluminum oxide, is known as corundum, emery, sapphire and ruby and has a hexagonal close packed (HCP) lattice structure. There are more than 25 different solid phases, or forms, have been described as γ, θ, κ, χ, δ and η. Alumina occurs in two crystalline forms.

Alpha Alumina is composed of colorless hexagonal crystals as shown in Figure 1.1, while 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. Alumina powder is

© Universiti Teknikal Malaysia Melaka

formed by crushing crystalline Alumina where it is white when pure. Dense Alumina microstructures with grain sizes of about 0.5 μm are common products of the grinding industry.

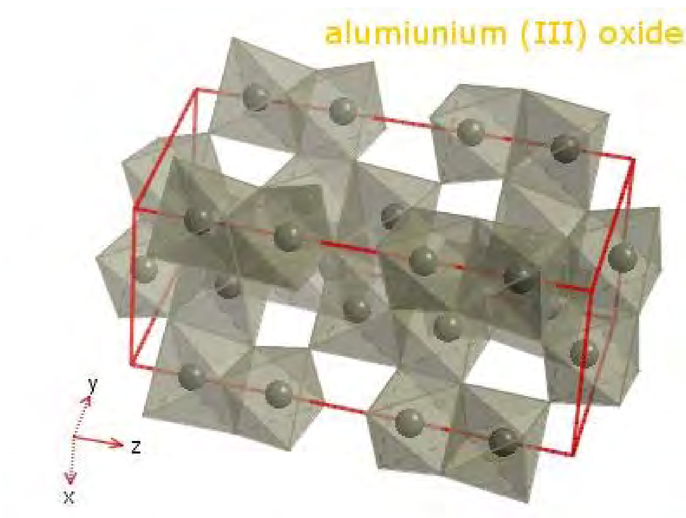

Figure 1.1: Structure of Alumina

Source: Greenwood and Earnshaw (1997)

1.1.2 Properties of Alumina

Among the oxide ceramics, Alumina, Al_2O_3, is the most commonly used ceramic because of its high hardness, wear resistance, high modulus, inertness, refractoriness and adequate strength. Some of the properties of Alumina are displayed in Table 1.1. Paradoxically, the development of high purity Alumina ceramics was driven not by their structural application but by the need for low electrical conductivity insulators.

Alumina ceramics have high melting temperatures, hot and cold mechanical strength and are good for abrasion and corrosion resistant applications where heat resistance is also important. Alumina ceramics can be extremely hard, exceeded only by silicon carbide, boron carbide and diamond. They also have outstanding electrical and thermal

© Universiti Teknikal Malaysia Melaka

properties. For example, spark plugs are made using high Alumina porcelain about 90% for its insulating properties coupled with its strength, heat and thermal shock resistance.

High purity of Alumina ceramics can provide such good resistance to chemical attack that can resist hydrofluoric acid and molten alkalis and alkali vapors. The chemical inertness of these same bodies make them ideal for making valves and seals exposed to severe corrosive and abrasive conditions. Alumina ceramics also resist the effects of radiation that can destroy other materials. Alumina ceramic can have very high dielectric strength, high resistivity and low dielectric loss.

Alumina is generally white but is sometimes pink (88% Alumina) or brown (96% Alumina). The color is derived from either the sintering additives or impurities in the raw materials. Good to chemical stability of Alumina, leads to a high corrosion resistance. It is insoluble in water and only slightly soluble in strong acid and alkaline solutions.

Table 1.1: Alumina Oxide (Alumina) properties

ELEMENT	VALUE
Sintered Density	3.97 g cm^{-3}, solid
Melting Point	2050 °C
Nominal Grain Size	5 μm
Tensile Strength(MPa)	620
Young's Modulus (GPa)	380

Source: M. Backer (2007)

© Universiti Teknikal Malaysia Melaka

1.1.3 Applications of Alumina

Alumina powders, that sinter at as low temperature as possible are needed for production of prime quality multilayer substrates for ultra large-scale integrated circuits. Simple yet precise systems for locating pores and controlling pore size must be developed. For the electronics grade Alumina, an extremely severe linear shrinkage tolerance (±0.15%) in sintering is required.

In the field of refractoriness, efforts to develop new or improved Alumina based raw materials will be continued. These products are required to meet the ever-increasing severity of service conditions in the steel and other industries utilizing refractoriness. Polycrystalline Alumina will continue to be used successfully as an artificial bone implant material for body joints and other parts. In biomedical, high purity Alumina are used as orthopedic implants particularly in hip replacement surgery. Tooth implants made on single-crystal Alumina will become more common in the future because of their strength. There are various example applications of Alumina ceramic in Figure 1.2.

Alumina is also the byproduct of hydrogen generation for the purposes of fuel generation when water is added to pellets comprised of aluminum and gallium. The other byproduct of the reaction is of course gallium.

The high hardness of Alumina imparts wear and abrasion resistance and hence it is used in diverse applications such as wear resistant linings for pipes and vessels pump and faucet seals, thread and wire guides. The high volume resistivity and dielectric strength make Alumina an excellent electrical insulator which leads to applications in electronics as substrates and connectors, and in lower duty applications such as insulators for automotive spark plugs.

© Universiti Teknikal Malaysia Melaka

Figure 1.2: Various application of Alumina ceramic

Source: Morgan Advanced Ceramics Inc. (2007)

1.2 Objectives

The main objective of this study is to determine the effectiveness of sintering aid for Alumina. In this study, Alumina is doping with Magnesium Hydroxide (MgO). A small amount of MgO will be added to control the grain size during sintering. The second objective is to observe the microstructures, physical and mechanical properties of dopant sintered Alumina which will be characterized by Scanning Electron Microscopy (SEM), Energy-Dispersive X-ray (EDX), Archimedes principle and Hardness Test (Vickers).

© Universiti Teknikal Malaysia Melaka

1.3 Problem Statement

Ceramic powders are particulates, usually with a micron or mesh size distribution, fabricated through atomization, crushing, milling, precipitation or other processes. Sintering of Alumina products usually occurred at temperature above 1700˚C. Because grain growth of Alumina is sensitive to sintering temperature, abnormal grain growth in Alumina would occur in the final stage of densification if sintering temperature is too high, which has a great influence on flexural strength and wear resistance of pure Alumina increase with decreasing grain size.

Sintering aid like MgO which will add in smaller composition (1%) may enhances sintering process. This is because MgO will control the aspect ratio of grains and for better densification. Alumina powder will be mixed with MgO by tumbling technique. The mixture will be sintered at three ranges of temperature which are 1300˚C, 1350˚C and 1400˚C. Micro-structural, physical properties and mechanical properties will be evaluated.

© Universiti Teknikal Malaysia Melaka

CHAPTER 2

LITERATURE REVIEW

2.1 Sintering

Sintering is a processing technique used to produce density-controlled materials and components from ceramic powders by applying thermal energy. Sintering will affect certain parameter during the process such as their purity, density and sintering temperature.

All the characteristic temperatures associated to phase's transformation, glass transitions and melting points, occurring during a sinterisation cycle of a particular ceramics formulation can be easily obtained by observing the expansion-temperature curves during optical dilatometer thermal analysis. In fact, sinterisation is associated to a remarkable shrinkage of the material due to the fact that glass phases flow, once their transition temperature is reached, and start consolidating the powdery structure and considerably reducing the porosity of the material (Wikipedia, 2008).

© Universiti Teknikal Malaysia Melaka

© Universiti Teknikal Malaysia Melaka

2.1.1 Purity

Rao et al. (1999) investigated that sintering temperature for Alumina densification usually increases with increases of its purity. If high purity Alumina powders are prepared by traditional methods, the sintering temperature should be over 1700˚C to get dense Alumina sintered body. Because grain growth of Alumina is sensitive to sintering temperature, abnormal grain growth in Alumina would occur in the final stage of densification if sintering temperature is too high, which has a great influence on flexural strength and wear resistance of pure Alumina increase with decreasing grain size. In recent years, high purity Alumina powders prepared by chemical methods enable them to sinter at temperatures lower than 1600˚C.

2.1.2 Density

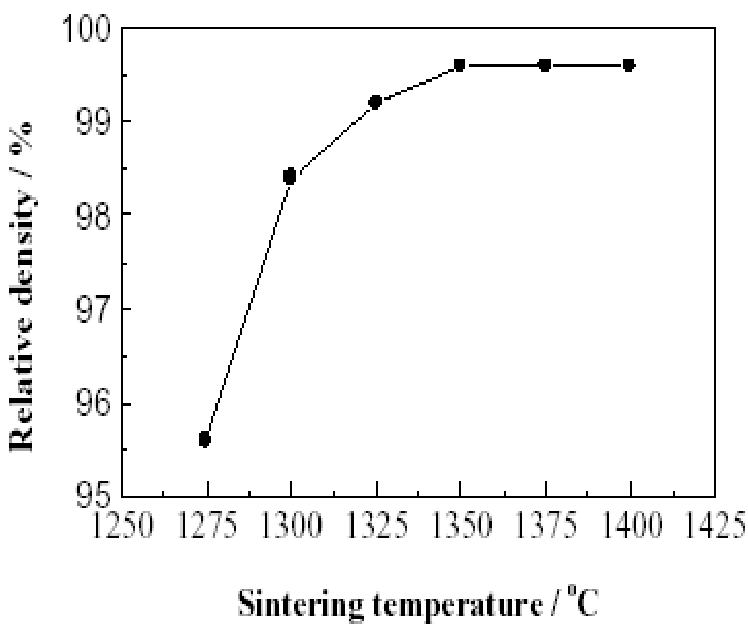

Figure 2.1: Effect of sintering temperature on relative density of Alumina ceramics

Source: Rao et al. (1999b)

© Universiti Teknikal Malaysia Melaka