UNIVERSITI PUTRA MALAYSIA

PHYSICAL AND MECHANICAL PROPERTIES OF NANOCOPPER PARTICLE-REINFORCED ALUMINA MATRIX COMPOSITES

MOHAMMED SABAH ALI

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By

MOHAMMED SABAH ALI

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfillment of the Requirements for the Degree of Doctor of Philosophy

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DEDICATION

To the spirit of my dear father (Sabah Ali Al-Mayali)

To my mother
For her unconditional love and support

To my siblings and family
For making my life complete

To my wife (Intisar), daughters (Noor and Tabark), and sons (Ali and Hussain)
For their love and care

To all my very wonderful friends
For making my life full of joy and happiness

Thank you all.

Mohammed Sabah Ali

May 2017

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfillment of the requirement for the Degree of Doctor of Philosophy

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September 2017

Chairman : Associate Professor Azmah Hanim Mohamed Ariff, PhD
Faculty : Engineering

Over the past century, there has been a dramatic increase in fabrication and synthesizing of porous ceramics. However, only a few of them used waste material to fabricate alumina porous ceramics and reinforced it using nano-copper (Cu) particles. The motivation behind these efforts are the increasing raw materials cost and decreasing natural resources consumption which requires the use of byproducts and wastes as raw material for different industrial processes. This is a step towards environmental protection, sustainable development, and also to produce porous alumina ceramics with good porosity and mechanical properties. Thus, in this study, porous alumina ceramics were fabricated using graphite waste, natural active yeast, and rice husk ash as pore-forming agents and source of silica (SiO_2). Series of porous alumina ceramics was prepared using powder metallurgy technique. The physical and mechanical properties of porous alumina ceramics with and without nano-copper (Cu) particles were measured by differential thermal analysis (DTA), energy-dispersive X-ray spectroscopy (EDX), linear shrinkage, average density (green and sintered) data measurement, and Universal Testing Machine (UTM). The average densities for both green and sintered samples decrease with increasing pore forming agent ratio for porous alumina ceramics with and without nano-copper (Cu) particles. While the linear shrinkage increases with the increase of pore forming agent ratio with and without nano-copper (Cu) particles. Besides, the structural properties of porous alumina ceramics with and without nano-copper (Cu) particles, ceramic phases, morphology, and porosity were examined using X-ray diffraction (XRD) and field-emission scanning electron microscopy (FESEM). The effects of the pore-forming agent ratios on the mechanical properties, the porosity and the microstructure with and without nano-copper (Cu) particles have been investigated in this study. The results showed that through increasing the pore-forming agent ratio for graphite waste, natural active yeast, and rice husk ash, the porosity increased from 37.3 to 61.1%, 30.2 to 63.8% and 42.9 to 49.0%, respectively. The hardness also decreased from 172.6 to 38.1 HV_1 and from 160.6 to 15.0 HV_1 for porous alumina ceramics using graphite waste and yeast as pore-forming agents, respectively.

However, the hardness of the porous alumina ceramics with rice husk ash as a pore-forming agent increased at 30 wt.% (150.9 HV_1) and 50 wt.% (158.9 HV_1). The tensile strength for porous alumina ceramics using graphite waste and natural active yeast as pore-forming agents decreased from 24.9 to 14.3 MPa and from 26.2 to 5.4 MPa, respectively. The compressive strength decreased from 112.3 to 34.3 MPa and from 19.5 to 1.8 MPa, respectively. The flexural strength decreased from 71.28 MPa to 30.42 MPa and from 72.56 MPa to 20.72 MPa, respectively. However, for porous alumina ceramics using rice husk ash, the tensile strength increased at 30 wt.% (24.1 MPa) and 50 wt.% (21.9 MPa). The compressive strength also increased at 30 wt.% (69.7 MP) and at 50% (60.1 MPa). The flexural strength increased at 30 wt.% (93.38 MPa) and 50 wt.% (92.38 MPa). The variation in mechanical properties was also attributed to the formation of ceramic phases such as mullite, cristobalite, corundum, and sillimanite other than the formation porosity. It is also found that with increasing porosity, the mechanical properties decrease. This is a good agreement with Rice's formula. While by adding nano-copper (Cu) particles all mechanical properties improved with increasing Cu ratio which attributed to decrease porosity and formation ceramic phases such as tenorite (CuO).

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk Ijazah Doktor Falsafah

SIFAT FIZIKAL DAN MEKANIKAL ZARAH TEMBAGA NANO BERTETULANG KOMPOSIT MATRIKS ALUMINA

Oleh

MOHAMMED SABAH ALI

September 2017

Pengerusi : Profesor Madya Azmah Hanim Mohamed Ariff, PhD
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Sejak ber abad yang lalu, terdapat peningkatan dramatik dalam fabrikasi dan sintesis seramik berliang menggunakan bahan-bahan buangan. Walau bagaimanapun, hanya sebahagian sahaja menggunakan bahan buangan untuk menghasilkan alumina seramik berliang dan diperkukuh menggunakan zarah nano tembaga (Cu). Motivasi di sebalik usaha ini adalah kerana kurangnya penggunaan sumber asli dan kos bahan mentah yang semakin meningkat yang memerlukan penggunaan hasil sampingan dan sisa sebagai bahan mentah untuk proses industri yang berbeza. Ini adalah satu langkah ke arah perlindungan alam sekitar dan pembangunan lestari serata untuk menghasilkan seramik berliang alumina dengan keliangan yang sesuai dan sifat-sifat mekanikal yang baik. Oleh itu, dalam kajian ini, alumina seramik berliang telah direka menggunakan sisa grafit, yis aktif semula jadi dan abu sekam padi sebagai ejen pembentuk liang dan sumber silika (SiO_2). Beberapa seramik berliang alumina telah disediakan dengan menggunakan teknik metalurgi serbuk. Sifat-sifat fizikal dan mekanikal seramik alumina berliang samada dengan dan tanpa zarah nano-tembaga (Cu) diukur melalui analisis terma (DTA), tenaga-serakan X-ray spektroskopi (EDX), pengecutan linear, ketumpatan purata (hijau dan tersinter) pengukuran data dan mesin ujian sejagat (UTM). Ketumpatan purata bagi kedua-dua sampel hijau dan tersinter menurun dengan peningkatan nisbah ejen pembentuk liang untuk seramik alumina berliang dengan dan tanpa zarah nano-tembaga (Cu). Manakala, pengecutan linear meningkat dengan peningkatan nisbah ejen pembentuk liang dengan dan tanpa zarah nano-tembaga (Cu). Di samping itu, sifat-sifat struktur alumina seramik berliang dengan atan tanpa zarah nano-tembaga (Cu), fasa seramik, morfologi dan keliangan telah diperiksa menggunakan X-ray pembelauan (XRD) mikroskop elektron pengimbas (FESEM). Kesan nisbah ejen pembentuk liang ke atas sifat mekanik, keliangan dan mikrostruktur dengan dan tanpa zarah nano-tembaga (Cu) telah disiasat dalam kajian ini. Hasil kajian menunjukkan bahawa dengan meningkatkan nisbah ejen pembentuk liang bagi sisa grafit, yis aktif semulajadi dan abu sekam padi, keliangan meningkat setiap satu daripada 37.3 ke 61.1%, 30.2 ke 63.8% dan 42.9 ke 49.0%. Kekerasan juga menurun 172.6 ke 38.1 HV_1 dan 160.6 ke 15.0 HV_1

untuk seramik alumina berliang menggunakan sisa grafit dan yis sebagai ejen pembentuk liang. Walau bagaimanapun, kekerasan seramik alumina berliang dengan abu sekam padi sebagai ejen pembentuk liang meningkat pada 30 wt.% (150.9 HV_1) dan 50 wt.% (158.9 HV_1). Kekuatan tegangan untuk seramik alumina berliang menggunakan sisa grafit dan yis aktif semulajadi sebagai agen pembentuk liang menurun daripada 24.9 ke 14.3 MPa dan 26.2 ke 5.4 MPa. Kekuatan mampatan menurun daripada 112.3 ke 34.3 MPa dan 19.5 ke 1.8 MPa. Kekuatan lenturan menurun daripada 71.28 MPa kepada 30.42 MPa dan dari 72.56 MPa kepada 20.72 MPa, secara respektif. Walau bagaimanapun, untuk seramik alumina berliang menggunakan abu sekam padi, kekuatan tegangan meningkat pada 30 wt.% (24.1 MPa) dan 50 wt.% (21.9 MPa). Kekuatan mampatan juga meningkat pada 30 wt.% (69.7 MP) dan pada 50% (60.1MPa). Kekuatan lenturan meningkat pada 30 wt.% (93.38 MPa) dan 50 wt.% (92.38 MPa). Perubahan dalam sifat-sifat mekanikal juga disebabkan oleh pembentukan fasa seramik seperti mullite, cristobalite, aluminum oksida dan sillimanite selain daripada pembentukan keliangan. Kajian mendapati dengan peningkatan keliangan, sifat-sifat mekanikal berkurangan. Ini adalah bersamaan dengan formula Rice. Walau bagaimanapun selepas menambah zarah nano-tembaga (Cu), semua sifat-sifat mekanikal meningkat dengan peningkatan nisbah Cu yang dikaitkan dengan mengurangkan bilangan keliangan dan pembentukan fasa seramik seperti tenorite (CuO).

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Mohammed Sabah Ali

May 2017

I certify that a Thesis Examination Committee has met on 12 September 2017 to conduct the final examination of Mohammed Sabah Ali on his thesis entitled "Physical and Mechanical Properties of Nanocopper Particle-Reinforced Alumina Matrix Composites" in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the Doctor of Philosophy.

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This is to confirm that:

- the research conducted and the writing of this thesis was under our supervision;
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LIST OF ABBREVIATIONS

ACP	Ammonium hexachloroplatinate
ASTM	American Society for Testing and Materials
DTA	Differential thermal analysis
DTS	Diametric tensile strength
EDX	Energy-dispersive X-ray
FESEM	Field-emission scanning electron microscopy
HAP	Hydroxyapatite
HRD	Hardness
JCPDS	Joint Committee on Powder Diffraction Standards
KP	Kenaf powder
PMMA	Polymethylmethacrylate
Pos.[2θ]	Position [2θ]
PVB	Polyvinyl butyral
R&D	Research and development
RBAO	Reaction bonding of aluminum oxide
RHA	Rice husk ash
RPC	Reticulated porous ceramic
S. D	Stander deviation
TEM	Transmission electron microscopy
TGA	Thermogravimetric Analysis
UTM	Universal Testing Machine
XRD	X-ray diffraction

CHAPTER 1

INTRODUCTION

Overview

This study investigates the effect of different pore-forming agents (graphite waste from the primary battery, natural active yeast, and rice husk ash) on the physical, microstructural and mechanical properties of porous alumina ceramics with and without nano-copper particles (Cu). The physical properties included porosity, density (green and sintered), and linear shrinkage. The microstructural properties involved morphology, pore shape, and grains while the mechanical properties included the hardness, compressive strength, tensile strength and flexural strength. This study involved using sacrificial and pressureless techniques to improve the mechanical properties of porous alumina ceramics using waste materials and sugar as a binder.

This chapter highlights the research background, problem statement, research hypothesis, research objective, the scope of this study and contributions to knowledge.

1.1 Research background

The solid materials that have been obtained from the burning of clays are known the ceramics, which derived from the Greek word keramos. Also, the ceramics can be defined as materials, which often include crystalline structure, inorganic and non-metallic materials. The ceramic materials involve of both nonmetallic and metallic elements such as Si_3N_4, ZrO_2, CaO, SiO_2, and Al_2O_3. In other words, based on the modern definition, ceramics materials are either amorphous or crystalline solid materials comprising only covalent, ionic or ionocovalent chemical bonds between nonmetallic and metallic elements. Firing and calcining are the important processes used in the preparation the ceramic and raw materials. Burning or firing is the final heat treatment conducted in the furnace on the green ceramic material to develop a strong chemical bond and produce other required chemical, mechanical and physical properties. Calcining involves the heat treatment of raw materials before used to produce the final ceramic materials. The point of calcination is to produce changes in volume and remove the combined constituents which will volatile chemically (Cardarelli, 2008).

Based on the industrial applications of ceramic materials, ceramics are classified to major categories such as cements, refractories, glasses, abrasives, and advanced porous ceramics. Today, one of the important industrial applications of ceramic materials is the advance porous ceramics due to their benefit in the scientific and industrial fields, which focus on the relationship between properties and microstructure, developments of processing and discovering new application. The unique properties of tailored porous ceramic, such as its excellent strain and damage tolerance, good thermal shock resistance, wear resistance, high corrosion and its lightweight, render advanced ceramic as potential components (Jean, 2014; Zhang *et al.*, 2012) of filtering materials for separation membranes, lightweight structural materials (Tang, 2004), catalyst supports, thermal insulation, bioreactors, gas filters for high temperature, (Dessai, 2013; Dong *et al.*, 2017; Yu, 2011) medical ultrasonic imaging and underwater sonar detectors. Therefore, these advantages, make advanced porous ceramic more distinctive compared to other materials such as polymeric and metallic materials in certain applications (Rahaman, 2006).

Ceramics with designed porosity is one of the most versatile materials for thermal insulation, filters, bio-scaffold for tissue engineering, absorption and as catalysts (Konrad *et al.,* 2014). The past decade has seen the rapid development of porous ceramic, several efforts have been devoted by the researchers on inventing porous ceramic processing technologies, that lead to a significant improvement in porous ceramic structure and properties (Hammel *et al.*, 2014; Ohji and Fukushima, 2012).

Macroporous ceramics with designed porosity have a wide application including 1- filtration in high temperature 2- diesel filters 3- thermal insulation 4- bone implants and others. In addition, replica, sacrificial templates, and direct foaming methods have been discovered by several scientists for manufacturing macroporous ceramics (Ahmad *et al*, 2014) as shown in Figure 1.1.

Generally, porous ceramics can be classified into three grades according to its pore diameter: 1) micro-pore ceramics in the range of $d < 2$ nm, 2) meso-pore ceramics in the range of 50 nm $> d >$2 nm, and 3) macro-pore ceramics in the range of $d > 50$ nm. (Ohji and Fukushima, 2012; Studart *et al.*, 2006). For example, meso- and macro-pore ceramics are desired in sensors and catalysis to supply a high surface area and to improve the accessibility of liquids and gases to reactive areas. Small pores in the range of 50-100 nm are desired to provide physical cues that promote differentiation, proliferation, the migration of cells and finally quick healing. Large pores >300 – 400 µm with hierarchical structures are desired in regenerative medicine for implanted scaffold vascularization (Studart *et al*., 2011).

Unfortunately, the mechanical properties of porous ceramics decreased when the porosity area increased and the fracture toughness of ceramic is also low.

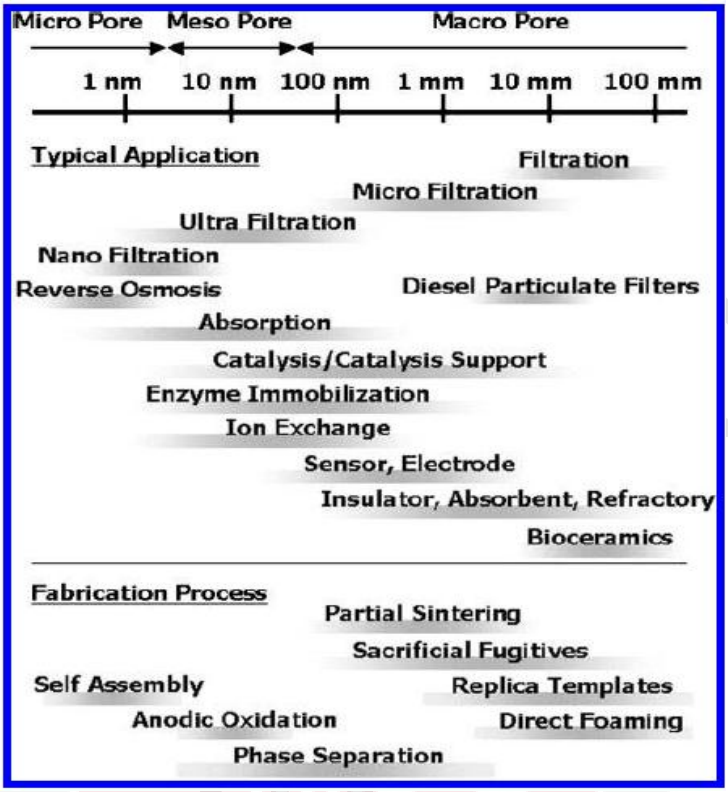

Figure 1.1: Classification of porous ceramics according to pore size, applications, and fabrication methods (Ohji and Fukushima, 2012)

1.2 Problem statement

The motivation behind these efforts are the increasing raw materials cost and decreasing natural resources consumption which requires the use of byproducts and wastes as raw material for different industrial processes. This is also a step towards environmental protection and sustainable development. Because of the large amounts of agricultural and industrial waste in the world this days, the present research would like to use graphite waste from primary battery as industrial waste, natural active yeast as microorganism's materials and rice husk ash as pore-forming agent to produce macroporous ceramic materials reinforced with ductile nano-metals particles (nano-copper).

In spite of the growth in macroporous ceramics with designed porosity and their wide applications including filtration in high temperature, diesel filters, thermal insulation, bone implants, absorptions, and catalyst. The main disadvantage of porous ceramic with designed porosity that is the decreasing mechanical properties when the porosity increase. In filters, the mechanical properties must be strong enough to withstand the pressure during operating time and must have thermal and chemical properties that is important for it to function sustainably especially in hot gas and molten metal filtration (Hammel *et al.*, 2014; Konrad *et al.*, 2014; Ohji and Fukushima, 2012). Therefore, in the case of the filtration of hot gas and molten metal, the fluctuation of temperature during the process will leave the materials liable to thermal shock. During service, the mechanical properties of the filter must be high enough to bear the operation pressure, and also the filter properties must not deteriorate with the temperature increase. In addition, the range of temperature (260-900ºC) in the filtration process is considered in the filtration of hot gas and these filters may face pressures of up to 8 MPa. Because filtration occurs under these conditions, it is important that the filters of ceramics have sufficient mechanical strength and thermal shock resistance (Hammel, 2014). Therefore, in this study, nano-copper particles have been used as a reinforcement factor to improve the mechanical properties of porous alumina samples. The conditions for the porous alumina ceramics include a reinforced phase when sintering at high temperatures using a new process that requires the addition of Cu metal in nanoscale directly through a combination of the sacrificial technique and pressure-less sintering methods which is a cost-effective procedure.

1.3 Research hypothesis

This study is carried out with three main hypotheses as follows.
1- Depending on the thermal properties of pore-forming agents, it can produce alumina porous ceramics with different level of porosity and mechanical properties through sintering at high temperature.
2-The presence of porosity with different levels leads to decrease in the mechanical properties of alumina porous ceramics however the presence of ceramic phases such as silica (SiO_2) plays a significant role in improving mechanical properties despite the presence of porosity.
3-Addition of nano-metal particles in porous alumina ceramics would affect strongly the mechanical properties by decreasing the porosity, toughening mechanism, and formation of ceramic phases.

1.4 Research objectives

In the present research work, porous alumina ceramics with and without nano-copper particles (Cu) have been prepared using pressureless and sacrificial techniques. All porous alumina ceramics were characterized for the physical and mechanical properties.

The research objectives are;

- To investigate the pore formation in alumina matrix with graphite waste, natural active yeast, and rice husk ash (RHA) and its effect on the physical properties.
- To determine the relationship between different pore modifier wt. % from 10 to 50% on the pore formation and the relationship to the mechanical properties.
- To investigate the physical properties of alumina matrix with different pore modifier reinforced with copper particles between 3-12 wt.%.
- To investigate mechanical properties of alumina matrix with different pore modifier reinforced with copper particles between 3-12 wt.%.

1.5 Scope of the study

In order to reach the objective of the study, the scope of the study are as follows.

1- Porous alumina ceramics have been prepared using different pore agents (graphite waste, natural active yeast, and rice husk ash) based on the ratios 10 wt.%, 20 wt.%, 30 wt.%, 40 wt.%, and 50 wt.% of pore agent using the sacrificial and pressureless sintering techniques.
2- A reinforced porous alumina ceramics have been prepared using Cu metal in nanoscale particles as reinforcement phase through the ratios of 3 wt. %, 6 wt. %, 9 wt. % and 12 wt. % of (Cu) metal for selected ratios of all pore agent.
3- The chemical phases and chemical composition of pore agents and alumina powder have been determined using XRD, TEM, and EDX in order to discover the chemical phases and chemical composition of pore agent and material matrix.
4- Identifying the first sintering temperature of green ceramics to remove the pore agent according to the weight loss by conducting the TGA and DTA of pore agent materials.
5- Mechanical properties of porous and reinforced porous alumina ceramics have been measured using UTM-machine.
6- Pore size distribution, physical and structural properties of porous and reinforced porous alumina ceramics have been analyzed using FESEM, XRD, Archimedes method, and linear shrinkage.

1.6 Importance of the study and limitation

1- Contribution of knowledge to the materials engineering field in the possibility of using new material as a pore former and improve the technique to strengthen and produce the macroporous ceramic with porosity designed by using ductile nano metal particles.
2- To manufacture porous ceramic composite by using industrial and agricultural waste.
3- To produce porous ceramic composites with high mechanical properties by adding the nano metal particle.
4- To produce macroporous ceramic materials that can be used in potentials application, for example, metal filters, hot gas filters, membranes, and bioceramics. In addition, one of the importance limitation of producing of porous ceramics using sacrificial fugitives is low interconnectivity among the pores.

1.7 Outline of thesis

The thesis arrangement is designed as follows.

Chapter 1 explains an introduction of porous and reinforced porous alumina ceramics, the problem statement, the objective, the scopes and also the importance of this research study. The theory, features and previous works including the past and current work that has been carried out by other researchers of porous ceramics are explained in **Chapter 2.**

The methodology and characterization of the porous and reinforced porous alumina using graphite waste, natural active yeast and rice husk ash as pore-forming agent are explained **in Chapter 3**.

The results regarding the effect of the addition of different pore agent (graphite waste, natural active yeast, and rice husk ash) to alumina matrix, the effect of the addition of Cu metal in nanoscale, on the physical and mechanical properties of porous alumina ceramics are analyzed and discussed in **Chapter 4**. Finally, the conclusion and suggestion for future works are showed **in Chapter 5.**

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