Fabrication and Characterization of Microwave Sintered PM Fe-Cr-Y_2O_3 Composite

by

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A thesis submitted in fulfilment of the requirements for the degree of Masters of Science (Manufacturing Engineering)

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2013

ACKNOWLEDGEMENT

Praise and thanks are due to Almighty Allah,

the Most Gracious the Most Merciful

I would like to take this opportunity to express my heartiest gratitude to the following people for their invaluable help rendered during my candidature as a M. Sc. Student.

Firstly, I would like to express my truly gratitude and highest appreciation to my supervisors, Mr. Mohd Zamzuri Mohammad Zain, Dr Mohd Asri Selamat and Dr. Mohd Nazree Derman for their invaluable advice, encouragement and patience throughout this research work.

I would also like to express my heartfelt thanks to my colleagues and friends, especially Mr. Wan Abdul Rahman, Mrs Kartini, Mrs. Nooraizedfiza for their support and knowledge sharing throughout the entire period of this research work. Many thanks also to fellow postgraduates and undergraduate students from 2012 FYP batch for their help and friendship.

My parents deserve special mention for their inseparable support and prayers. To my father, Marzuki Abu Bakar and my mother, Aminah Baser Ahmad, thanks for your love and support for me to pursue my dream and ambition in the academic field. Words fail me to express my appreciation to my husband, Mohd Fahmi Shaari whose love, affection, persistent strength and confidence helps me going through thick and thin.

This thesis is my special dedication to my son, Muhammad Ammar Haziq. Raising him while working with this research has been a rewarding experience for me.

MARINA BINTI MARZUKI
2013

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LIST OF ABBREVIATIONS

Al	Aluminum
Al_2O_3	Alumina
Ap	Area concerned with pressure applied
Ar	Argon
ASTM	American Society for Testing and Materials
B_2Cr	Boranylidynechromiumylidyne
Be	Berilium
BN	Boron Nitride
C	Carbon
Co	Cobalt
Cr	Chromium
Cr_2O_3	Chromium oxide
Cr_2Ti	Titanium chromium compound
Cr_3C_2	Chromium carbide
CS	Conventional sintering
CTE	Coefficient of Thermal Expansion
Cu	Copper
CuSo4	Cuprum Sulphate
EDX	Energy Disperse X-ray Spectrometer
Fe	Iron
Fe(CO)5	Iron pentacarbonyl
Fe-Cr	Iron-chromium
Fe-Cr-Al2O3	Iron-chromium-yttria
FeO	Ferrum oxide
H_2SO_4	Sulphuric acid
HIP	Hot isostatic press
Mg	Magnesium
Mg-Y2O3	Magnesium-yttria
MHz	Megaherz
MMC	Metal matrix composite
Mn	Mangan
Mo	Molybdenum
MPa	Mega Pascal
MS	Microwave Sintering
N_2	Nitrogen gas
Ni	Nickel
$Ni(CO)_4$	Nickel carbonyl
P	Phosphorus
PM	Powder Metallurgy
ppm	part per million
S	Sulphur
SEM	Scanning Electron Microscope
SiC	Silicon carbide

Ti	Titanium
TiC	Titanium carbide
TiN	Titanium nitride
V	Vanadium
VC	Vanadium carbide
W-Ni-Fe	Tungsten – nickel –iron
XRD	X-ray diffractogram
Y_2O_3	Yttria
YAG	Yttria-alumina-garnet
Zr	Zirconia

LIST OF SYMBOLS

°C	degree Celcius
μm	micron
E	Young’s Modulus of Elasticity
F	Force
GHz	Gigaherz
m	meter
mm	millimeter
Pc	Compacting pressure
rpm	rotation per minute
vol.%	volume percentage
wt.%	weight percentage

Pencirian Komposit Metalurgi Serbuk Fe-Cr-Y_2O_3 disediakan dengan Kaedah Pensinteran Gelombang Mikro

ABSTRAK

Kajian ini tertumpu kepada penilaian keupayaan teknologi sinter gelombang mikro untuk menghasilkan komposit besi-kromium menerusi kaedah metalurgi serbuk. Komposit ini juga telah dibuat perbandingan dengan komposit yang sama dihasilkan dengan kaedah rawatan haba konvesional dari segi ciri-ciri fizikal dan mekanikal (kekerasan mikro dan kekuatan tumpat). Teknik sinteran gelomabng mikro terbukti sesuai bagi penyediaan komposit tersebut. Keputusan juga melihatkan bahawa komposit gelombang mikro besi-kromium mempunyai ketumpatan, kekerasan mikro dan kekuatan tumpat yang lebih baik dari komposit sinteran konvensional.Penilaian proses juga mendapati pensiteran gelombang mikro dapat mengurangkan 70% daripada masa pensinteran dibandingkan dengan kaedah pensinteran konvensional. Satu lagi matlamat kajian ini ialah untuk mengkaji kesan penambahan bahan penguat yttria ke atas komposit besi-kromium dengan berat berbeza dari 5, 10, 15 dan peratusan berat.Daripada kajian didapati cirri mekanikal komposit gelombang mikro bertambah baik dengan penambahan 5 % berat bahan penguat seramik ini.; Bagi komposit konvensional, ciri mekanikal terbaik adalah dari komposit yang ditambah bahan penguat sebanyak 10 % berat. Bagi cirri fizikal yanglain seperti ketumpatan pula menurun dengan peningkatan peratusan berat bahan penguat. Ini disebabkan oleh peningkatan kehadiran liang di dalam komposit. Bagaimanapun komposit sinteran gelombang mikro mempunyai ketumpatan yang lebih baik relatif kepada ketumpatan teori dan juga aktiviti penumpatannya.

Characterization of Microwave Sintered PM Fe-Cr-Y_2O_3 Composites

ABSTRACT

This research is focused on assessing the feasibility of the innovative microwave sintering technology for fabricating iron – chromium composites via powder metallurgy route. The microwave sintered composites were compared with their conventionally sintered counterparts in terms of physical and mechanical properties (micro Vickers hardness and compressive strength). Microwave sintering is proved feasible to consolidate the composite. The result also revealed that the microwave sintered iron chromium composites possess improved density, micro hardness and compressive strength compared to the conventionally sintered composites. Process evaluation also revealed that microwave assisted sintering can lead to reduction of 70% of sintering time when compared to conventional sintering. Another aims of this research is to study the effect adding varying weight fraction of yttria reinforcement to the iron chromium composites. For this purpose iron-chromium composites is reinforced with 5, 10, 15 and 20 wt.% of the ceramic particulates. From the study it is observed that the mechanical properties of microwave sintered iron – chromium composites improved with the addition of 5 wt.% ceramic content. For the conventional sintered composites, highest micro hardness and compressive strength were obtained from the 10 wt.% reinforced composites.The highest hardness value is given by microwave sintered composite. Other physical properties such as density was decreased as the reinforcement content increased. This is due to the increasing presence of porosity in the composites. However, microwave sintered composites exhibit better density relative to theoretical density and densification behaviour.

CHAPTER 1

INTRODUCTION

1.1 Background

The need for new materials which able to match increasingly stringent engineering requirements has led to the development of material composites for many manufacturing and industrial applications such as aerospace and automotive. The effort to improve the properties of new and existing materials has been receiving attention across the globe for decades. An ability to tailor the properties of the materials to meet specific needs on an application lies in the benefits of composite materials (Ramakrishnan, 2009).

A composite material consists of two or more physically and/or chemically distinct, suitably arranged or distributed material with an interface separating them. It has characteristics that not depicted by any of the components in isolation. These specific characteristics, in fact are the purpose of combining the materials.

The combination of metal and ceramics in which the metal is continuous and one or more addition material is dispersed and non-continuous phase is termed as metal matrix composites (MMC). The development of MMC has been driven by the advancement in the aerospace, automotive; process engineering applications, biocompatible materials and many more due to the combinations of higher strength, toughness and ductility of metals with high hardness of ceramics reinforcements.

There are many manufacturing technologies available to be used to fabricate metal based materials. The trend of MMC production has been toward particulate reinforcement for ease of processing, reduced cost, enhanced strength and stiffness along with reasonable ductility.

Within a great number of processes which ever developed to produce the selectively particulate reinforced material, powder metallurgy (PM) is competitive because of the material and energy saving which are economical advantages for large production; and possibility of producing near net shape products of complex geometries (German, 1997) with good mechanical properties. This is practically economical for small components which ordinarily would require a prohibitive amount of machining and material waste (Upadhyaya and Upadhyaya, 2006). PM also enables the fabrication of materials having different constituents which yields unique property combination (Iftekhar, 2004).

PM route consists of mixing and blending to incorporate different raw powders, compaction and sintering. Among these steps, sintering the most important step due to its role to evolve microstructural features that govern the end properties (Tun, 2009). In conventional sintering, the term solid state sintering or solid phase sintering are used because the metal remains solid at the treatment temperature (Groover, 2004). Nearly all metals of technical importance react with gas of their surrounding atmosphere at room temperature, and the reaction is higher when treated at higher temperatures. In conventional sintering method the heating rate is limited by the capability of the machine to achieve fast heating rates. Prolong exposure at high temperature could expose the treated products to defects such as crack and micro structural coarsening. Padmavathi et al. (2006) suggested that the limitation of the prolonged high temperature sintering in conventional way can be overcome by applying faster heating rates and shorter soaking time during sintering. Novel technique such as microwave sintering has been adopted to achieve faster heating rates for sintering Fe based composites (Anklekar et al., 2001; Anklekar et al., 2005; Padmavathi et al., 2007; Padmavathi et al., 2008). Meng (2010) reported that microwave sintering (MS) technique improves the grain microstructure and homogeneity of Nd-Mg-Fe_3O_4 hydrogen storage alloy compared to the same alloy prepared by its conventionally sintered (CS) counterparts (Meng et al., 2010).

Wear is one of the major engineering issues that can be found in many industries. Among various study on wear resistance material, Al and Mg matrices have been widely investigated. On the other hand, Fe is the most abundant material on earth and widely used in many industries with a variety of commercially available steel grades but less investigated as matrix materials. The focus of cost reduction in producing composite systems has increased and leads to an interest in Fe matrix composites which is cheaper compared to Co, Ni and their alloys that are scarce and expensive (Pagounis and Lindroos, 1998). Fe and its alloys are used in engineering applications where enhanced properties are part of the requirement. To resist abrasive wear, Fe based alloys are usually used. On the other hand, Fe based composites reinforced with hard ceramic particulates are a new class of advanced materials proposed mainly as inexpensive wear resistance parts or as substitutes for the more expensive cemented carbides materials (Pagounis, 1996).

The incorporation of ceramic particulates to Fe matrices suggests improvement in certain material properties such as higher hardness, higher strength at elevated temperature and wear resistance compared to monolithic Fe. When the aim is to enhance the wear resistance, alumina (Al_2O_3) and yttria (Y_2O_3) are used on account of their hardness (Velasco et al., 2003). Significant improvement of dry sliding wear resistance of PM MMC of austenitic stainless steel matrix reinforced with yttria (Y_2O_3) with chromium diboride (B_2Cr) as the sintering activator (Vardavoulias et al., 1996). Lal et. al (1989) reported the densification of PM Y_2O_3 reinforced stainless steel compacts were more pronounced and mechanical properties were found optimum for phosphorus (P) contained composites compared to plain stainless steel compacts. Researches elsewhere also have shown that Yttria (Y_2O_3) is a good choice as reinforcement in other matrix metal such as magnesium (Mg). Y_2O_3 reinforced Mg alloy is found stronger than conventionally processed Mg alloys and most Mg matrix composites in compression. Y_2O_3 also exhibits excellent temperature creep properties (Debata and Upadhyaya, 2001).

In this research, Fe-Cr composites reinforced with Y_2O_3 particulates were fabricated via microwave assisted sintering technique. The mechanical properties were investigated by determining the micro hardness value and compressive strength. The microstructure was observed to determine the correlation between the varying content of reinforcement to the mechanical properties. Accordingly, the composites were benchmarked against composites sintered using the conventional crucible furnace.

1.2 Problem statement

Conventional sintering process consumes high amount of time and energy because of the slow heating rate and long sintering duration. This is not economical for large production. Prolonged sintering would expose samples to potential damages, cracks and oxidation. Conventional sintering at high temperatures would also resulted in micro structural coarsening (Padmavathi et al., 2008). One of the way to overcome such limitation is by applying faster heating rates and shorter soaking time during the consolidation process. Among the emerging technology for consolidating materials at high temperature is microwave sintering technique.

However, limited research has been conducted on metal based materials. Most of the work on heating and sintering was applied extensively for the processing of ceramic materials (Barnett et al., 2004; Brown et al., 2005; Garces et al., 2006). Only limited research was conducted on investigating interaction between microwave and metal based materials (in the Fe-based material, most existing works is on PM stainless steel composites). This is due to the fact that metals reflect microwave causing arching phenomenon during microwave heating, thus limiting the penetration of the microwave into the specimen and causing damage to the equipment. However, the arching phenomenon only applies to bulk metal and metallic material in powder or particle form can couple with microwave energy (Tun, 2009).

The research work on microwave sintered Fe-Cr matrix composites reinforced with Y_2O_3 is not well reported. An attempt was made in this study to produce Fe-Cr matrix composites reinforced with Y_2O_3 by microwave assisted powder metallurgy route as an alternative in choosing a hard and wear resistance material for engineering applications.

Cr is added to give better corrosion resistance and to increase bonding strength of Y_2O_3. Surface oxidation of Cr matrix component causes diffusion to ceramic components in the composite , and makes up a solid solution at contact areas, thus forming strong bonds between the grains (Saidatulakmar et al., 2010). The use of Cr as activator materials to improve wetting is well known in brazing. Steel with

Cr addition has exhibited the highest tensile and highest hardness value in PM steel (Shanmugasundaram and Chandramouli, 2009). Cr is a ferrite stabilizers, it is therefore ferrite phase will be stable even at high temperature. Cr also provides corrosion resistance in Fe based alloys (Lin et al., 2002).

Y_2O_3 particles are used as the reinforcement to increase the hardness of the Fe-Cr composite due to their unique properties; hard and thermally stable at elevated temperatures and high resistance to wear. They are important in engineering applications for example to fabricate high density Y_2O_3 items such as substrates crucibles and nozzles which is utilized for improving resistance to molten titanium and metals prepared by rapid solidification process (Macheli et al., 1992). Among various ceramic particulates, good densification and wear resistance of Y_2O_3 with Fe based matrix has been reported by Vardavoulias et al. (1996). Improvement on densification between sintering temperature of 1200°C to 1250°C for the composites is observed by Velasco et al. (1996).

Many existing works on microwave sintering of PM MMCs has exploited the advantage of fast heating rates of microwave to obtain composites with good properties or better than CS counterparts.

1.3 Research objectives

This research embarks two objectives which are:

i. To study the feasibility of microwave sintering technique in producing PM Fe-Cr composites as opposed to the conventional sintering method using crucible furnace; and

ii. To study the effect of Y_2O_3 reinforcement into Fe-Cr matrix on the physical and mechanical properties of the composite.

1.4 Scope of research

This research is focused on the PM process which is to investigate the influence of Y_2O_3 reinforced PM Fe-Cr composites prepared with the energy-efficient microwave sintering furnace on the physical and mechanical properties. This study was done based on the following aspect:

i. Specimens were produced by via PM process using microwave sintering and conventional sintering as the consolidation process.

ii. Consolidation process of the composites is solid state sintering at 1200°C.

iii. The composite samples were axially compacted at 750 MPa.

iv. The variable studied is weight percentage of Y_2O_3 reinforcement.

v. Characterization of physical properties was determined by Archimedes principle method, microscopic analysis, EDS and XRD.

vi. Characterization of mechanical properties was determined by micro Vickers hardness test and compression test.

1.5 Thesis outline

The thesis is presented in five chapters including introduction, literature review, methodology, results and discussion and finally conclusion. Chapter 1 consists of background of the study, research objectives and thesis outline. Chapter 2 consists of the literature review. The previous work done by other researchers regarding to the fabrication and characterization of iron based PM MMCs are reviewed and discussed. Chapter 3 describes the methodology used in this study. Chapter 4 presents the results and also the discussion on the research outcomes. Chapter 5 presents the conclusion and recommendation of the present work.

CHAPTER 2

LITERATURE REVIEW

2.1 Overview

A material system which consists of a mixture or combinations of two or more constituents that differ in form and chemical composition and which are essentially insoluble in each other is termed as composite material (Smith and Hashemi, 2004). The concept of composite materials is to combine different materials to produce a new material with performance which is unattainable by the individual constituents (Budhoo et al., 2012).

Most commonly, composite materials have a bulk phase, which is continuous which is called the matrix, and one dispersed, non-continuous phase called the reinforcement. One of the roles of matrix is to determine the operating temperature regime for the composite. The basic requirements for selecting reinforcements include: reinforcements usually are stronger and stiffer than the matrix; having a shape, size and surface character so as to promote effective mechanical coupling with the matrix; not interacting with the matrix and not being too difficult to handle under commercial conditions (Clyne, 2000; Goni et al., 2000).

MMC is the term used for describing the combination of metal and ceramics in which the metal is continuous to produce a new material having attractive engineering attributes of its own. MMC also being described as having a metal or alloy as the continuous phase, in which the reinforcing phase is distributed (Chawla