STUDY ON THE EFFECT OF ALUMINA REINFORCEMENTS ON Fe-Cr MATRIX COMPOSITE

SAIDATULAKMAR SHAMSUDDIN

UNIVERSITI SAINS MALAYSIA

2011

STUDY ON THE EFFECT OF ALUMINA REINFORCEMENTS ON Fe-Cr MATRIX COMPOSITE

by

SAIDATULAKMAR SHAMSUDDIN

Thesis submitted in fulfillment of the requirements for the degree of Doctor of Philosophy

UNIVERSITI SAINS MALAYSIA

JANUARY 2011

KAJIAN MENGENAI KESAN PENGUAT ALUMINA KE ATAS KOMPOSIT MATRIK Fe-Cr

oleh

SAIDATULAKMAR SHAMSUDDIN

Tesis yang diserahkan untuk memenuhi keperluan bagi
Ijazah Doktor Falsafah

UNIVERSITI SAINS MALAYSIA

JANUARI 2011

ACKNOWLEDGEMENTS

In the name of Allah swt, the most Gracious and the most Merciful and may His blessing be upon the prophet Muhammad saw. Alhamdullillah, my greatest thanks to Allah swt.

My sincere thanks to my main supervisor Prof. Dr. Haji Zainal Ariffin Ahmad, who has done his best in encouraging, supporting, understanding and sharing his knowledge and remarkable experience to enable this study to be completed. My appreciation is extended to Dr. Zuhailawati Hussain and Prof. Dr. Shamsul Baharin Jamaludin for their valuable discussion, encouragement, helpful and guidance in supporting throughout the whole study.

Acknowledgement to all my friends, staffs and students of Universiti Sains Malaysia and Universiti Malaysia Perlis who have been cooperative by sharing their knowledge and technical support during this study. Thanks to Universiti Teknologi MARA for given me the opportunity to further study with the scholarship and study leaves.

To my dearest husband and son thank you for loving, understanding and supporting me in completing this thesis and for making my life more meaningful. Specially thanks to my dearest mother who always pray for me with endless love. This thesis is dedicated to my beloved almarhum father.

Thank you to all who have contributed in the success of this study. May Allah bless be with all of us forever. Amin!

TABLE OF CONTENTS

CHAPTER 2: LITERATURE REVIEW 11

LIST OF TABLES PAGE

LIST OF FIGURES **PAGE**

LIST OF ABBREVIATIONS

δ	:	Delta
°C	:	Degree Celsius
°F	:	Degree Farrenheit
A	:	Area
Al	:	Aluminium
$Al(NO_3)_3$	:	Aluminium Nitrate
Al_2O_3	:	Alumina
$Al_6Si_2O_{13}$	:	Mullite (a compound of aluminum, silicon, and oxygen)
$AlCr_2$	:	Aluminium Chromium alloy
AP	:	Abrasive Particles
Ar	:	Argon
ASTM	:	American Society for Testing and Materials
B_2Cr	:	Barium Chromate
BCC	:	Body Centered Cubic
BN	:	Barium Nitrate
C	:	Carbon
CERMET	:	Composite materials composed of Ceramic and Metallic
CH_2	:	Dichloromethane (methylene chloride)
$CH_3(CH_2)_{16}COOH$	:	Stearic Acid
CIP	:	Cold Isostatic Pressing

CMC : Ceramic Matrix Composite

Co : Cobalt

Cr : Chromium

Cr_2O_3 : Chromium (III) Oxide

Cr_2Ti : Chromium Titanium alloy

Cr_3C_2 : Chromium Carbide

CTE : Coefficient of Thermal Expansion

Cu : Copper

D : Diffusion

EDX : Energy Dispersive X-ray

F : Force

FCC : Face Centered Cubic

Fe : Iron

Fe_2O_3 : Iron (III) oxide

Fe_3Al : Iron Aluminide

FeCrAl : Iron Chromium Aluminium alloy

$FeSO_4$: Iron (II) Sulfate/Ferrous Sulfate

GB : Grain Boundary

gf : Gram Force

H_2 : Hidrogen

H_2O : Water

HCP : Hexagonal Close Packed

HIP : Hot Isostatic Pressing

HP : Hard Phases

HSS : High Speed Steel

HV : Hardness Vickers

ICDD : International Centre for Diffraction Data

ISO : International Standard Organisation

kgf : Kilogram Force

KML : Komposit Matrik Logam

L : Length

Li : Lithium

$M_{23}C_6$: Chromium Rich Alloy Carbide (Final Precipitation)

M_3C : Chromium Rich Alloy Carbide (Initial Precipitation)

M_7C_3 : Chromium Rich Alloy Carbide (Second Precipitation)

Mg : Magnesium

min : Minute

mm : Millimeter

MMC : Metal Matrix Composite

MPa : Mega Pascal

MPIF : Metal Powder Industries Federation

MS : Metalurgi Serbuk

MW : Molecular Weight

N_2	:	Nitrogen
NbC	:	Niobium Carbide
Ni	:	Nickel
Ni_3Fe	:	Nickel Ferrite
P	:	Pressure
Pb	:	Plumbum
PM	:	Powder Metallurgy
PMC	:	Polymer Matrix Composite
PVA	:	Polyvinyl Alcohol
Q	:	Activation Energy
R	:	Gas Constant
SEM	:	Scanning Electron Microscopy
Si	:	Silicon
SiC	:	Silicon Carbide
SiO_2	:	Silicon Dioxide
T	:	Temperature
t	:	Time
Ti	:	Titanium
Ti_2O_3	:	Titanium (III) oxide
TiAl	:	Titanium Aluminide
TiB_2	:	Titanium Diboride

TiC : Titanium Carbide

TiCN : Titanium CarboNitride

TiN : Titanium Nitrate

V : Vanadium

V_2O_3 : Vanadium Trioxide

VC : Vanadium Carbide

W : Weight

WCI : White Cast Iron

wt% : Weight Percentage

XRD : X-ray Diffraction

Y : Young Modulus

Y_2O_3 : Yttria

γ : Gamma

μ : Friction Coefficient

μm : Microns

LIST OF PUBLICATIONS

1. Saidatulakmar Shamsuddin, Shamsul Baharin Jamaludin, Zuhailawati Hussain, Zainal Ariffin Ahmad. (29 Mei – 1 Jun, 2007) Influence of Binder in Iron Matrix Composites. In Proceeding of the International Conference on Advance Materials and Nanotechnology. Langkawi, Malaysia.

2. Saidatulakmar Shamsuddin, Shamsul Baharin Jamaludin, Zuhailawati Hussain, Zainal Ariffin Ahmad. (26-27 Nov, 2007) Compaction study of particulate iron-chromium matrix composite reinforced with alumina. In Proceeding of the National Metallurgical Conference 2007. Johor Bahru, Johor, Malaysia.

3. Saidatulakmar Shamsuddin, Shamsul Baharin Jamaludin, Zuhailawati Hussain, Zainal Ariffin Ahmad. (27-29 Nov, 2007) Preliminary study of iron-chromium matrix composite reinforced with alumina. In Proceeding of the 23rd Regional Conference on Solid State Science and Technology 2007. Johor Bahru, Johor, Malaysia.

4. Saidatulakmar Shamsuddin, Shamsul Baharin Jamaludin, Zuhailawati Hussain, Zainal Ariffin Ahmad. (10-12 Dec, 2007) Microstructure and Hardness Properties of Fe-Cr-Al_2O_3 Composites. In Proceeding of the 6th Asean Microscopy Conference 2007. Cherating, Pahang, Malaysia.

5. Saidatulakmar Shamsuddin, Shamsul Baharin Jamaludin, Zuhailawati Hussain, Zainal Ariffin Ahmad. (2007) Microstructure and Hardness Properties of Fe-Cr-Al_2O_3 Composites. Malaysian Journal of Microscopy 4, 118-123.

6. Saidatulakmar Shamsuddin, Shamsul Baharin Jamaludin, Zuhailawati Hussain, Zainal Ariffin Ahmad. (2008) Characterization of Fe-Cr-Al_2O_3 Composites Fabricated by Powder Metallurgy Method with Varying Weight Percentage of Alumina. Journal of Physical Science. 19(1), 89-95.

7. Saidatulakmar Shamsuddin, Shamsul Baharin Jamaludin, Zuhailawati Hussain, Zainal Ariffin Ahmad. (30 Nov – 2 Dis, 2008) The Characteristics of vacuum sintered Fe-Cr-Al_2O_3 Composites. In Proceeding of the 24th Regional Conference on Solid State Science and Technology 2008. Port Dickson, Negeri Sembilan, Malaysia.

8. Saidatulakmar Shamsuddin, Shamsul Baharin Jamaludin, Zuhailawati Hussain, Zainal Ariffin Ahmad. (2-4 Dis, 2008) Effect of Particle Size on Fe-Cr-Al_2O_3

Metal Matrix Composite. In Proceeding of Malaysian Metallurgical Conference 2008. Bangi, Selangor, Malaysia.

9. Saidatulakmar Shamsuddin, Shamsul Baharin Jamaludin, Zuhailawati Hussain, Zainal Ariffin Ahmad. (2009) Preliminary study of iron-chromium matrix composite reinforced with alumina. Solid State Science and Technology Letters. 16(1 & 2), 38-43.

10. Saidatulakmar Shamsuddin, Shamsul Baharin Jamaludin, Zuhailawati Hussain, Zainal Ariffin Ahmad. (2010) Influence of Binder in Iron Matrix Composites. American Institute of Physics Conference Proceedings. 1217, 419-423.

11. Saidatulakmar Shamsuddin, Shamsul Baharin Jamaludin, Zuhailawati Hussain, Zainal Ariffin Ahmad. (9-10 June, 2010) Heating Rate Effects on Properties of Powder Metallurgy Fe-Cr-Al_2O_3 Composites. In Proceeding of the International Conference on X-rays & Related Techniques in Research & Industry. Langkawi, Malaysia.

12. Saidatulakmar Shamsuddin, Shamsul Baharin Jamaludin, Zuhailawati Hussain, Zainal Ariffin Ahmad. (2010) The effects of Al_2O_3 amount on the microstructure and properties of Fe-Cr matrix composites. Metallurgical and Materials Transactions A. Vol 41A, 3452-3457.

13. Saidatulakmar Shamsuddin, Shamsul Baharin Jamaludin, Zuhailawati Hussain, Zainal Ariffin Ahmad. (2010) Heating Rate Effects on Properties of Powder Metallurgy Fe-Cr-Al_2O_3 Composites. Advanced Materials Research Journal (Under Consideration).

14. Saidatulakmar Shamsuddin, Shamsul Baharin Jamaludin, Zuhailawati Hussain, Zainal Ariffin Ahmad. (2010) Optimization of processing parameters on the properties of Fe-Cr-Al_2O_3 composites (Under Consideration).

KAJIAN MENGENAI KESAN PENGUAT ALUMINA KEATAS KOMPOSIT MATRIK Fe-Cr

ABSTRAK

Komposit Matrik Logam (KML) telah menunjukkan kemajuan yang berkesan daripada aspek intelek dalam dunia sains ke bidang teknologi dan perdagangan sejak tiga dekad yang lepas. Dalam kajian ini, percubaan untuk memfabrikasi KML baru iaitu matrik Fe-Cr diperkuat dengan partikel Al_2O_3 melalui kaedah Metalurgi Serbuk (MS) konvensional telah berjaya diperolehi. Selepas proses persinteran tiada fasa lain yang terbentuk selain daripada komposisi asal bahan komposit. Puncak-puncak XRD telah dikenalpasti sebagai fasa-fasa Fe, Cr dan Al_2O_3 serta analisa EDX mengecam komposit ini terdiri daripada Fe, Cr, Al dan O. Berdasarkan prinsip Archimedes, nilai ketumpatan dan liang dihitung, Mikro kekerasan Vickers, ciri-ciri rintangan haus, kelakuan kekuatan mampatan dan pemerhatian SEM dijalankan untuk menentu keadaan optima. Penggunaan pengikat sebanyak 2 bt% asid stearik, tempoh percampuran selama 30 minit, tekanan mampatan ekapaksi sebanyak 750 MPa dan kadar pemanasan 10°C/min dalam keadaan vakum menghasilkan keadaan optima untuk memfabrikasi komposit ini. Suhu persinteran yang tinggi menghasilkan ciri-ciri ketumpatan dan mekanikal yang lebih baik. Nilai kekuatan mampatan bagi sampel tanpa penguat, sampel dengan 5 bt% penguat dan sampel dengan 25 bt% penguat adalah 628 MPa, 648 MPa and 221 MPa masing-masing. Kandungan sebanyak 5 bt% partikel Al_2O_3 menunjukkan mikrostruktur yang sekata, ketumpatan yang lebih baik dan kekuatan mampatan yang lebih kuat berbanding dengan sampel tanpa partikel penguat dan sampel yang mengandungi lebih banyak partikel Al_2O_3. Walaubagaimanapun, bacaan optima pada Mikro kekerasan

Vickers dan rintangan haus ditemui pada komposit yang diperkuat dengan 20 bt% partikel Al_2O_3. Peningkatan kandungan partikel Al_2O_3 sebanyak 25 bt% mengakibatkan pengurangan dalam sifat mekanikal disebabkan penggumpalan partikel penguat dalam matrik mengurangkan interaksi antara matrik dan penguat. Nilai koefisien haus bagi sampel tanpa penguat, sampel dengan 20 bt% penguat dan sampel dengan 25 bt% penguat adalah 3.28×10^{-11}, 2.43×10^{-11} and 2.77×10^{-11} masing-masing. Menggunakan penguat partikel Al_2O_3 bersaiz lebih daripada 13 μm di dalam matrik Fe-Cr menyebabkan mikrostruktur dan ciri-ciri mekanikal komposit merosot. Sampel yang diperkuat dengan partikel Al_2O_3 bersaiz 13 μm menghasilkan nilai koefisien haus 2.46×10^{-11} dan kekuatan mampatannya ialah 278 MPa. Bagi sampel yang diperkuat dengan partikel Al_2O_3 bersaiz 64 μm mempunyai keofisien haus 5.09×10^{-11} dan kekuatan mampatan 81 MPa.

STUDY ON THE EFFECT OF ALUMINA REINFORCEMENET ON Fe-Cr MATRIX COMPOSITE

ABSTRACT

Metal Matrix Composites (MMCs) have shown significance improvement from intellectual interest in scientific world to the technological and commercial applications for over the past three decades. In the present study, an attempt to fabricate a new MMC of Fe-Cr matrix composites reinforced with Al_2O_3 particles through conventional Powder Metallurgy (PM) method was successfully obtained. No phases other than the constituent were developed in the composites after sintering. The peaks of the XRD patterns have been identified as belonging to the phases of the Fe, Cr and Al_2O_3 and the EDX analysis of the composites confirm the existence of Fe, Cr, Al and O. Based on the Archimedes' principle, the density and porosity are computed. Vickers micro hardness, wear resistance characteristics, compressive strength behavior and SEM evaluations were established to determine the optimum condition. Using a binder of 2 wt% stearic acid, mixing duration of 30 minutes, uni-axial compaction pressure of 750 MPa and heating rate of 10°C/min in vacuum condition was found as an optimal condition to fabricate this composite. Higher sintering temperature promotes better densification and mechanical properties. The compressive strength value for unreinforced sample, sample with 5 wt% Al_2O_3 and sample with 25 wt% Al_2O_3 are 628 MPa, 648 MPa and 221 MPa respectively. The amount of 5 wt% Al_2O_3 particles revealed homogeneous microstructure, better densification and stronger compressive strength compared to the unreinforced sample and those samples with larger content of Al_2O_3 particles. Nevertheless, optimum reading in Vickers micro hardness and wear resistance were

found in those composites reinforced with 20 wt% Al_2O_3 particles. Increasing to 25 wt% of Al_2O_3 particles results in a decrease in mechanical properties due to agglomeration of the reinforcement particles in the matrix that lower the interaction between matrix and reinforcement. The wear coefficient value for unreinforced sample, sample with 20 wt% Al_2O_3 and sample with 25 wt% Al_2O_3 are 3.28×10^{-11}, 2.43×10^{-11} and 2.77×10^{-11} respectively. Reinforcing the Fe-Cr matrix with Al_2O_3 particles bigger than 13 μm deteriorated the microstructure and mechanical properties of the composites. The wear coefficient for sample reinforced with 13 μm Al_2O_3 particles is 2.46×10^{-11} with compressive strength of 278 MPa. Meanwhile for sample reinforced with 64 μm Al_2O_3 particles its wear coefficient is 5.09×10^{-11} and compressive strength is 81 MPa.

CHAPTER 1

INTRODUCTION

1.1. Introduction

As fast progress of modern high technology develops, more demands on new materials properties with various special functions are raised in the development of society and the quality of human life. The advancement of many fields of technology is conditioned by the acquisition of materials, with ever increasing performance. Therefore an effort to improve the properties of new and existing materials has been receiving attention across the globe for a number of years. An ability to tailor the properties of the materials to meet specific needs of an application lies in benefit of composite materials.

A metal matrix composites (MMC) combines into a single material a metallic base with a reinforcing constituent, which is usually non-metallic and is commonly a ceramic (Clyne, 2000). The development of MMC has been driven by the advancement in the aerospace, automotive, process engineering applications, biocompatible materials, pharmaceutical and food production due to the combinations of higher strength, toughness and ductility of metals with high hardness of ceramic reinforcements.

By definition, MMCs are produced by means of processes other than conventional metal alloying. Processes commonly used include powder metallurgy, diffusion bonding, liquid phase sintering, squeeze-infiltration, stir-casting and in situ technique (Clyne, 2000). The trend of MMC production has been toward particulate reinforcements for ease of processing, reduced cost, isotropic properties, enhanced strength and stiffness along with reasonable ductility. Within a great number of processes which were developed to produce the selectively particulate reinforced materials, powder metallurgy (PM) processing is competitive because of its low cost, ability to produce composites with high volume fraction, high productivity and possibility to fabricate components with complex geometry (German, 1994). Complex shaped machine elements like gears, bearings, connecting rods, cams, etc., made by PM compete favorably in terms of properties and cost (Ibrahim, et al., 1991). In the economy aspect PM method is suited in manufacturing large series of small and relatively complex shapes components with smaller materials consumption (Upadhyaya, 2000).

1.2. Problem Statement

Wear is one of the major engineering issues that can be found in many industries as well as ordinary life. Traditionally, wear resistance materials that was widely employed in various industries were made from solidification of castings in sand mould of Co, Ni or Fe alloy. The crystallographic structure and hardness of these materials depends on alloying and heat treatment. In the mining, cement industry and road construction, white cast iron (WCI) of hypo-to-hypereutectic composition consist of hard phases (HP) of carbide or borides embedded in a hardened metal matrix are the workhorses of wear protection. The problems with this material are solidification of castings (develop from the melt) which depends on phase equilibrium, decreases in size of HP due to sliding abrasive particles (AP) and linkage of HP which results in a brittle skeleton that promotes crack extension (Berns, 2003) and (Berns & Wewers, 2001). PM of MMC has appeared as a bright option for wear applications which allow the selection of suitable materials, lower cost, eased of processing and possible property improvement.

Among various study on wear resistance materials, Al and Mg matrices have been widely investigated. On the other hand, Fe the most abundant element on the earth and widely used materials with a variety of commercially available steel grades were less investigated as matrix materials. Until recently, its unique properties have attracted attention. A sharper focus on cost reduction in producing advanced composites systems has increased and leads to an interest in Fe matrix composite which is cheaper compared to Co, Ni and their alloys that are scarce, expensive and their dust is especially harmful (Pagounis & Lindroos, 1998) and (Gordo et al., 2005). Fe and its alloys are used in engineering applications with excellent properties such as high stiffness, high strength and toughness, good machinability and weldability with a possibility to be hardened by heat treatment without changing the nature of the reinforcing phase and thus reduce the quantity of hard phase present in the material, subsequent benefits in terms of cost and processing and ability to wet most transition metal carbides.

To resist abrasive wear, Fe based alloys are usually used (Al-Rubaie, 2000). According to Pagounis et al. (1996), Fe based composites reinforced with hard ceramic particles are advanced materials proposed mainly as inexpensive wear resistance parts or as substitutes for the more expensive cemented carbide. Bautista et al. (2007) described that powder metallurgical ferritic stainless steels have proven to be an adequate material for the manufacturing of some components exposed to elevated temperatures. The incorporation of particulate ceramics to Fe matrices significantly improve certain material properties, it offers higher hardness, higher strength at elevated temperature and wear resistance compared to monolithic Fe.

Many investigations on Fe matrix composites have been focused on Fe alloy or steel as the matrix materials. Pagounis & Lindroos (1998) compared the effect of Al_2O_3, Cr_3C_2, TiC and TiN as the particulate reinforcement in the steel matrix composites and concluded that TiC reinforcement has the best bonding behavior with the steel matrix due to the formation of a thin Fe, Cr rich layer at the interface. Based on this study, many other scholars have published work on the effect of steel-TiC MMC, e.g, Li et al. (2009), Akhtar & Guo (2008) and Degnan et al. (2001). A number of studies on alloyed Fe MMC has been reported by Pagounis et al. (1996), Pagounis & Lindroos (1997), Pagounis & Lindroos (1998). Sakamoto et al. (2001) and Lu et al. (2003) have investigated on high Cr cast Fe. Report on stainless steels MMC can be reviewed from article's of Abenojar et al. (2003) and articles based on high speed steels MMC has been reported by Gordo et al. (2000), Velasco et al. (2001), Velasco et al. (2002) and Ruiz-Navas et al. (2003).

When the goal is to improve the wear resistance, Al_2O_3 and Y_2O_3 are used on account of their hardness (Velasco et al., 2003). Mukherjee & Upadhyaya (1985) evaluated the mechanical behavior of sintered ferritic stainless steel-Al_2O_3 particulate composites containing ternary additions and the results showed that the yield and tensile strengths are high for Al_2O_3 containing composites. Vardavoulias et al. (1996) reported the significant improvement of dry sliding wear resistance of PM MMC of austenitic stainless steels matrix reinforced with Al_2O_3 particles with B_2Cr as the sintering activator. Lemster et al. (2005) produced MMC of Al_2O_3 and steel for use in food industry applications where high wear resistance in combination with food-safe materials to avoid contamination is a key requirement. Stempflé et al. (2008) studied on the influence of metallic nanoparticle content on wear behavior of $Fe_{0.5}$-$Cr_{0.5}$-Al_2O_3 nanocomposites rubbing on Ti-6Al-4V in fretting.

Some reported studies on Fe-Al_2O_3 composites fabricated by other method and for other purposes are as follows; Mukherjee & Bandyopadhyay (1995) and Subramaniam et al. (1997) described the way of processing the composites of Fe_3Al-Al_2O_3 for high-temperature structural applications. Santos et al. (1998) investigated the structural and magnetic properties Fe-Al_2O_3 obtained by sol-gel method. Synthesis and characterization of Fe-Al_2O_3 composites using arc-melting compacted pellets has been published by Paesano et al. (2003). Travitzky et al., (2003) reported on rapid synthesis of Al_2O_3 reinforced Fe-Cr-Ni composites. Mechanical properties of nanocrystalline Fe-Pb and Fe-Al_2O_3 was reported by Gurudu et al (2006). Karayannis & Moutsatsou (2006) produced Ni_3Fe-Al_2O_3 from ferrous scrap by a hydrometallurgical process. Li et al. (2007) investigated the Al_2O_3-FeCrAl composites as a candidate for high temperature applications. Nowacki & Rylska (2001) studied on low carbon or carbon free Fe sinters with addition of Al and Cr as heat resistant materials.

However, Fe-Cr matrix composite reinforced with Al_2O_3 are still not established. An attempt was made in this study to produce Fe-Cr matrix composites reinforced with Al_2O_3 by PM as an alternative in choosing a wear resistance material for engineering applications.

Cr is added to give better corrosion resistance and to increase bonding strength of Al_2O_3. Surface oxidation metallic matrix components of Cr causes diffusion of this oxide to Al_2O_3, and makes up a solid solution at contact areas, thus forming strong bonds between the grains (Uygur, 1997, Ceccone et al., 1996, Zhang et al., 2002). The use of Ti or Cr, or combinations thereof, as activator materials to improve wetting is well known in brazing. Their effect to enhance wetting of alumina has been studied, for example, in copper and nickel-base alloys used in brazing and soldering technology (Lemster et al., 2005). The steel with Cr addition has exhibited the highest tensile strength and highest hardness value in PM steel (Shanmugasundaram & Chandramouli, 2009). Cr is a ferrite stabilizers, it is therefore ferrite phase will be stable even at high temperature (Das et al., 2008). Cr provides corrosion resistance in Fe base alloy (Fontana, 1987).

Al_2O_3 particle is used as the reinforcement to increase friction coefficient (μ) due to their unique properties; hard and thermally stable at high temperatures, high strength with high resistance to wear and corrosion. They are important in engineering applications, such as grinding media, gas turbines, engines and solid fuel cells (Pagounis & Lindroos, 1998) and (Lu, 2006). Among various ceramic particulates, good wettability of Al_2O_3 with Fe based matrix has been reported by Pagounis & Lindroos (1998), Vardavoulias et al. (1996) and Murkherjee & Upadhyaya (1985).

1.3. Research Objective

The basic research goal focuses on developing an understanding of producing particulate Fe-Cr matrix composites reinforced with Al_2O_3 composites by conventional PM method. The objectives emphasize on:

(i) To produce homogeneous Fe-Cr matrix composites reinforced with Al_2O_3.

(ii) To find the most suitable binder to fabricate the composites.

(iii) To find optimum parameters to fabricate the composites by conventional PM route: mixing, pressing and sintering.

(iv) To study the effect of Al_2O_3 weight percentages, sizes and morphologies on the physical and mechanical properties of the composites.

(v) To identify the influence of each parameter in correlation of the processing; microstructure, physical and mechanical properties of the composites.

(vi) To evaluate wear resistance and compressive strength of the composites.

1.4. Scope of Study

In general the study is divided into two parts. In the first part, the raw materials of Fe, Cr and Al_2O_3 are evaluated to determine their particle size, density, morphology and phases. The second part of this study is designed to obtain the optimum paramater of processing in powder metallurgy route; mixing, compacting and sintering. Furthermore the optimum amount of Al_2O_3 particles, the optimum size of Al_2O_3 particles and the optimum morphology of Al_2O_3 particles are also investigated.

The particle size distributions of the starting powders were analyzed using a laser diffraction analyzer HELOS Particle Size Analysis from Sympatec Gmbh System-Partikel-Technik. The particle size distribution of the powder is determined based on the Fraunhofer theory. Micromeritics AccuPyc 1330 Pycnometer Density was used to measure true density of the powders. Starting powders morphologies were analyzed using Scanning Electron Microscope JSM-6460LA JEOL. The purpose is to observe the changes of powders particle shapes due to the process of obtaining the composite. XRD was carried on a Bruker AXS D8 Advance with copper K_α radiation for phase analysis.

To achieve successful results in compaction and sintering, the metallic powders must be thoroughly homogenized beforehand. Binders are added in the mixing powder to reduce friction between particles, improved flow of the powder metals into the dies and at the die wall during compaction; and longer die life (Liu, et. al., 1994). There are four types of binders evaluated in this study; stearic acid, gummi arabisch, polyvinyl alcohol 15000 MW and polyvinyl alcohol 22000 MW.

The mixing time and the intensity of mixing powder and lubricant is an important factor because it will affect the properties of the mixture such as flow and apparent density, moreover it controls the final distribution of reinforcement particle in green compacts after compaction, which strongly affects the mechanical properties of powder metallurgy materials produced (Lenel, 1980). A range of eight mixing duration from 5 to 360 minutes are studied.

The purposes of compaction are to obtain the required shape, density and particle to particle contact and to make the part strong enough to be processed further. As pressure increases, the particles are plastically deformed, causing interparticle contact area to increase and additional particles to make contact. This is accompanied by a further reduction in pore volume (Groover, 2002). This study focused on uni axial compaction pressure in a range of 250 until 875 MPa studied.

Sintering is a heat treatment operation performed on the compact to bond its metallic particles, thereby increasing strength and hardness. Sintering of green compacts made of steel powder mixture must be performed in vacuum or in a reducing atmosphere because water-atomised steel powder particles are oxidized on the surface and in this way some deoxidation reaction can occur during sintering (Sustarsic, 2003). To study the effect of heating rate during vacuum sintering to fabricate the composites a range of 3 until 15°C/min heating rate are investigated. To determine the optimum sintering temperature a range of eight temperatures (1050 to 1400)°C are used.

The optimum amount of reinforcement are selected from a range of 5 to 25 wt%, meanwhile the effect of reinforcement particle size are chosen from 13 to 23 μm and the morphologies are selected between the irregular and nodular shape.

The optimum conditions are due to the optimum physical and mechanical properties achieved by the composite. The relative density and total porosity of the composites were calculated using the rule of mixture based on the bulk density and apparent porosity from Archimedean principle. The microstructures of the composites were examined by scanning electron microscopy and the phase analysis was carried out by X-ray Diffraction. Micro-hardness data were obtained using a Mitutoyo Hardness Testing Machine. The pin on disk wear resistance test was employed to determine the wear properties of the composites and the compressive strength test were used to evaluate the strength of the composites.

CHAPTER 2

LITERATURE REVIEW

A composite material is a materials system composed of a mixture or combinations of two or more micro or macro constituents that differ in form and chemical composition and which are essentially insoluble in each other (Smith & Hashemi, 2004). The concept of composite materials is to combine different materials to produce a new material with performance unattainable by the individual constituents. In nature, examples abound: a coconut palm leaf, wood, bone, etc.

Most commonly, composite materials have a bulk phase, which is continuous, called the matrix, and one dispersed, non-continuous, phase called the reinforcement. The roles of a matrix are protection of the reinforcement against mechanical damage, maintenance of reinforcement position, resistance from corrosion and degradation and determine the operating temperature regime for the composite. Several basic requirements for the reinforcement are; the reinforcement for most composites are stronger and stiffer than the matrix, having a size, shape 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).

Composites can be classified into three categories; Polymer Matrix Composites (PMCs), Ceramic Matrix Composites (CMCs) and Metal Matrix Composites (MMCs) based on the type of matrix materials.

PMCs are used in a variety of applications; load bearing structures, tubing, electronic packaging, automobile and aircraft components. They are comparatively inexpensive but have a number of limiting features including poor bonding to fibers, low maximum working temperature, high thermal expansion coefficient and sensitivity to moisture (Rawlings & Matthews, 1994).

CMCs have been developed to overcome the intrinsic brittleness and lack of reliability of monolithic ceramics, with a view to introduce ceramics in structural parts used in severe environments, such as rocket and jet engines, gas turbines for power plants, heat shields for space vehicles, fusion reactor first wall, aircraft brakes, heat furnaces, etc (Clyne, 2000) and (Mazdiyasni, 1990).

2.1 Metal Matrix Composites

If the metal has a volume fraction greater than 50%, the composite is called a MMC and if the ceramic has a volume fraction greater than 50% it is called a CERMET. MMCs offer engineers and designers new freedom in designing high performance parts and components. MMCs are being proposed for increased use in structures that require high specific modulus, strength and thermal stability.

There are generally seven ways in which MMCs have advantage over metals:

i. Higher strength and higher stiffness to density ratio.
ii. Consolidating many parts in an assembly into one part. Part consolidation reduces part count, fasteners and assembly time.

iii. Tailor the matrix and reinforcement to meet stiffness, strength and manufacturing requirements.

iv. Better fatique resistance.

v. Better elevated temperature properties, higher strength and lower creep rate.

vi. Lower coefficients of thermal expansion.

vii. Better wear resistance.

(www.macinedesign.com, www.smcpowdermetallurgy.com)

2.2 Classification of MMCs

MMCs are classified into continuous or discontinuous reinforced composites.

2.2.1 Continuous MMCs

A fibrous reinforcement is characterized by its length being much greater than its cross-sectional dimension known as an aspect ratio. The high performance alloys contain continuous fibers that all lie in the same direction and have extremely good specific properties in the direction of the fibers, however they are more expensive to fabricate and limited to highly specialized applications (Clyne, 2000).

2.2.2 Discontinuous MMCs

SiC and Al_2O_3 in a form of particles, platelets and whiskers are generally used in discontinuous reinforced composites, which can be fabricated using conventional techniques such as powder metallurgy and casting, with or without secondary processing such as extrusion and rolling. The arrangement of the particulate reinforcement may be random or with a preferred orientation. In the majority of particulate reinforced composites the orientation of the particles is considered, for practical purposes, to be random. It exhibits relatively isotropic mechanical properties compared to continuous reinforced composites and significantly improved mechanical properties compared to the unreinforced matrix (Rawlings & Matthews, 1994), (Lucchini, et al., 2003) and (Zhu, & Kishway, 2005).

2.3 Methods of Producing MMCs

There are four major types of methods to produce MMCs:

(i) Solid State Processing
(ii) Liquid State Processing
(iii) Vapor State Processing
(iv) In Situ Processing

This study focused on fabricating the composite using a solid state processing through PM method.

2.4 Powder Metallurgy

PM is especially suited for the production of discontinuous fiber, whisker or particulate reinforced metals. In PM, the matrix metal and reinforcement powders are;

i. mixed,
ii. compacted and
iii. sintered to form the composites.

The PM technique in the synthesis of MMCs found initial use because of the difficulty in wetting ceramic particles with molten metal. It is an important processing technique for MMCs that tend to offer homogeneity of both composition and microstructure of the matrix materials together with more control over the reinforcement distribution. This uniformity not only improves the structural properties but also the reproducibility level in the properties.

There are several advantages of PM;

i. Opportunity to mix various powders and mixture of two metals that are substantially insoluble in each other and therefore cannot be produced by melting and casting (i.e. SiC reinforcing Ti alloys).
ii. Particles or whiskers as reinforcement can be obtained easily by PM than by other alternative routes; moreover, particles are cheaper than continuous fibers of the same composition.
iii. Low manufacturing temperature that avoids strong interfacial reaction, minimizing the undesired reactions between the matrix and the reinforcement.
iv. Smaller material consumption.
v. Suited to the economic production of complicated shapes because of its ability to produce the required shape and dimensions without recourse to machining. This not only contributes largely to the amount of scrap, but also saves the cost of machining which can be very high for complicated shapes.
vi. Avoiding the limitations and defects of casting techniques and in producing materials with superior properties inherited from the powder characteristics usually related to rapid solidification.

(Dowson, 1990), (Zhang, et al., 2003), (Ejiofor, et al., 1997) and (Torralba, et al., 2003).

In production of PM components, the properties achieved are to a considerable extent a function of the properties of the starting materials, the individual powders and the powder mix (Dowson, 1990). The behavior of metal powders during processing largely depends upon the particle size, particle size distribution, particle shape and structure of the powder, but data on these properties cannot be translated directly into values characterizing processing behavior. For this reason a number of specific tests for processing behaviour have been developed (Lenel, 1980).

(a) Sampling of Powders

Standard method for sampling finished lots of metal can be found in ASTM Standard B 215 and in MPIF Standard 1. Samples from different layers (top, center, and bottom) of drums filled with powder are taken and blended. From the resulting blend a sample of proper size for the desired test is obtained using a sample splitter (Lenel, 1980).

(b) Chemical Test

The basic chemical composition has a role in determining the mechanical properties, corrosion resistance, electrical and magnetic properties, etc (Dowson, 1990). The two tests for the chemical analysis of metal powders which have been standardized by ASTM and MPIF are: ASTM standard E 159, MPIF standard 2 for the hydrogen loss of copper, tungsten and iron powder and ASTM standard E 194, MPIF standard 6 for acid-insoluble content of copper and iron powder (Lenel, 1980).

(c) Particle Characteristics

(i) Particle Size

For spherical particles, the diameter of the sphere defines the particle size. For irregular particles, particle size will depend upon the method by which this size is measured. Size is usually expressed in microns (μm) (Lenel, 1980).

(ii) Particle Size Distribution

Particle size distributions may be presented in the form of tables or graphs. Distributions by number percent frequency are directly obtained in microscopic sizing, while sieving would yield distribution by weight percent frequency (Lenel, 1980).

(iii)Particle shape and Structure

Particle shape has a major influence on the packing density of powders. Density can be improved by mixing different sizes of particles. Addition of irregular particles may not harm packing density for spheres, yet may improve compact strength by providing more interparticle friction (German, 1989).

ISO standard 3252 illustrates a number of commonly occurring shapes under the names;

Acicular:	needle shape
Angular:	sharply edged or roughly polyhedral
Dendritic:	branched shape
Fibrous:	regularly or irregularly shaped threads

Flaky: plate like
Granular: approximately equidimensional but of irregular shape
Irregular: lacking any symmetry
Nodular: rounded irregular shape
Spheroidal: roughly spherical

Particle structure affect homogenization, hard particles are difficult to deform during the compaction process and can, therefore, result in different microstructures than would be formed using soft, annealed particles. Oxide layers on particle surfaces can form diffusion barriers which impede the interdiffusion process (Kuhn & Lawley, 1978).

(iv) Specific Surface

The specific surface of a powder is the surface area in square meters per kg or square centimeters per gm. If the specific surface of a powder is known, an average particle size may be calculated with the assumption that all particles are of equal size and spherical. Permeametry and gas adsorption are two principal methods used for determining the specific surface of metal powders (Lenel, 1980).

2.4.1 Mixing

In PM the process of mixing raw materials is an important first step since it controls the distribution of particles and porosity of the composites both of which influence the mechanical and tribological behaviors (Groover, 2002).

Mixing are carried out because the powders made by various processes may have different sizes and shapes, they must be mixed to obtain uniformity. The ideal mix is one which all the particles of each material are distributed uniformly. Powders of different metals and other materials may be mixed in order to impart special physical and mechanical properties and characteristics to the P/M product (Kalpakjian & Schmid, 2003). A greater packing density can be obtained if the particles of different sizes are mixed together so that small particles may be introduced into a packing (interstices) of large particles and still smaller particles into the voids of the medium-sized particles without increasing the overall volume of the bed (Jastrzebski, 1977).

Powder mixing must be carried out under controlled conditions in order to avoid contamination and deterioration. Deterioration is caused by excessive mixing, which may alter the shape of the particles and work harden them, thus making the subsequent compacting operation more difficult. Agglomeration can also occur during mixing due to cold welding at the particle contacts, large agglomerates will reduce packing density. Powders can be mixed in air, in inert atmosphere (to avoid oxidation), or in liquids, which act as lubricants and make the mix more uniform (Kalpakjian & Schmid, 2003) and (German, 1989).

Mixing is accomplished by mechanical means. Several mixing devices are rotating drum, rotating double-cone, screw mixer and blade mixer. Best results seem to occur when the container is between 20% and 40% full (Groover, 2002). The mixing time and the intensity of mixing powder and lubricant will affect such properties of the powder mixture as flow and apparent density.

Other ingredients are usually added to the metallic powders during mixing step. These additives include;

(i) **lubricants**, such as stearates of zinc and aluminium, in small amounts to reduce friction between particles, improved flow of the powder metals into the dies and at the die wall during compaction; and longer die life. Lubricants are chosen which attach themselves strongly to the metal surface and are not easily penetrated.

(ii) **binders** or surface active agents are added to particles to alter packing or mixing characteristics, to improve the powders' flow characteristics. It reduced friction between the metal particles. Some common additives are polyvinyl alcohol, stearic acid, sodium oleate, glycerine and oleic acid. Generally, the flow and packing of particles are improved by the presence of the appropriate surface active agent. The level of improvement is dependent on the molecular size of the additive, its polar character, the layer of coverage, the particle surface condition, the particle size and the temperature. Polar molecular coatings with short range interactions aid in keeping particles from agglomerating. The amount of binder added to a powder should slightly exceed the void space between particles. There are disadvantages to either an excess or deficiency of binder, compact distortion or defect formation. Complete burnout of the binder during sintering is necessary to avoid deleterious by-product that may affect mechanical properties (Chawla et al., 2001).

(iii) **deflocculants**, which inhibit agglomeration of powders for better flow characteristics during subsequent processing. The additive changes the electrical charges on the particles so that they repel instead of attract each other. Water is added to make the mixture more pourable and less viscous. Typical deflocculants include Na_2CO_3 and Na_2SiO_3 in amounts of less than 1% (Groover, 2002), (German, 1989), (Lenel, 1980) and (Kalpakjian & Schmid, 2003).

2.4.2 Compaction

In compaction, high pressure is applied to the powders to form them into the required shape. The compaction stage not only gives shape to the powder, but also decreases the porosity and increases the coordination number (German, 1989). Other function of compaction are to impart as much as possible the desired final dimensions with due consideration for any dimensional changes resulting from sintering and to provide adequate strength for subsequent handling (Upadhyaya, 2000). Presses used in conventional PM compaction are mechanical, hydraulic, or a combination of the two.

The capacity of a press for PM production is generally given in tons or kN or MN. The required force for pressing depends on the projected area of the PM part multiplied by the pressure needed to compact the metal powders. Reducing this to equation form;

$$F=A_pP_c \qquad \text{… (Eq. 2.1)}$$

Where; F = required force, (N); A_p = Projected area of the part, (mm^2);

P_c = Compaction pressure required for the given powder material, (MPa).

The workpart after pressing is called a green compact, the word green meaning not yet fully processed. As a result of pressing, the density of the part, called the green density is much greater than the starting bulk density. The green strength of the part when pressed is adequate for handling but far less than that achieved after sintering (Groover, 2002).

The green density depends primarily on the compaction pressure, the metal powder composition, and the hardness of the powder. Higher pressure and softer powder give a higher green density. Furthermore, pure iron powder will compact to a higher density than powders composed of alloyed steels (Kalpakjian & Schmid, 2003).

(a) Uniaxial Pressing

Widely used conventional compaction method is uniaxial pressing it involves rigid dies and special mechanical or hydraulic press. Proper amount of powder is placed in the die close to its final mould location. Since pressure is applied unidirectionally in die pressing, it is important that the design permits ejection of the part from the die, thus such features as undercuts, re-entrant angles cross holes and threads are not able to be incorporated into die-pressed parts. Design must ensure uniform density throughout the part (Upadhyaya, 2000). Irregular shape particles are appropriate for uniaxial cold compaction due to their mechanical interlocking (Sustarsic, et.al., 2003)

(b) Isostatic Pressing

In isostatic compaction, a uniform pressure is applied simultaneously to all the external surfaces of a powder body. For this purpose the powder is sealed in a flexible container and the assembly is immersed in a fluid which is pressurized (German, 1994) and (Lenel, 1980). If the isostatic pressing is done at room temperature, then it is called cold isostatic pressing (CIP). Two variations of CIP pressing are wet bag and dry bag (Eksi & Saritas, 2002).

In hot isostatic pressing (HIP), the container is usually made of a high-melting point sheet metal, and the pressurizing medium is inert gas or vitreous (glasslike) fluid. The system utilizes heated tooling and heated powder to achieve improved green density, higher green strength and improved ejection characteristics. The HIP process is relatively expensive and is used mainly in making superalloy components for the aerospace industry to close internal porosity and improved properties (Kalpakjian & Schmid, 2003) and (Upadhyaya, 2000).

(c) Other compacting and shaping processes

Other compacting and shaping processes used in powder metallurgy include: Metal injection molding, rolling, extrusion, pressureless compaction, ceramic molds and spray deposition.

2.4.3 Solid State Sintering

The terms solid-state sintering or solid-phase sintering are used for conventional sintering because the metal remains unmelted at these treatment temperatures (Groover, 2002).

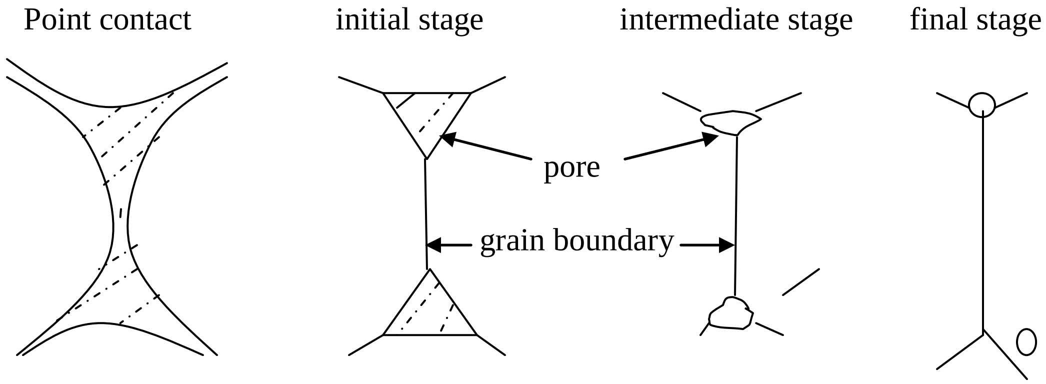

Figure 2.1. Interparticle bond development during sintering (German, 1996).

Figure 2.1 shows the series of sketches of sintering on a microscopic scale. Sintering involves mass transport to create the necks and transform them into grain boundaries. The mechanism by which this occurs is diffusion; other possible mechanisms include plastic flow. Shrinkage occurs during sintering as a result of pore size reduction. This depends to a large extent on the density of the green compact, which depends on the pressure during compaction (Groover, 2002).

With prolonged high-temperature sintering there will be a decreased in the number of pores, the pore shape will become smooth and grain growth can be expected. However, porosity cannot be completely eliminated, because voids remain after compaction, and gasses evolve during sintering. Porosities can consist of either a network of interconnected pores or closed holes.

In the latter stage of sintering, interaction between pores and grain boundaries can take one of the following forms;

(i) The pores can retard grain growth, they can be dragged by the moving grain boundaries during grain growth or the grain boundaries can break away from the pores, leaving them isolated in the grain interior,

(ii) Separation of pores from the boundaries limits the final sintered density. It is therefore important to minimize breakaway by careful temperature control during sintering. The selection of a correct isothermal sintering temperature is important in successful densification. Higher temperature lead to faster densification, but the rate of coarsening also increases. This increased coarsening rate may lead to abnormal grain growth with pores trapped inside large grains. Thus, although densification proceeds faster, the final density may be limited (Kalpakjian & Schmid, 2003) and (Upadhyaya, 2000).

The heat treatment consists of three steps:

(i) Preheat, in which lubricants and binders are burned off to improve bond strength and prevent cracking;

(ii) Sinter; and

(iii) Cool down.

Nearly all metals of technical importance react with gas of their surrounding atmosphere even at room temperature, but more so when treated at higher temperatures. The main purpose for using a special sintering atmosphere is to provide protection against oxidation, providing a reducing atmosphere to remove existing oxides, providing