FABRICATION AND CHARACTERISATION OF Mg/MgO METAL MATRIX COMPOSITES

Thesis submitted in partial fulfillment for the

Award of Degree

Doctor of Philosophy

By

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Asgar Shakil

Dedicated

To

My Teachers

&

My Beloved Family Members

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LIST OF ABBREVIATIONS

APS	Average Particle Size
BSE	Back Scatter Electrons
CDH	Central Drug House
CIP	Cold Isostatic Pressing
CMCs	Ceramic Matrix Composites
CP	Commercially Pure
CTE	Coefficient of Thermal Expansion
CYS	Compressive Yield Strength
DMD	Disintegrated Melt Deposition
DRTi	Discontinuously Reinforced Titanium
DSC	Differential Scanning Calorimetry
EDS	Energy Dispersive Spectroscopy
FRPs	Fiber Reinforced Plastics
FWHM	Full Width Half Maxima
HA	Hydroxy Apatite
HIP	Hot Isostatic Pressing
HVSFS	High Velocity Suspension Flame Spraying
MMCs	Metal Matrix Composites
PMCs	Polymer Matrix Composites
PRMMCs	Particulate Reinforced Metal Matrix Composites
R & D	Research and Development
SE	Secondary Electrons
SEM	Scanning Electron Microscopy
SiC	Silicon Carbide
TGA	Thermo - Gravimetric Analysis
UCS	Ultimate Compressive Strength
UTM	Universal Testing Machine
W-EDM	Wire cut Electric Discharge Machine
YS	Yield Strength
XRD	X – Ray Diffraction

LIST OF SYMBOLS

$\AA$	Angstrom
H	Hardness of the abraded surface
$HV_{0.05}$	Vickers microhardness using a load of 50g
K	Wear coefficient
Q	Wear volume per unit sliding distance
v_m	Vol. fraction of matrix
v_r	Vol. fraction of reinforcement
w	Weight
W	Normal load
ρ	Density
μm	Micron
λ	Wavelength
θ	Theta
ε	Lattice strain
β	FWHM

PREFACE

Magnesium is an energy efficient material having least density among all the light weight metals like aluminium, titanium, etc. There has been extensive studies on Mg based materials in past two decades due to huge pressure among researchers to search for materials having high specific strength for weight reduction applications. This weight reduction improves the engine efficiency and reduces fuel consumption especially in aerospace and automotive sector. Magnesium also has good damping capacity, superior machinability, good electromagnetic shielding and biocompatibility properties which makes it very suitable for use in automobile, aerospace, sports, electronics, and biomedical applications. But the low strength, low wear resistance and corrosion properties limit its widespread use. Reinforcing magnesium with ceramic particulates imparts high strength and wear properties to pure magnesium. Therefore, magnesium matrix composites having combination of properties like low weight coupled with high strength, hardness and wear resistance has potential to replace many aluminium and some polymer based parts in different applications. MgO is a ceramic particle having high melting point, hardness, high compressive strength and high chemical and thermal stability even in harsh conditions making it an appropriate choice for reinforcement which can improve hardness, strength, and wear properties of pure Mg. Moreover, it is cheap, easily available, and easy to handle. It is also compatible with human body.

Therefore, fabrication of Mg/MgO composites have been attempted by different processing techniques like stir casting, disintegrated melt deposition technique, in situ process, reactive cryomilling and high pressure consolidation, powder metallurgy, etc., but all processes have common disadvantage of high cost of production. Powder metallurgy is a near net shape process which imparts refined microstructural features and give superior strength levels. Very few studies are on Mg/MgO MMCs by powder metallurgy and most of them are for in situ MgO reinforced Mg. Therefore, in the present work ex-situ Mg/MgO composites have been fabricated by powder metallurgy process using two different low cost sintering processes - rapid microwave sintering process which has above 90 % power saving than conventional sintering and a novel sintering process which uses aluminium foil to prevent oxidation of compacts during sintering without the use of vacuum or inert gases. These two sintering processes reduces overall cost of powder metallurgy process for Mg/MgO composites.

Owing to various research gaps, following objectives are drawn for current study.

1. To fabricate Mg/MgO MMCs through powder metallurgy route assisted by rapid microwave sintering method.
2. To study different physical and mechanical properties of microwave sintered Mg/MgO composites.
3. To fabricate Mg MMCs through powder metallurgy utilising a novel sintering method.
4. To study the distribution of phases and its effect on properties through diffraction and microscopy techniques.
5. To study the effect of varying weight fraction of MgO particulates on physical and mechanical properties like density, hardness, and compressive behaviour of composites.
6. To study the dry sliding wear behaviour of composites fabricated by cost effective sintering method under varying load of 5, 10, and 15 N and at each sliding velocity of 0.6, 0.9 and 1.2 m/s.

The thesis comprises of total eight chapters. The brief description of each chapter has been given below.

Chapter 1 begins with introductory remarks about composites and its historical journey. Then it points out the importance of MMCs especially Mg MMCs and the problems associated with high cost of production. It also describes the potential advantages of MgO reinforced MMCs. Finally the chapter ends with brief description of present work.

Chapter 2 begins with introduction of composites, their history as well as applications. Then metal matrix composites are described particularly particulates reinforced metal matrix composites. The chapter also illustrates the factors which led to the selection of matrix and the reinforcement. A detailed description is provided for different fabrication methods for Mg MMCs along with references of Mg MMCs fabricated by those techniques. An extensive literature of previously reported Mg/MgO MMCs fabricated by different researchers are also provided. A brief about friction and wear laws, types of wear and previously reported works on dry sliding wear behaviour of Mg based MMCs also forms a part of this chapter. The chapter ends with formulation of problem and major objectives of present work.

Chapter 3 outlines the details of starting powders and the experimental procedures followed for fabrication of Mg/MgO composites. Both the sintering methods have been elaborated separately regarding the details of powders and experimental procedure. In the first part, characterisation of Mg and MgO powders are shown followed by details of processing parameters during mixing, compaction and sintering of samples. Then, the sample preparation is described followed by details of testing equipment, testing parameters and calculation methods for density and porosity, phase analysis and quantification, phase and elemental distribution, hardness and compression tests. The Mg/MgO composites fabricated by cost effective sintering technique have also been studied for dry sliding wear behaviour. So, the details of tribometer, testing parameters have been described. The details of characterisation methods for worn surface analysis are also mentioned.

Chapter 4 describes the results of fabrication of pure Mg and Mg/5MgO composites at varying sintering temperature of 450 °C, 500 °C and 550 °C by rapid microwave sintering. The post processing observations are also described. Pure Mg and Mg/MgO composites were successfully fabricated at all sintering temperature except Mg/5MgO composites at 550 °C sintering temperature. The addition of MgO in Mg matrix improved the density, hardness and strength of composites. The chapter firstly describes the results of different characterisations like density, XRD, SEM, hardness and compression test. Then, the discussions are made regarding the effect of varying sintering temperature on different properties of pure Mg as well as Mg/MgO composites by employing the observations of phase analysis and quantification, microstructural and elemental analysis and other tests.

Chapter 5 describes the results of characterisations of Mg/MgO composites fabricated by the novel sintering technique. Mg/xMgO, x = (0, 5, 10, 15) wt. % MMCs were fabricated and composites were tested on XRD, SEM, Vickers hardness tester, UTM, etc to study different physical and mechanical properties and their correlation with different phases and their distribution. The chapter firstly describes the results of density, XRD, SEM, hardness and compression tests. Then detailed discussions are made regarding the reasons for variation in properties due to change in MgO wt. %.

Chapter 6 describes the comparison of physical, mechanical and microstructural features of pure Mg and Mg/MgO composites fabricated by rapid microwave sintering and the novel sintering approach.

Chapter 7 describes the results of pin on disc dry sliding wear test of Mg/xMgO, x = (0, 5, 10, 15) wt. % MMCs. Each composition of sample was tested at 5 N, 10 N and 15 N load at each sliding velocity of 0.6 m/s, 0.9 m/s and 1.2 m/s for a constant sliding distance of 1000 m. The results of variation in wear rate and coefficient of friction have been described for each combination of sample, load and sliding velocity. The dominant wear mechanism at different load and sliding velocity have been identified by studying the micrographs, elemental maps and topographical images of worn surfaces.

Chapter 8 presents the salient conclusions on microstructural, mechanical and tribological characteristics of Mg/MgO composites synthesised by both sintering methods along with the scope of future study.

The important findings are mentioned below:

- Pure Mg and Mg/MgO composites were successfully fabricated by rapid microwave sintering method. The composites showed presence of only Mg and MgO phases and MgO particles are predominantly settled at the grain boundaries of Mg matrix. Agglomeration at few places were observed in Mg/MgO MMCs sintered at 500 °C. The average grain size gradually decreases with increasing sintering temperature in both pure Mg and Mg/MgO MMCs.
- Pure Mg and Mg/MgO composites were nearly 97 % and 96 % dense respectively. The density increases with increase in sintering temperature in pure Mg but vice versa occurred in Mg/5MgO MMCs. The addition of hard MgO reinforcements

improves the hardness of composites due to distribution of MgO particles at grain boundaries and grain refinement effect. However, there is very modest change in the average hardness value due to change in sintering temperature. The average compressive yield strength of pure Mg samples showed negligible improvement due to increase in sintering temperature. However, when 5 wt. % MgO is added in pure Mg, considerable improvement in CYS is observed due to load bearing effect, grain refinement and coefficient of thermal expansion mismatch. Ductility in pure Mg is improved as sintering temperature increases but the addition of MgO particles deteriorated the ductility in composites.

- Mg/xMgO, x = (0, 5, 10, 15) wt. % MMCs were successfully fabricated by a novel sintering method. The attempt to employ carefully wrapped aluminium foil on samples to effectively prevent oxidation from the surrounding environment was successful during the entire sintering duration. Composites were nearly 97 % and 98 % dense for Mg/5MgO and Mg/10MgO samples, respectively. The porosity decreases by 6.4 % and 33.3 % compared to pure Mg in Mg/5MgO and Mg/10MgO respectively. However, porosity sharply increases in Mg/15MgO samples.
- The composites showed an increase of 9 %, 13 % and 29 % hardness value with respect to pure Mg in Mg/5MgO, Mg/10MgO, and Mg/15MgO samples, respectively. Also an improvement of 6 % and 23 % in average compressive yield strength for Mg/5MgO and Mg/10MgO samples was observed when compared to pure magnesium.
- The wear rate of all samples generally increases with increase in load as well as sliding velocity. The coefficient of friction was lowest for pure Mg samples compared to the composite samples owing to soft nature of Mg and absence of MgO particles. The COF value generally increases as load is increased. There is

very minimal variation in COF value with respect to change in MgO weight fraction in composite samples.

- Wear in Mg/MgO composites does not occur by single wear mechanism. In fact, multiple mode of wear mechanism works simultaneously. In Mg/0MgO samples, wear due to abrasion was dominant at low load followed by plastic deformation at higher loads. In composite samples, wear due to oxidation and delamination were dominant. Adhesive wear was dominant at high load and high sliding velocity conditions.

This study explores the performance of MgO reinforcement for strength and wear characteristics improvement in pure Mg matrix through rapid microwave and a novel sintering method. It also explores the effectiveness of the new sintering approach for its use in the fabrication of Mg MMCs. The knowledge generated through this study is expected to provide a better understanding of Mg/MgO MMCs system.