UNIVERSITI PUTRA MALAYSIA

PROCESS PERFORMANCE AND CHARACTERIZATION OF MICROWAVE HYBRID AND CONVENTIONAL SINTERING METHODS ON IRON/SILICON CARBIDE

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By

SITI NURUL ADURA BINTI DAUD

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PROCESS PERFORMANCE AND CHARACTERIZATION OF MICROWAVE HYBRID AND CONVENTIONAL SINTERING METHODS ON IRON/SILICON CARBIDE

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August 2018

Chairperson: Suraya binti Mohd Tahir, PhD
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Microwave sintering has become one of the well-known sintering processing methods used by researchers to replace the conventional sintering process. The use of microwave for sintering in powder metallurgy has promoted cost reduction and energy savings for the industries. Microwave sintering has also been studied to sinter various kinds of Metal Matrix Composites (MMCs). The latest technology had combined microwave and conventional sintering into microwave hybrid sintering. In this research, the use of microwave hybrid sintering has been studied in terms of its viability to sinter the metal matrix composite of iron and silicon carbide (Fe/SiC). Comparison of the sintering processes and performance, physical (relative density, shrinkage and microstructure) and mechanical (tensile strength and hardness) properties of Fe/SiC were made for the samples sintered by both sintering processes. In order to determine the relative density and the shrinkage, the direct measurements were taken using Vernier caliper while for microstructure analysis, the morphology tests were conducted using Field Emission Scanning Electron Microscope (FESEM). For physical properties, the Brazillian Disc Test was conducted to analyze the tensile strength and Rockwell Hardness Tester was used to analyze the hardness. Three different compositions of Fe/SiC which are pure Fe, Fe-10SiC and Fe-20SiC were studied. The sintering temperatures used for both sintering processes were fixed at 1000 ^{o}C, 1050 ^{o}C, 1100 ^{o}C and 1200 ^{o}C with a heating rate of 10 ^{o}C/min and soaking time of 45 minutes in argon atmosphere. Fe-10SiC and Fe-20SiC were found damaged when sintered using microwave hybrid sintering at temperature above 1050 ^{o}C. The overall results show that Fe/SiC sintered by microwave hybrid had a faster sintering time compared to conventional sintering and the best sintering temperature to sinter Fe/SiC using microwave hybrid sintering was 1000 ^{o}C. In addition, the results also show that relatively higher shrinkage and swelling occurred for samples sintered under microwave hybrid process. The addition of SiC led to the decrease in the relative densities of the sintered Fe/SiC samples. The tensile strength of the Fe/SiC samples decreased with the increased in the SiC content. Microwave hybrid sintering produced

samples with relatively higher tensile strength values, especially for pure Fe and for 10 wt. % added SiC at 1050^{o}C sintering temperature

different compositions of Fe/SiC which are pure Fe, Fe-10SiC and Fe-20SiC were studied. The sintering temperatures used for both sintering processes were fixed at 1000 ^{o}C, 1050 ^{o}C, 1100 ^{o}C and 1200 ^{o}C with a heating rate of 10 ^{o}C/min and soaking time of 45 minutes in argon atmosphere. Fe-10SiC and Fe-20SiC were found damaged when sintered using microwave hybrid sintering at temperature above 1050 ^{o}C. The overall results show that Fe/SiC sintered by microwave hybrid had a faster sintering time compared to conventional sintering and the best sintering temperature to sinter Fe/SiC using microwave hybrid sintering was 1000 ^{o}C. In addition, the results also show that relatively higher shrinkage and swelling occurred for samples sintered under microwave hybrid process. The addition of SiC led to the decrease in the relative densities of the sintered Fe/SiC samples. The tensile strength of the Fe/SiC samples decreased with the increased in the SiC content. Microwave hybrid sintering produced samples with relatively higher tensile strength values, especially for pure Fe and for 10 wt. % added SiC at 1050^{o}C sintering temperature.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Sarjana Sains

PRESTASI PROSES DAN CIRI-CIRI KETUHAR GELOMBANG MIKRO HIBRID DAN KAEDAH SINTER KONVENSIONAL KEATAS FERUM/SILIKON KARBIDA

Oleh

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Pensinteran gelombang mikro telah menjadi satu proses pensinteran yang terkenal yang digunakan oleh para penyelidik untuk menggantikan proses pensinteran konvensional. Penggunaan gelombang mikro dalam metalurgi serbuk telah membantu dalam pengurangan kos dan penjimatan tenaga elektrik untuk industri. Pensinteran gelombang mikro juga telah dikaji untuk mensinter pelbagai jenis komposit logam matrik. Teknologi terkini telah meggabungkan kaedah pensinteran gelombang mikro dan konvensional kepada pensinteran gelombang mikro hibrid. Dalam penyelidikan ini, kajian terhadap kebolehupayaan gelombang mikro hibrid untuk mensinter Ferum and Silikon Karbida (Fe/SiC) telah dijalankan. Perbandingan terhadap prestasi proses pensinteran, serta ciri-ciri fizikal (ketumpatan relatif, pengecutan dan mikrostruktur) dan mekanikal (kekuatan regangan dan kekerasan) telah dijalankan kepada sampel yang disinter oleh kedua-dua proses pensinteran. Untuk menentukan ketumpatan relatif dan pengecutan, pengukuran terus diambil menggunakan Angkup Vernier manakala untuk analisis mikrostruktur, ujian morfologi telah dijalankan menggunakan mikroskop pelepas emisi pengimbasan elekron. Untuk ciri-ciri fizikal, Ujian Cakera Brazillian telah digunakan untuk analisis kekuatan regangan dan penguji kekerasan Rockwell telah digunakan untuk analisis kekerasan. Tiga komposisi Fe/SiC telah dikaji iaitu Fe asli, Fe-10SiC dan Fe-20SiC. Suhu pensinteran yang digunakan untuk kedua-kedua proses pensinteran ditetapkan pada 1000 ^{o}C, 1050 ^{o}C , 1100 ^{o}C dan 1200 ^{o}C dengan kadar pemanasan 10 ^{o}C/min dan masa rendaman 45 minit di dalam atmosfera argon. Fe-10SiC dan Fe-20SiC didapati rosak apabila disinter menggunakan pensinteran gelombang mikro hibrid pada suhu melebihi 1050 ^{o}C . Secara keseluruhannya, pensinteran gelombang mikro hibrid mempunyai masa pensinteran yang lebih cepat berbanding proses konvensional dan suhu optimum untuk mensinter Fe/SiC menggunakan pensinteran gelombang mikro hibrid adalah 1000 ^{o}C. Selain itu keputusan juga menunjukkan bahawa pengecutan dan pengembangan berlaku agak tinggi pada sampel yang disinter menggunakan ketuhar gelombang mikro hibrid. Penambahan SiC juga telah menyebabkan pengurangan ketumpatan relatif SiC. Kekuatan regangan juga berkurang apabila SiC meningkat akan tetapi sampel yang

disinter menggunakan ketuhar gelombang mikro hibrid menghasilkan sampel yang mempunyai kekuatan tegangan yang lebih tinggi terutamanya untuk Fe asli dan sampel yang ditambah dengan 10 wt. % SiC pada suhu pensinteran 1050 ^{o}C.

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Last but not least, once again I would like to express my thankful and deep appreciation for those who had been involved, directly and indirectly, in completing this report. Thank you very much.

I certify that a Thesis Examination Committee has met on 1 August 2018 to conduct the final examination of Siti Nurul Adura binti Daud on her thesis entitled "Process Performance and Characterization of Microwave Hybrid and Conventional Sintering Methods on Iron/Silicon Carbide" 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 Master of Science.

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This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of Master of Science. The members of the Supervisory Committee were as follows:

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LIST OF ABBREVIATIONS

h	Hours
^{o}C	Degree Celsius
min	minutes
rpm	Revolution per Minute
N	Newton
µm	Micrometre
wt. %	Weight Percent
MPa	Mega Pascal
Ar	Argon
Al_2O_3	Aluminium Oxide
Al/SiC	Aluminium Silicon Carbide
Al/TiC	Aluminium Titanium Carbide
CMCs	Ceramic Matrix Composites
DLMS	Direct Laser Metal Sintering
Fe	Iron
FESEM	Field Emission Scanning Electron Microscope
Mg	Magnesium
Mg/SiC	Magnesium Silicon Carbide
MMCs	Metal Matrix Composites
PMCs	Polymer Matrix Composites
PM	Powder Metallurgy
SiC	Silicon Carbide
Fe/SiC	Iron Silicon Carbide
UTM	Universal Tensile Machine
ZrO_2	Zirconium Oxide

CHAPTER 1

INTRODUCTION

1.1 Background of Research

The advancement of technology in materials engineering has led to the production of advance composites to meet industrial requirements such as aircraft parts, automotive parts and structural systems. There are several types of advanced composites used, specifically metal matrix composites (MMCs), polymer matrix composites (PMCs) and ceramic matrix composites (CMCs). Advanced composites have superior properties that a metal, polymer and ceramic can achieve such as the ability to withstand high temperature, higher wear resistance, higher strength-to-weight ratio and higher hardness properties. The current interest in MMCs field is iron/silicon carbide (Fe/SiC) as evident from the high number of research carried out by researchers on its properties and behaviour (Chakthin et al., 2008; Pelleg, 1999; Prabhu et al., 2014; Ramesh & Srinivas, 2009; Shao et al., 2008; Shen et al., 1992; Song et al., 2014; Srinivasa et al., 2010; Yodkaew et al., 2008). The development of Fe/SiC started in 1992 when a group of researchers started to investigate the characteristics of a mixture of Fe/SiC powders produced by mechanical alloying process (Shen et al, 1992). SiC is known as a perfect reinforced material, and has high hardness as well as high wear and corrosion resistance properties. The addition of SiC to Fe has enhanced the properties of the material such as outstanding wear resistance, high hardness, wear and creep resistance. The application of Fe/SiC in the industry includes the roll ring for high speed wire mills and material coating (Pelleg, 1999).

Most of Fe/SiC are produced using powder metallurgy (PM) route because PM is considered as a promising process method for producing MMCs; it provides benefit such as microstructural homogeneity, materials saving and near net shape product. The PM route is including mixing the materials in powder form, compacting the green compacts using a specific pressure and lastly sintering the green compacts at elevated temperatures. Sintering is the fundamental and significant processes in PM as it plays the crucial role in determining the properties of a product (Feng et al., 2008). The sintered properties of composites are greatly influenced by particle size, matrix composition, soaking time, temperature and atmosphere control, as well as heating and cooling rate (Rahman et al., 2014). Based on the review, previous researchers had used conventional sintering method (Yodkaew et al., 2008) and direct laser metal sintering (DMLS) method (Ramesh et al., 2009) to sinter Fe/SiC, but the use of microwave sintering method to sinter Fe/SiC is not yet explored.

Microwave sintering has been used in a variety of application for over 50 years and the research on it is getting attractive as the industry seek for green technology, as well as time and electrical saving process as it will contribute to the cost of final products. The evolution of microwave sintering has also been tested on composites as many researchers claimed that it improves the properties of materials and some materials have superior properties when sintered by microwave. Microwave sintering only involves the conversion of energy which is electromagnetic into thermal, compared to conventional methods that use conduction, convection or radiation heating.

For this purpose of study, Fe/SiC with several compositions of SiC (0, 10 and 20 wt.%) are fabricated through PM route and name as pure Fe, Fe-10SiC and Fe-20SiC. Two sintering methods will be used which are microwave hybrid and conventional sintering methods. The evaluation of physical and mechanical properties after sintering has been made and the comparison of both sintering processes are analysed in order to understand the effect of sintering process on physical and mechanical properties of the samples.

1.2 Problem Statement

In the past, various studies have been carried out on microwave sintering of MMCs. The effect of microwave on Al/SiC, Mg/SiC and Al/TiC have been studied and the results obtained which include physical and mechanical properties data have been compared with conventional sintering results. Previously, microwave sintering or microwave heating process is only focused on ceramics and metals, but the advantages of microwave sintering have opened up the researchers' eyes to carry out investigations on the MMCs.

Fe/SiC is a new kind of MMCs and still under investigation phase by the researchers. Liu et al. (2010) mentioned that the research on Fe/SiC is still immature when compared to Mg-Al light matrix composites. There are some problems are still not clear on Fe/SiC composites such as the reaction mechanism of Fe/SiC interface, ways to control the interfacial reaction and ways to enhance interfacial bonding. Based on their research, they found out that Fe/SiC have outstanding wear resistance, high hardness, high creep resistance and high temperature performance. In Chaktin et al. (2008a), the tensile properties of the sintered Fe/SiC were significantly higher than sintered Fe material. They also described that the tensile properties and hardness of the sintered Fe/SiC composite increased with increasing sintering temperature. Chaktin et al. (2008b) also stated that, the presence of particulate reinforcements reduced the density of Fe/SiC. However, Liu et al. (2010) discussed that the higher SiC content cause the incompability between SiC and the matrix hence resulted in increasing in pore which further tend to cause emergence and expansion of cracks so that the strength of materials decreased.

Various methods have been studied to produce Fe/SiC which are mechanical alloying (Shen et al., 1992), mechanical activation (Jia et al., 1999), pulsed laser deposition (Kumar et al., 2015) and powder metallurgy (PM) with conventional sintering method (Chakthin et al., 2008; Yodkaew et al., 2008), electrodeposition method (Ping et al., 2011) and laser sintering (Srinivasa et al., 2010), but producing Fe/SiC using PM with microwave or microwave hybrid sintering method has not been studied yet. PM with conventional sintering methods is the easiest and cheapest ways to produce Fe/SiC but the problem faced by conventional PM processing of composites are the long sintering times and often results in grain coarsening and poor performance of mechanical properties (Ghasali et al., 2016). Hence, in order to improve the properties of Fe/SiC sintered by conventional sintering, microwave hybrid sintering method is adopted and the results of both sintering methods are compared in terms of sintering time consumption as well as physical and mechanical properties of the samples.

1.3 Aims and Objectives

1. To evaluate the sintering performance process on Fe/SiC using microwave hybrid and conventional method.
2. To characterize the physical and mechanical properties of sintered Fe/SiC.

1.4 Scope of Study

Microwave sintering of composites is globally emerging and the MMCs of Fe/SiC has gained industrial demand for wear resistance application. The scope of this work is focused on processing of Fe/SiC with different compositions (0, 10 and 20 wt. % of SiC) developed by PM route but using different sintering method which are conventional and microwave hybrid. The parameters of both sintering methods were fixed to be the same. The sintering temperatures used were 1000 ºC, 1050 ºC, 1100 ºC and 1200 ºC with the heating rate of 10 ºC/min. The controlled atmosphere used was Argon (Ar) gas and all the samples undergo natural cooling process after soaking time (45 minutes) ended.

The sintering process as well as physical and mechanical properties of the samples were analysed and comparisons were made after sintering. The physical properties will be analysed in term of relative density, shrinkage and microstructure while the mechanical properties will be evaluated in term of tensile strength and hardness.

1.5 Thesis Outline

The first chapter in the thesis contains the introduction of the studies as a whole including the project background, problem statements, significance of study aim as well as objectives and scope of study.

Chapter two stresses on the literature review of powder metallurgy, microwave sintering of different kinds of materials including metals, ceramics and composites. The comparison of microwave and conventional sintering are made and the sintering method used to sinter Fe/SiC are also discussed.

Chapter three highlights the method of evaluation and characterization of Fe/SiC starting from preparing the mixture of Fe/SiC, compaction and sintering of the samples. Method of evaluation and characterization of sintering processes and physical and mechanical properties of the samples were also explained briefly in this chapter.

Chapter four describes the results of the analysis and evaluation of the study that has been made. Some graphs are plotted to show the relationship between the samples and their properties. Interpretation and justifying of data are discussed in this chapter.

The conclusion and summary of the results of this study are presented in chapter five, followed by recommendations for future research study.

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