# Physical and strength properties of Fe/SiC composites under microwave hybrid sintering method

Siti Nurul Adura DAUD<sup>1</sup>, Suraya Mohd TAHIR<sup>1,\*</sup>, Mohd Shamsul ANUAR<sup>2</sup>, Che Nor Aiza JAAFAR<sup>1</sup>, and Mohd Zuhri Mohamed YUSOFF<sup>1</sup>

<sup>1</sup> Department of Mechanical and Manufacturing Engineering, Faculty of Engineering, Universiti Putra Malaysia, 43400, Serdang, Selangor, Malaysia

<sup>2</sup> Department of Process and Food Engineering, Faculty of Engineering, Universiti Putra Malaysia, 43400 Serdang, Selangor, Malaysia

\*Corresponding author e-mail: su\_mtahir@upm.edu.my

Received date: 15 October 2018 Revised date: 23 March 2019 Accepted date: 12 May 2019

Keywords: Fe/SiC Microwave hybrid sintering Tensile strength Shrinkage Relative density

#### Abstract

Two different methods for the sintering of Fe/SiC composite prepared via uniaxial powder compaction are investigated in this work; the conventional furnace sintering and the microwave hybrid sintering methods. The important variables considered are the compositions of SiC in the Fe/SiC composite and the sintering temperatures used. The compositions of the SiC in the Fe/SiC used are 0, 10 and 20 wt. % and the sintering temperatures used are 1000°C, 1050°C, 1100°C and 1200°C. Damaged samples are obtained at the sintering temperatures of 1100°C and 1200°C for microwave hybrid method. For the undamaged samples obtained at 1000°C and 1050°C sintering temperatures, the physical and strength properties are studied. The physical properties of the sintered samples studied are the occurrence of shrinkage and swelling, the relative density and also their microstructure. The results show that the samples sintered under microwave hybrid process exhibit relatively higher shrinkage and swelling. The addition of SiC leads to the decrease in the relative densities of the sintered Fe/SiC samples. In addition, the tensile strengths of the Fe/SiC samples decrease with increasing SiC content. Microwave hybrid sintering produces relatively stronger samples by having relatively higher tensile strength values, especially for pure Fe and at 10 wt% SiC at 1050°C sintering temperature.

# **1. Introduction**

Powder Metallurgy (PM) is a process when powder material is compacted into a green body and sintered to a net shape at high temperatures [1]. It offers various advantages such as energy saving and near net shape parts [2,3]. There are two important steps in powder metallurgy which are compacting and sintering [4]. PM is a promising processing method for producing homogeneous [5] particulate reinforced metal matrix composites (MMCs) and high volume of reinforcement with fairly good volume distribution [6]. At present, conventional sintering method involves vacuum furnace or electrical furnace to sinter green body in PM process. However, conventional sintering process is time-consuming and operates at relatively high temperatures. There is a need to improve the sintering method to reduce the sintering temperature and time [7]. Thus, microwave sintering method is introduced to solve the problem faced by the conventional sintering method. Microwave is an electromagnetic radiation having a wavelength between 1mm to 1m with a frequency between 0.3GHz to 300GHz [8,9]. It offers rapid heating, therefore offering a cost effective and environmental friendly process [10].

In microwave sintering, the conversion of energy occurs, where electromagnetic energy is converted into thermal energy [11]. The use of microwave for material processing has been studied intensively in 1970's and 1980's and has been applied to a wide variety of materials [2]. Microwave processing of materials is mostly confined to ceramics, semiconductors, inorganic and polymeric materials until the year 2000 [12]. Leonelli et al. [2] relates that the initial success in microwave heating and sintering is mainly related to oxide and some non-oxide ceramics but recently the technique has been extended to carbide used in cutting tools. In the last two decades, microwave is used to heat and sinter ceramics materials but recent applications show that there are a lot of materials that have been sintered using microwave including iron and steel, copper, aluminium, nickel, Mo, Co, Ti, W and their alloys [12]. In the normal mode of resistance heating which is conventional sintering, the direction of heat flow is from the external to the internal structure of the material and this method does not yield very good microstructural properties of the surface. Therefore, Venkateswalu et al. [13] suggest that the combination action of microwave coupled with external heating can overcome the drawbacks of both sintering methods through rapid heating from both external and internal structures of the materials. A susceptor material such as SiC is used to assist sintering from the external surface of the material. This method is called microwave hybrid sintering [14]. Microwave hybrid sintering method allows immediate, smooth, programmable and reproducible thermal cycling of materials in comparison to normal microwave heating.

The role of susceptor in microwave hybrid is to absorb microwave energy and re-radiate the heat back to the surface of the sample. In this case, the heating of the sample to the external surface will be provided by the heat radiated from the susceptor in addition to the internal heating due to the microwave heating. This will reduce the temperature gradient between the external surface and internal sample structure and therefore lowering the risk of sample thermal shock. The common susceptor that is used in microwave hybrid is SiC and MoSi<sub>2</sub>. Electrical conducting materials such as cutting tool materials based on Al<sub>2</sub>O<sub>3</sub>/TiC, WC-Co hard metals, ceramic such as Al<sub>2</sub>O<sub>3</sub> and metal-ceramic ZrO<sub>2</sub>-Ni steel and Al<sub>2</sub>O<sub>3</sub> steel have been sintered using microwave hybrid method [15, 16].

Iron-Silicon Carbide (Fe/SiC) composite is utilized when there is a need in processes requiring high hardness, high temperature, high creep resistance, high speed and high wear resistance conditions [17] such as for the case of the roll ring of high speed wire rod mills and directive wheels [18] and for low and high-braking speeds [19]. However, there are limited published works on Fe-based composites with reinforcing agents such as SiC because previous research focus more on the light structural materials such as Al, Mg, and Ti based MMCs [19]. In addition, previous researchers used conventional sintering method [3,4] and direct laser metal sintering (DMLS) method [20] in the sintering process of Fe/SiC, but the use of microwave hybrid sintering method to sinter Fe/SiC has not yet been explored. Eventhough it has been reported that the density of Fe/SiC composites decreases as SiC content increases [4, 18, 21, 22], the high strength and hardness to weight ratio of Fe/SiC composites offers an attractive MMC to be explored. Therefore, this current work aims to evaluate the effects of microwave hybrid sintering process on the properties of the sintered Fe/SiC composite products.

### 2. Experimental

#### **2.1 Materials**

The powder metallurgy route was utilized to produce Fe/SiC. Fe (with 99.9% purity) was obtained from Sumitumo Sdn Bhd, Malaysia. SiC was acquired from Sigma Aldrich Corporation, USA. The particle size and density for both Fe and SiC powders are shown in Table 1.

# 2.2 Powder sample preparation and uniaxial die compaction

SiC was added to Fe at three different SiC compositions; namely 0, 10 and 20 wt. % of SiC added as listed in Table 2. Cylindrical shaped compacts were produced via the uniaxial die compaction process. All samples were compacted at 300 MPa using a universal testing machine (model 3382, Instron, USA) at the compaction speed of 5mm/min. The die set used in this work (Figure 1) has a diameter of 20 mm (Specac, UK). The mass of each 20 mm diameter cylindrical shaped green compact samples is approximately 7 g.

Table 1. Details of Fe and SiC powder.

Material	Iron (Fe)	Silicon Carbide (SiC)
Particle Size	45-212µm	33-74µm
Density (solid)	7870 kg/m <sup>3</sup>	3220 kg/m <sup>3</sup>

Table 2. Sample label.

Ratio	Sample
Fe 0 wt. % SiC	Pure Fe
Fe 10 wt. % SiC	Fe-10SiC
Fe 20 wt. % SiC	Fe-20SiC



Figure 1. Cylindrical die parts (a) top view (b) side view.

#### 2.3 Sintering process

After the cylindrical green compacts samples were formed, these samples were then sintered. Two types of sintering methods were used; a conventional sintering method through a conventional sintering furnace (Vecstar tube furnace, Vecstar, UK) as in Figure 2 and through a microwave hybrid sintering furnace (Dawynx Technology, Malaysia) as in Figure 3. The microwave power for microwave hybrid sintering used is 2.5kW. For both methods, four different sintering temperatures were used; 1000°C, 1050°C, 1100°C and 1200°C respectively at a constant holding time of 45 minutes. The atmosphere control used was Argon (Ar) gas, 99.9% purity.



Figure 2. Vecstar tube furnace



Figure 3. Microwave hybrid sintering furnace

#### 2.4 Physical properties

#### 2.4.1 Shrinkage and swelling

The shrinkage was determined by calculating the linear shrinkage using Equation (1) [23, 24]. The density of the sample was taken before and after sintering and the values obtained were substituted in Equation 1:

$$\frac{\Delta t}{t_{o}} [\%] = (1 - \left(\frac{\rho_{o}}{\rho_{s}}\right)^{1/3}) \times 100$$
(1)

Where  $\Delta t$  is the differences in thickness,  $t_o$  is the original thickness,  $\rho_o$  is original sample density, and  $\rho_s$  is the sample density after sintering.

#### 2.4.2 Relative density

Since the mass of each sample was fixed as 7 g, the physical dimensions (thickness and diameter) of the samples were measured to calculate the final volume of the compacts after the sintering process. These dimensions were then used to calculate the density and relative density of each sample using Equation 2.

% Relative density = 
$$\frac{\text{density of powder compact}}{\text{density of solid material}} \times 100$$
 (2)

Based on Table 1, the density of iron is 7870 kg/m<sup>3</sup> while the density of SiC is 3220 kg/m<sup>3</sup>. Thus, to find

the theoretical solid density of Fe/SiC powder mixture, the mixing rule (Equation 3) was used.

Solid density of Fe/SiC powder mixture =

$$\frac{\left(V_{Fe} \times \rho_{Fe}\right) + \left(V_{SiC} \times \rho_{SiC}\right)}{v_{total}}$$
(3)

where  $V_{Fe}$  is Fe volume,  $\rho_{Fe}$  is Fe density,  $V_{SiC}$  is SiC volume,  $\rho_{Fe}$  is SiC density and  $v_{total}$  is the total volume of Fe and SiC.

#### 2.4.3 Microstructure study

The metallographic sample preparation was carried out before performing the sample microstructure study. The first step was cold mounting and the aim was to hold the small sized samples before polishing and grinding. Two types of chemicals were used; epoxy resin and epoxy hardener in the ratio of 5:1. The chemical was then poured into the mounting cap holding the sample and was left overnight. Afterwards, grinding and polishing were conducted to reduce the imperfection of the sample surfaces. There were two steps in grinding; coarse grinding using silicon carbide paper of P120 and P280 grits as abrasives and followed by fine grinding which used silicon carbide paper of P400, P800, P1200 and P4000 grits. The grinding process was conducted at the speed of 200 rpm for 2 minutes for each grade of SiC papers. Following the grinding step, the polishing step was performed to ensure the samples were free from scratches and foreign elements. Polishing of the samples were carried out using a nap micro cloth at the speed of 150 rpm using a 0.05 µm Masterprep alumina polishing suspension as a lubricant. All the samples were polished and then coated with gold sputter using hot compression prior to the FESEM study The microstructure of the sintered samples was then examined using a field emission scanning microscopy (FESEM) (Nova NanoSEM 230, Thermo Fisher Scientific, USA).

#### 2.5 Tensile strength

The tensile strength of the sintered samples was determined using the Brazillian Disc Test [25]. The test was conducted using a universal testing machine (model 3382, Instron, USA). The maximum load (P) measured at the point where the sample was fractured diametrically was then obtained from the test and used to calculate the sample tensile strength in Equation 4:

Tensile strength, 
$$\sigma = \frac{0.636 \text{ P}}{\text{Dt}}$$
 (4)

where P is the load, D is diameter of sample and t is the thickness of sample.

# 3. Results and discussion

## 3.1 Sample condition after sintering

The sample physical condition after sintering is described in Table 3. It can be observed that pure Fe, Fe-10SiC and Fe-20SiC are successfully sintered at 1000°C and 1050°C for both methods of sintering. However, Fe-10SiC and Fe-20SiC samples are found to be damaged (crack) when sintered using microwave hybrid sintering at the higher 1100°C and 1200°C sintering temperatures.

It has been reported by a previous research [3] that sintering of Fe/SiC using conventional sintering method at sintering temperatures above 1250°C will cause the decomposition of carbide and reaction between Fe and Carbide, hence will not improve the mechanical properties of the composite. In this research, it is similarly found that good intact samples were also obtained when sintered using microwave hybrid sintering for the range of sintering temperatures used between 1000°C up to 1050°C.

Sample	Sintering Type	Temperature (°C)				
		1000	1050	1100	1200	
Pure Fe	Microwave Hybrid	Good	Good	Good	Good	
	Conventional	Good	Good	Good	Good	
Fe-10SiC	Microwave Hybrid	Good	Good	Crack	Crack	
	Conventional	Good	Good	Good	Good	
Fe-20SiC	Microwave Hybrid	Good	Good	Crack	Crack	
	Conventional	Good	Good	Good	Good	

#### **Table 3.** Sample condition after sintering.

#### 3.2 Shrinkage and relative density

The physical dimension of the sintered samples is an important physical characteristic that determines the quality of the formed Fe/SiC sintered composite samples. Generally, all the samples show physical dimensional changes after sintering, either experiencing swelling (represented by negative % shrinkage values) or shrinkage (represented by positive % shrinkage values) as illustrated in Figure 4. The highest shrinkage occurs at the relatively low sintering temperature of 1000°C when using microwave hybrid method for Fe-10SiC samples. The highest swelling also occurs for samples sintered under microwave hybrid process but at the higher sintering temperature of 1050°C for Fe-20SiC samples.

Based on Figure 4, it can be observed that when SiC is added to Fe, shrinkage occurs at the relatively higher sintering process of 1050° for both sintering methods. In contrast, swelling occurs for both methods when the sintering temperature used is slightly lower at 1000°C. Only samples sintered under the conventional method at the higher sintering temperature of 1050°C change from shrinkage for pure Fe to swelling when SiC is added. Other conditions display consistent swellings or shrinkages for pure and across the different SiC % weight addition.

In terms of the sintering methods applied, it can clearly be observed that the samples produced under microwave hybrid sintering exhibit higher sensitivity to the sintering temperature, where the highest shrinkage is observed at the low sintering temperature of 1000°C while the highest swelling is observed at the higher sintering temperature of 1050°C. The samples produced under the conventional method display relatively lower shrinkages and swellings at both sintering temperatures in comparison to the sample produced under microwave hybrid sintering.

The relative density obtained for samples sintered by both conventional furnace and microwave hybrid methods at two different sintering temperatures can be observed in Figure 5. Generally, the relative density of Fe/SiC composite samples is reduced when SiC is added to Fe. This is due to the comparatively lower density of SiC which is 3220 kg/m<sup>3</sup> compared to Fe which is 7870 kg/m<sup>3</sup>. The highest relative density is obtained for pure Fe samples regardless of the sintering method used at the highest sintering temperature of 1050°C. The lowest relative density obtained is also found for the samples sintered at the highest sintering temperature of 1050°C but using the microwave hybrid process at the highest SiC content of 20 wt. % used in this work.

Referring to Figure 5, it is noted that for pure Fe, the relative densities of the samples sintered by microwave hybrid and conventional sintering exhibit only a slight difference between them at both sintering temperatures of 1000°C and 1050°C. The effect of the sintering temperature is more pronounced when SiC is added to the Fe, especially for the case at the higher sintering temperature of 1050°C. At 1050°C, the addition of 10 wt. % and 20 wt. % of SiC causes the relative density of the Fe/SiC samples produced through the microwave hybrid process to be lower in comparison to the Fe/SiC samples produced under conventional sintering method as shown in Figure 5. This decrement is due to the negative shrinkage or swelling of the sintered samples and it is more significant for the samples sintered by

microwave hybrid method as discussed earlier (Figure 4). The swelling condition of the samples occurs due to fracture that can be seen through the surface morphology of the samples in Figure 6. The Fe/SiC samples sintered by microwave hybrid at 1050°C experience comparatively higher swelling where the samples start to form internal crack. Further increment of the sintering temperature above 1050°C results in total failure of samples as tabulated in Table 3. This finding is in line with Liu et al. [17] where the occurrence of fracture increases when SiC particles are added. As the SiC content increases in the samples, green densification of the samples will become more difficult and shrinkage will then transpire after sintering

[17]. The highest swelling can be observed for Fe-20SiC samples sintered at 1050°C under microwave hybrid method.

In contrast, at the lower sintering temperature of 1000°C, only slight difference can be observed when 10 wt. % SiC was added. The relative density of the samples obtained through microwave hybrid is slightly higher in comparison to conventional sintering when the SiC content increases to 20 wt. %. Therefore, the effect of SiC addition on the sample relative density is more pronounced at the higher sintering temperature of 1050°C in comparison to the case at the lower sintering temperature of 1000°C utilized in this study.



**Figure 4.** Shrinkage of pure Fe, Fe-10SiC and Fe-20SiC sintered by microwave hybrid and conventional at 1000°C and 1050°C.



**Figure 5.** Relative density of pure Fe, Fe-10SiC and Fe-20SiC sintered by microwave hybrid and conventional method at 1000°C and 1050°C.



**Figure 6.** Fracture morphology experienced by the swelling sample (a) Fe-10SiC at 1050°C by microwave hybrid (b) Fe-20SiC at 1050°C by microwave hybrid (c) Fe-10SiC at 1050°C by conventional method (d) Fe-20SiC at 1050°C by conventional method.



**Figure 7.** Tensile Strength of pure Fe, Fe-10SiC and Fe-20SiC sintered by microwave hybrid and conventional at 1000°C and 1050°C.



**Figure 8.** Microstructure of (a) Fe-10SiC at 1000°C by microwave hybrid (b) Fe-10SiC at 1000°C by conventional method (c) Fe-10SiC at 1050°C by microwave hybrid (d) Fe-10SiC at 1050°C by conventional method (e) Fe-20SiC at 1000°C by microwave hybrid (f) Fe-20SiC at 1000°C by conventional method.

#### 3.2 Tensile strength

The addition of SiC decreases the tensile strength of the sintered composite Fe/SiC samples at both sintering temperatures and sintering methods used as illustrated in Figure 7. This is due to the difficulties in green densification and sintering shrinkage as a result of incompatibility between SiC and the matrix, resulted in fragmented matrix and an increase in pore, which later cause emergence and expansion of cracks [26]. The highest tensile strength is obtained for samples formed under microwave hybrid sintering process for pure Fe at the sintering temperature of 1050°C. Relatively low tensile strength values are also recorded for samples containing the highest 20 wt. % SiC content for microwave hybrid sintering process at 1050°C, and conventional sintering process at both sintering temperatures of 1000°C and 1050°C respectively. Microwave hybrid drying process at the highest 20 wt. % SiC content yield slightly higher tensile strengths in comparison to the other aforementioned sintering conditions.

In terms of the influence of sintering temperature, it is also interesting to note that based upon the data illustrated in Figure 7, the samples produced under the microwave hybrid sintering at the higher temperature of 1050°C yield higher tensile strength values than the samples produced at the lower sintering temperature of 1000°C for pure Fe samples. Instead, as 10 wt. % SiC is added to the samples, higher tensile strength values are recorded at the lower sintering temperature of 1000°C. These trends are also evident for the samples obtained under conventional sintering process. Further addition of SiC content to 20 wt. % results in similar low tensile strength values except for the case of the microwave hybrid sintering process at the higher temperature of 1050°C that exhibits slightly higher tensile strength values than the rest of the samples obtained.

In terms of sintering methods, microwave hybrid sintering of pure Fe samples leads to relatively higher tensile strength values compared to the samples sintered under conventional method at both sintering temperatures. The difference between the tensile strength values under these two methods decreases upon addition of SiC as shown in Figure 7. These higher values of tensile strength obtained through the use of the microwave hybrid method is due to the reduction of pore formation within the samples, as illustrated in Figure 8. The formation of pores can be seen for Fe-10SiC sample sintered by microwave hybrid sintering at 1000°C as in Figure 8 (a). Instead, when Fe-10SiC sample is sintered at the same sintering temperature using conventional sintering, the crack formation develops within the sample as in Figure 8 (b). This is also clearly evident at the higher sintering temperature of 1050°C for the same Fe-10SiC composition. However, when the SiC composition increases to 20 wt. %, there is no clear surface differences between the two sintering methods as

depicted in Figure 8 (c) and (d). Hence, the use of microwave hybrid sintering method for the same composition of Fe/SiC composite and same temperature as in the conventional method has successfully prevent crack formation within the samples and hence yielding higher tensile strength values for the case of Fe-10SiC samples.

# 4. Conclusions

Damaged samples are obtained at the higher sintering temperatures of 1100°C and 1200°C after undergoing microwave hybrid sintering. For the undamaged samples obtained at the sintering temperatures of 1000°C and 1050°C, the addition of SiC to Fe decreases the sample tensile strengths and relative densities for both methods. The microwave hybrid process produces samples that display relatively higher shrinkages and swellings in comparison to the samples produced under the conventional method. Relative densities of the samples decrease with the addition of SiC. However, the tensile strength of microwave hybrid sintering samples are relatively higher in comparison to the ones produced under the conventional method, especially at the sintering temperature of 1050°C with pure Fe and at the lower SiC content of 10 wt. %. Hence, the physical and strength properties of the samples produced under the two different sintering methods are found to be dependent upon both the SiC content and the sintering temperatures.

# 5. Acknowledgements

The authors' want to thanks Mybrain15 and Universiti Putra Malaysia for sponsoring this project.

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