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The Effect of the Nano Silicon Carbide on Mechanical Properties of Aluminium

Rizal Mohamed Noor Khairul Fauzi Karim Aznifa Mahyam Zainuddin

ABSTRACT

This study focuses on nano silicon carbide particulate-reinforced metal-matrix composites processing route using different powder metallurgy techniques; cold press-sintered, hot press-vacuum and effect of milling process. SiC particulates, whose diameters 50~200 nm were choosen for incorporated into Al-3wt% SiC. The microstructures of the samples were investigated by means scanning electron microscopy and correlated to their mechanical properties.

Keywords: Powder metallurgy, compaction, sintering, nano particles

Introduction

The development in discontinuously reinforced aluminium matrix composites over the past twenty-five years has attracted the attention of materials producers and end users because of their outstanding mechanical properties. Wide ranges of production techniques have been developed for aluminium matrix composites like powder metallurgy or via melt based approaches. However the powder metallurgy (PM) method is the most attractive due to several reasons. Firstly, PM offers micro structural control of the phases. Secondly, the lower temperature employed during the process accounts for the strict control of the inter-phase kinetics.

In addition, when Al-SiC composites produced by a melt process, SiC often reacts with molten aluminium to form Al_4C_3 which may result in a degradation of the reinforcement strength and the interfacial strength. For certain temperatures and matrix compositions, SiC is prone to be attacked by the molten aluminum matrix according to the following reaction,

4 Al + 3 SiC \rightarrow Al₄C₃ + 3 Si

The reaction is deleterious to the properties of the composites since it leads to the degradation of the SiC reinforcement and produced a brittle intermetallic compound, Al_4C_3 .

On the other hand, powder metallurgy tends to offer more control over reinforcement distribution, and require less energy input than conventional foundry route. The PM route for making Al matrix composites include:

- i. Blending
- ii. Hot Pressing

A survey of the existing literature shows that limited work has been done on composite produced via milling and hot press process route. Furthermore, there is a limited amount of information on metal matrix composites with lower weight fraction, 3wt% nano SiC.

Objective

This paper reports part of the study undertaken to improve the understanding of the effect of processing route on the mechanical properties of Al with an addition of 3wt% nano SiC.

Raw Materials and Experimental Procedure

Raw Materials

The raw materials being used and their physical properties are shown below in Table 1.

Raw Material	Chemical Formula	Melting Temp (°C)	Average Particle size	Theoretical Density, g/cm ³	Purity %
Aluminum powder	Al	660.1	42 µm	2.700	99.9
Micron-Silicon	SiC	2650-2950	76 μm	3.217	99.0
Carbide powder					
Nano Silicon	β-SiC	2650-2950	50~200 nm	3.217	99.0
Carbide powder					
Stearic Acid	$CH_{2}(CH_{2})_{16}$	67.46	-	-	95.0
	CO,H ² ¹⁰				
Acetone	CH ₃ CÔCH ₃	- -	-	-	-
Toulene	C ₆ H ₅ CH ₃	-		-	-

Table 1: Physical Properties of Raw Material

Experimental Procedure

To evaluate the effect of process route, we will use Aluminium-SiC matrix. Table 2 illustrates the details.

Composition	Process Route
Al (pure)	Blend-Cold Press-Sintered Blend-Hot Press
Al-3% Wt. nano SiC	Blend-Cold Press-Sintered Blend-Hot Press Blend-Milling-Cold Press-Sintered Blend-Milling-Hot Press

Table 2: Process Route for the Powder Mixture

The as-blended mixture powder were loaded into stainless steel vial together with thirty balls of stainless steel for mechanical milling by using Planetary ball mill (PM 400). Total mass of the powder was thirty grams. The weight ratio of ball to powders was 19:1. Milling of these mixture powders was performed five hours was stopped periodically every five minutes.

The powders are then being compressed into two different dies; rectangular and cylinder shape by using Instron press machine. Compaction process was performed in a single acting die. The pressure

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during the compaction is maintained at 62.3 kN. Sintering process is carried out by using firing furnace with sintering temperature of 600 °C for 2 hours with heating rate of 10 °C/minutes. Hot press is performed under pressure of 17.8 kN and temperature 580 °C for one hour.

The microstructure characterization of the sample was performed using X-ray diffractor (XRD) and scanning electron microscopy (SEM). The Malvern Instrument Mastersizer was used to measure the particle size distribution (PSD) of composite powders. Density test of the sample carried out using Micromeritics AccuPyc 1330 and hardness test with Vickers hardness tester with load of 10 kg. Three point bending test (flexure test) was performed using Flexural Test-Instron 5582 by using a standard span dimension 30.0 mm and loading rate of 1.0 mm/s.



Figure 1(a): Flask and Ball Mill



Figure 1(b): Planetary Ball Mill -PM 400



Figure 1(c): Cylindrical Die



Figure 1(d): Rectangular Die



Figure 1(e): Hot Presses Group 1400

Result and Discussion

Microstructure Analysis

Microstructure of the specimen will be observed through SEM. Figure 2(a) and 2(b) indicates SEM micrograph of the mixed powder through blend and mechanical milling. The distribution of particle, size and agglomeration can be observed through this morphology.



2(a): Blending

2(b): Mechanical milling

Figure 2(a) and 2(b): SEM Micrographs of Al-3Wt% Nano SiC after (a) Blended and (b) Mechanical Milling

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The blended sample shows in Figure 2(a), which indicated that the present of original shape of Al. SiC could not be determined due to smaller quantity of the particles. For milled sample from Figure 2(b), Al has been deformed and forming agglomerate with some flaky particle. During milling, particles size has been reduced and the particles were rounded, although some big flake still present.







2(d): Hot Press Vacuum



The as-blended samples undergo two different routes; cold presssintered and hot press-vacuum. Figure 2(c) and 2(d) shows microstructure of Al-3wt% nano SiC produced from cold press-sintered and hot press vacuum at temperature of 600°C. Some pores were present in the cold press sample, which is due to poor sintering condition. During the early stage of sintering, diffusion between powder particles begins to occur. As the diffusion process continues, it formed more dense body and shrink. From details examination of the powder by EDS and SEM, it was found that the as-received Al powder has high concentration of O₂ indicating the Al powder has initially oxidized to some unknown degree. However the pores level significantly reduce through process route milling-hot press. The milling process, which involves cold working, has refined the particle size and break-off the oxide layer from Al, thus improving the particle contact and sintering behavior. Hot press process which a combination of simultaneous pressure and sintering resulted a relatively better densification, thus further increase the number of contacts and size of contact planes, which produced low porosity. Micrographs in Figure 2(d) indicate very small amount of pore present and the pores size was very small.

Density Analysis

The powders were consolidated by cold press-sintered and hot press process separately. The apparent densities of cold press-sintered and hot press were measured experimentally and compared to the theoretically calculated density. F.Thumbler (1993) suggested the relationship between density and strength which stated that the strength increase linearly with density. The density versus process route is shown in Figure 3.



Figure 3: Density of the Consolidated Sample

It can be observed from the Figure 3 that milled sample has a higher density than as-blended, this is to be expected since milled sample shows a reduction amount and size of pores. This reduction results in an increase in the density of the compact powder.

Hardness Analysis

The micro-Vickers hardness was measured at five points from the along the surface of the polished samples. The variation of hardness of the sample is plotted as shown in Figure 4.



Figure 4: Hardness for consolidated sample

Figure 4 shows that the cold press-sinter sample the hardness value increase up to 105.7 Hv from 28.7 Hv. Similar trend indicated for hot press sample, which significantly increase from 41.7 Hv to 450.6, which is more than ten times. Generally Al surface in contact with ambient air is coated with an oxide film, Al₂O₃ which having a high energy of formation and may have a strong influence on the sintering behavior. They strongly inhibit sintering since the diffusion exchange between the metallic surfaces is reduced. The way to destroy the oxide films is by introduce mechanical milling process, which spalls off the oxide films mechanically and provides fresh metallic surfaces. This effect brings the particles weld together by diffusion and leads to progressively increased hardness. As a result, from Figure 4 the milled sample demonstrated a much higher hardness compared to non-milled sample. Besides that, milling process also provide a homogeneous mixture of SiC particle inside Al matrix compared to individual blending process. Figure 4 also shows that hot press process demonstrated an increase in hardness compare to cold press. The trend is parallel with an increase of density and the reduction of the porosity in the composite (C.P Ling 1998).

Three-Point Bending Analysis

The test is conducted to obtain the strength of each consolidated samples. The variation of strength for each process route is plotted versus consolidation of the sample as in Figure 5.



Figure 5: Strength of the Consolidated Sample

From Figure 5, it can be seen that the strength of the cold presssinter sample increase from 43.5 MPa to 4.6 MPa with incorporation of 3wt% SiC. A similar trend in strength is observed for hot press sample, the strength increase drastically from 6.8 MPa to 16.2 MPa. The above result is not including milled sample, this is due to highly brittle behaviour during the test. Hot press process constantly produce a superior strength than cold press-sinter, this is linearly with the increasing of density and reduction of pores.

Conclusion

Al-3% wt nano SiC composites were produced successfully by hot press process and demonstrate a high strength and hardness. A dramatic improvement has been observed in mechanical properties for consolidated sample Al-3% wt nano SiC via mill-hot process compared to cold presssinter, the hardness improves from 105.7 Hv to 450.6 Hv. For hot press sample, the strength also demonstrated the similar trend, which increased from 4.7 MPa to 16.2 MPa. Mechanical milling proven to be a successful method in remove the oxide layer from the surface of particle. Hot press is a useful technique, which can improve the density and reducing the porosity of the composites.

Suggestion Works

A study on the effect of hot isostatic press (HIP) could be done to further improve the density of the consolidated samples. A details study also need to carry out to understand and improve brittle behavior of milling sample.

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