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Al-CNT composites produced from Friction Stir Processing and Selective Laser Melting

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Keywords

Friction Stir Processing; Selective Laser Melting; Nano-composites; Carbon Nano-tubes; Mechanical Properties.

Abstract

Aluminium reinforced with multi-wall carbon nanotubes was produced using friction stir processing and selective laser melting. Very fine grains were successfully achieved in both methods with high Vickers hardness values. Cavities were present in selective laser melting of AlSi10Mg reinforced with multi-wall carbon nanotubes parts resulting in higher stress concentration brittle fracture appearance. The high laser absorption and higher thermal conductivity of multi-wall carbon nanotubes resulted in the formation of the cavities in the melted parts. However, the fractography images showed dimpled like appearance indicating a ductile fracture mechanism. Multi-wall carbon nanotubes were observed at the fracture sites indicating the presence of a crack-bridging mechanism. In comparison to carbon nanotubes reinforced aluminium by friction stir processing, selective laser melting produces aluminium reinforced aluminium with better mechanical properties.

1 Introduction

Particulate-reinforced metal composites have a superior high elastic modulus, stiffness and wear resistance and have drawn much attention in defence, aerospace and automobile industries [1-3]. The addition of small concentrations of reinforcement particles has the ability to significantly enhance the strengthening effects [3]. Carbon nanotubes have gained much attention in the fabrication of particulate-reinforced metal composites, due to its remarkable mechanical and geometrical properties (extremely high Young's modulus and rupture strength, high aspect ratio, nanometre-range diameter) [4-7]. The processing of multi-wall carbon nanotubes reinforced aluminium composites using powder metallurgy were studied and multi-wall carbon nanotubes were observed to have slowed fatigue crack propagation by crack-bridging, and also significantly improved the mechanical properties [8].

Friction stir processing is a solid state processing method capable of modifying material microstructure [9, 10]. It consists of a non-consumable rotating tool being plunged into a work piece. The material is processed as the rotating tool transverses along a path while the friction generated between the tool and the material results in the plasticity without melting. Friction stir processing is a desirable fabrication process of particulate-reinforced metal composites as it avoids the intermetallic formations between the reinforcement and the matrix [11, 12].

Friction stir processing has attracted much research attention ever it was reported of its capability to fabricate particulate-reinforced metal composites [13]. A study was conducted on the friction stir processing of the nano- Al_2O_3 reinforced AA6061 alloy [14]. The grain structure evolution and mechanical properties study observed that the reinforcement particles were uniformly dispersed with significant increases in micro-hardness and tensile test strength. Recently, a study on the friction stir processing of multi-wall carbon nanotubes reinforced AA6061 alloy was conducted [15]. Multi-wall carbon nanotubes were successfully dispersed with improved micro-hardness and yield strength was observed.

Selective laser melting is an additive manufacturing technique using a laser beam melting powder layer by layer. With the aid of a CAD computer technique and layer wise production, high quality parts with dimensional accuracy, superior mechanical properties and complex geometry could be produced.

There were some studies conducted on the fabrication of metal matrix composites using the selective laser melting technique [16-18]. To the best of the authors' knowledge, no study has been done on the production of multi-wall carbon nanotubes reinforced aluminium composites using the selective laser melting process. The mechanical alloying AA6061-CNT composite using semi-solid powder processing was studied, however, after mechanically alloying the powders and multi-wall carbon nanotubes, the multi-wall carbon nanotubes became significantly shorter and the aluminium powders were no longer spherical [19]. This will affect the flowability of the powder and prevent the uniform dispersion of the powder layers during selective laser melting. Hence, this study aims to study a new powder preparation process to produce homogenous AlSi10Mg-CNT powders for the selective laser melting of Al-CNT composites.

1.1 Friction stir processing experimental details

In the study of friction stir processing of multi-wall carbon nanotubes reinforced AA6061 alloy, rolled AA6061-T6 with the nominal grain diameter of 70 μm was used as the base material. An array of 720 cylindrical holes measuring 1mm in diameter and 2 mm in depth were machined on the surface to provide a reservoir for pre-placing the reinforcement particles. Multi-wall carbon nanotubes with other diameter ranging from 10 nm to 20 nm, length ranging from 10 μm to 30 μm from *Showa Denko* were used (see Fig. 1). A friction stir welding robot was used to perform multi-pass friction stir processing on the work-piece. The rotating tool used was a threaded conical probe with three flats, probe length of 2 mm, base diameter of 5 mm and tool shoulder of 12.5 mm. friction stir processing was performed at 1200 rpm, 3 mm/s and tilt angle 3°. 0.5 weight percentage of multi-wall carbon nanotubes were used as it gives the optimal mechanical properties [20]. The detailed process of friction stir processing of multi-wall carbon

nanotubes reinforced AA6061 alloy was discussed in the previous study; hence it will not be repeated in this journal [15].

Fig 1. Field emission scanning electron microscopy image of multi-wall carbon nanotubes used in the study. Scale bar shows 1 μm .

Table 1. Chemical composition of the AA6061 and AlSi10Mg aluminium alloy.

Material	Element Weight (%)							
	Al	Si	Fe	Cu	Mn	Mg	Zn	Ti
AA6061 [21]	98.22	0.47	0.11	0.26	0.01	0.91	0.01	0.01
AlSi10Mg [22]	88.585	10.08	0.16	0.001	0.002	0.35	0.002	0.1

1.2 Selective laser melting experimental details

In this study, AlSi10Mg was used instead of AA6061 powder. Dense parts can be achieved from the selective laser melting of AA6061, however, cracks and several pores were present. Therefore, it was recommended by *Selective Laser Melting Solutions GmbH* to use AlSi10Mg. AlSi10Mg is capable of achieving fully dense parts due to its higher silicon content in the alloy matrix (see Table 1).

A volatile solvent was added to the multi-wall carbon nanotubes before it was placed in the Branson Digital Sonifier. Ultrasound was applied at 30% intensity to the suspension

for a total of 3 minutes with intervals of 1 minute each. The suspension was stirred in between each interval. The AlSi10Mg powder was then added to the suspension and stirred using a magnetic stirrer for 1 hour before leaving it to air dry. The powder was then examined under the field emission scanning electron microscopy. Multi-wall carbon nanotubes were observed to have loosely attached itself on to the spherical metal powders (see Fig. 2). However, it is evident that the multi-wall carbon nanotubes are still in small clusters. The average diameters of the powders are of 21-27 μm . Finer powders with diameters smaller than 10 μm tend to agglomerate to form bigger irregular shape. These clusters significantly affected the flowability of the powder. The powder was sieved with a 63 μm sieve prior to selective laser melting process. The *Selective Laser Melting 250HL* by *Selective Laser Melting Solution GmbH* was used to process the AlSi10Mg powder, with a scanning speed of 1140 mm/s, power at 350 W, layer thickness at 50 μm , and hatch spacing at 0.17 mm.

Fig 2. Field emission scanning electron microscopy image of the spherical AlSi10Mg powder. Scale bar shows 10 μm .

Metallographic samples were extracted from the transverse section of the built parts and polished using conventional mechanical polishing methods. They were then characterised using field emission scanning electron microscopy. Micro-hardness was measured using the Vickers hardness with 50 grams force loading with a minimum of 5 points. Tensile samples were wire cut from the platform and machined to “I”-shaped,

flat rectangular sub-sized samples with gage length of 25 mm and tested according to ASTM-E8-04 standards at test speed of 1 mm/min (see Fig. 3). Fractography was done using field emission scanning electron microscopy on the fracture site.

Figure 3. Dimensions of the samples used for tensile test in millimetres.

2 Results and discussions

2.1 Friction stir processing particle dispersion

The rotating tool plunges into the work-piece and transverse along during friction stir processing. The stirring motion mixes the material and reinforcement together resulting in a uniform dispersion when multiple passes were applied. It was observed that 3 passes were sufficient to uniformly disperse multi-wall carbon nanotubes [15]. The large dendrites and silicon particles as in the received AA6061-T6 samples were broken down during friction stir processing (see Fig. 4a). No multi-wall carbon nanotubes were observed; it is believed to have broken down to smaller clusters and uniformly dispersed in the aluminium matrix (see Fig. 4b) [15].

Significant grain refinement was observed from friction stir processing samples. The addition of reinforcements resulted in even smaller grains (see Table 2). In a previous study, the flow stress and processing temperature were kept constant by using the same processing parameters for all samples, to study the effects of multi-wall carbon nanotubes [15]. It was concluded that the observation of more sub-grain boundaries were present in the friction stir processed samples. The material experiences intense

dislocation resulted in several deformations. The main contribution factor was from the continuous dynamic recrystallization which occurred under migration of sub-grain/ grain boundaries and continuous strain [23-25]. A study on multi-pass friction stir welding observed that dynamic crystallization is strongly correlated with flow stress and not the temperature during the deformation [26]. However, there were studies that showed that welding temperature affects the grain sizes in the stir zone [26-28].

2.2 Selective Laser Melting particle dispersion

During the processing of selective laser melting, the laser beam intensity was modulated to ensure that the new powder layer is melted and penetrates the previous layer [22]. This ensures that the layers are well connected. The laser irradiation exposure period is in the range of milliseconds. Due to the short exposure period, very high cooling rates ranging between 10^3 and 10^{11} K/s are achieved, promoting greater undercooling [29]. Hence, finer grains were produced. Gradual change in the solidification regime from dendritic to cellular-dendritic microstructure was therefore experienced [30].

Significant grain refinement was observed from the selective laser melting specimens. The addition of reinforcements resulted in even smaller grains (see Table 2). As all the parameters used for selective laser melting processing of AlSi10Mg and AlSi10Mg with multi-wall carbon nanotubes are kept constant, the refinement of grain size could only be due the addition of multi-wall carbon nanotubes.

The addition of reinforcement particles resulted in finer grains in both friction stir processing and selective laser melting processes. This could be due to the Zener pinning effect by the nano-sized multi-wall carbon nanotubes retarding the grain growth of the aluminium matrix. It is reported that the rate of grain growth during recrystallization of metal with dispersed second phase particles can be described using the following equation (1) [31],

$$\frac{dR}{dt} = M(P - P_z) = M\left(\frac{\alpha\gamma_b}{R} - \frac{3F_v\gamma_b}{2r}\right) \quad (1)$$

From the equation, F_v represents the volume fraction, M represents the boundary mobility, P represents the driving pressure from the curvature of the grain boundaries, P_z represents the Zener pinning pressure, R represents the radius of the grain, r represents the radius of the pinning particles, α represents a small geometric constant and γ_b represents the boundary energy. The grain growth will stop when $P=P_z$. Result in the following equation (2),

$$\frac{\alpha\gamma_b}{R} = \frac{3F_v\gamma_b}{2r} \quad (2)$$

Therefore, when the mean grain radius (D) and the radius of curvature (R) are taken to be the same, the Zener limiting grain size ($\alpha=1$) can be obtained resulting in the following equation (3),

$$D_z = \frac{4r}{3F_v} \quad (3)$$

Table 2. Grain size measurements using EBSD.

Material and Process	AA6061-T6 [15]	Friction stir processed AA6061-T6 [15]	Friction stir processed AA6061 with multi-wall carbon nanotubes [15]	Selective laser melted AlSi10Mg	Selective laser melted AlSi10Mg with multi-wall carbon nanotubes
Average Grain Size (μm)	70.04	5.04	4.67	4.96	4.41
Standard Deviation	39.04	3.53	3.16	5.55	5.50

Cavities were present in the samples of AlSi10Mg with multi-wall carbon nanotubes (see Fig. 5). Tiny spherical structures were observed in the cavities measuring about 0.1 μm . These spherical cavities could not be from the vaporisation of the multi-wall carbon nanotubes as the multi-wall carbon nanotubes were 10 nm to 20 nm and the melting temperature for multi-wall carbon nanotubes is around 2800°C vacuum and 750°C in air [5]. Moreover, the build chamber was being purged with argon gas and oxygen level was maintained at 0.02%. Hence, it could only be caused by the vaporising activity of the AlSi10Mg and immediately freezing as it comes into contact with the cold air and surfaces.

Figure 4. Field emission scanning electron microscopy image of AA6061-T6 samples (a) with 3 pass condition; (b) Friction stir processed AA6061 with multi-wall carbon nanotubes composite with 3 passes [15]. Selective laser melting of AlSi10Mg samples (c) without multi-wall carbon nanotubes (d) with multi-wall carbon nanotubes. Scale bar shows 10 μm.

Figure 5. Field emission scanning electron microscopy image of a cavity on the (a) AlSi10Mg with multi-wall carbon nanotubes sample; (b) showing a higher magnification of the cavity. Scale bar shows 10 µm.

2.3 Mechanical properties

2.3.1 Micro-hardness

Vicker's hardness values were measured from the samples (see Table 3). The dissolution of hardening precipitates is believed to have decreased the hardness value of AA6061-T6 when friction stir processing was performed. The addition of multi-wall carbon nanotubes to the friction stir processing resulted in a significant increase in the hardness value. The Orowan strengthening effect is believed to be the main reason for this behaviour observed [14].

For the selective laser melting of AlSi10Mg, it has hardness values superior to those of the samples that went through friction stir processing. The high hardness value is believed to be mainly contributed from Orowan strengthening and also the small grain size produced from the process [14]. The addition of multi-wall carbon nanotubes in AlSi10Mg results in a significant improvement in the hardness of the material.

Table 3. Microhardness values measurement of the base material, friction stir processing of base material and friction stir processing of Al-CNT composite.

Materials and Process	AA6061-T6 [15]	Friction stir processed AA6061-T6 [15]	Friction stir processed AA6061 with multi-wall carbon nanotube [15]	Selective laser melted AlSi10Mg	Selective laser melted AlSi10Mg with multi-wall carbon nanotubes
HV	109.5	66.2	90.6	98	128.4
Standard Deviation	1.38	0.72	0.87	0.79	1.15

2.3.2 Tensile test

The results from the tensile test were tabulated (see Table 4). Friction stir processing resulted in the decrease in ultimate tensile strength and yield strength due to the dissolution of hardening precipitates [32]. This resulted in the increase in the ductility of the material as seen in the increase in elongation. The addition of multi-wall carbon nanotubes in the aluminium matrix via friction stir processing resulted in an increase in the yield strength. This is attributed to the further reduction in the grain size after the addition of multi-wall carbon nanotubes which inhibits the grain growth via Zener's pinning effect. The interaction between the pinning particles and grain boundaries is very complicated [33]. The reduction in ultimate tensile strength and elongation for the samples that underwent friction stir processing with the addition of multi-wall carbon nanotubes could have been due to having the multi-wall carbon nanotubes acting as defects resulting in stress concentration sites.

Selective laser melting of AlSi10Mg has the most desirable mechanical properties among all the samples and they are in agreement with other studies done on AlSi10Mg [22]. This is attributed to smaller grain size from the high temperature gradient during

the selective laser melting process which inhibits grain growth. The increase in mechanical strength contributed by the finer grain size could be explained using the Hall-Patch equation [14]. The fine grains together with the well-established network of hardening precipitates (see Fig. 4d) which could be explained using the Orowan effect, hence, achieving superior mechanical properties [14]. The addition of multi-wall carbon nanotubes to the aluminium matrix resulted in several visible cavities in the material. These cavities resulted in reduction of the mechanical properties. However, it is still superior than the samples that went through friction stir processing.

Table 4. Tensile results.

Tensile Properties of Al base metal and Al-CNT composites produced by friction stir processing				
Materials & Process	Ultimate Tensile Strength (MPa)	Yield Strength (MPa)	Elongation (%)	Young's Modulus (GPa)
AA6061-T6 [21]	290	240	8	69
Friction stir processed AA6061-T6	206±2	96±9	29±1	71±1
Friction stir processed AA6061 with multi-wall carbon nanotubes [15]	178±28	112±2	10±5	65±12
Selective laser melted AlSi10Mg	315±14	220±10	10±2	72±9
Selective laser melted AlSi10Mg with multi-wall	287±11	218±10	2±1	61±7

2.3.3 Fractography

Field emission scanning electron microscopy was used to examine the fracture sites of the tensile samples. Relatively larger dimples were observed in the friction stir processing of Monolithic AA6061 as compared to the selective laser melting samples. This dimple-like structures indicate that ductile fracture have taken place.

The failure of the samples that were friction stir processed and the selective laser melting of AlSi10Mg without multi-wall carbon nanotubes are believed to occur at the triple junctions or sub-grain/ grain boundaries with void initiation before rotation and sliding occur [14]. This resulted in either trans-granular or inter-granular crack propagation along the weakest path, e.g., neighbouring sub-grain/grain boundaries which in turn lead to the formation of larger cracks. Based on the observation of the failure sites of the selective laser melting parts, more ductile and finer dimples were observed at higher magnifications (see Fig. 7d).

The failure of the samples of AlSi10Mg with multi-wall carbon nanotubes is believed to have been mainly due to the presence of cavities in the material. These cavities resulted in a higher stress concentration and failure at lower strength as compared to the AlSi10Mg without multi-wall carbon nanotubes.

Upon closer examination of the fracture sites, multi-wall carbon nanotubes (some in clusters) were found on the samples. The presence of multi-wall carbon nanotubes suggested crack bridging mechanism occurring [8]. This is in agreement with the improvement in the mechanical strength for friction stir processing AA6061 with multi-wall carbon nanotubes.

Figure 6. Field emission scanning electron microscopy image of the fracture site of the friction stir processing Monolithic AA6061-T6 plates with (a) 3 passes condition [15] (b) 3 passes with multi-wall carbon nanotubes, selective laser melting of AlSi10Mg (c) without multi-wall carbon nanotubes (d) with multi-wall carbon nanotubes. Scale bar shows 1 μm .

Figure 7. Multi-wall carbon nanotubes were observed on the fracture sites for samples that underwent (a) friction stir processing and (b) selective laser melting. Scale bar shows 1 μm .

3 Conclusion

Aluminium based nano-composites reinforced with multi-wall carbon nanotubes were being fabricated using 2 methods, *viz.* friction stir processing and selective laser melting. The effect of the different processing resulted in different grain sizes and microstructures which in turn significantly improved various mechanical properties. The addition of multi-wall carbon nanotubes created pinning effects retarding the rate of grain growth causing the reduction in grain size. The presence of multi-wall carbon nanotubes has significantly improved the hardness and yield strength of the material that underwent friction stir processing. The mechanical properties of selective laser melted AlSi10Mg with multi-wall carbon nanotubes samples were not good due to the presence of cavities in the material. It is believed that selective laser melted AlSi10Mg with multi-wall carbon nanotubes samples will have superior mechanical properties if the cavities are eliminated, for example, via the following methods:

1. More uniform dispersion of the multi-wall carbon nanotubes could be achieved using longer ultrasonification duration, or by using of surfactant to aid in the dispersion of the multi-wall carbon nanotubes in the suspension.
2. As multi-wall carbon nanotubes are more ready in absorbing the laser power and have higher heat conductivity, the laser power used currently might be too high, resulting in the multi-wall carbon nanotubes to heat up to a very high

temperature. This in turn causes the aluminium in contact to vaporise and creating cavities. Therefore, a lower laser power or higher scanning speed should be used for the next study to optimise the parameters.

3. To use friction stir processing on the selective laser melting printed parts to remove porosities and cavities, and further disperse the multi-wall carbon nanotubes uniformly.

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