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Microstructure, Densification, and Compressive Properties of Al-CNT Metal Matrix Composites Fabricated by Flake Powder Metallurgy and Conventional Sintering Process

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ABSTRACT

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In this study, flake-like aluminum reinforced with different volume fractions of carbon nanotubes (CNTs) was prepared by mechanical milling and conventional sintering at 600 °C under argon atmosphere. To this end, X-Ray Diffraction (XRD) and Scanning Electron Microscopy (SEM) were used to investigate the microstructure of the powders and bulk composite samples. Compression tests were also conducted on the samples to determine their mechanical properties. The obtained results showed that flake powder metallurgy was an effective method for dispersing CNTs on the surface of Al particles. At the sintering temperature of 600 °C, the highest relative density was obtained for the composites. The compression test results showed that the amount of CNT less than 2 vol. % caused an increase in the yield and compressive strength values as well as Young's modulus. However, the values of the mentioned factors decreased in higher volume percentages. Finally, the contributions of the load bearing and grain size refinement strengthening mechanisms on the final strength of the composites were addressed.

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1. INTRODUCTION

Today, aluminum-carbon nanotube (Al-CNT) composites are praised for their mechanical properties such as their high wear and corrosion resistance, enhanced tensile strength, and high hardness as well as their low density, excellent electrical, and thermal properties, especially in automotive and aerospace industries [1-3]. Different methods have been developed to fabricate these composites, among the most significant of which are thermal sprays, powder metallurgy (mechanical mills) and sintering, hot extrusion, casting, etc. [4,5].

The distribution of CNTs, depending on the production method, affects the mechanical properties of Al-CNT composites. A number of challenges still remain in the metals reinforced with CNTs, including poor dispersion of CNTs in Al matrix due to agglomeration, low wettability between nanotubes and Al surfaces, and Van der Waals forces between CNTs [6-8].

Different methods such as ultrasonication of CNTs in a solvent before adding CNTs to the matrix, ball milling, coating of the CNTs using some specific metals, etc. have been used to better distribute the CNTs in the Al matrix [9]. Flake powder metallurgy is a new method that is extensively used to provide better distribution of CNTs

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on metal particles [1,4,6]. Given a highly specific surface area and flat surface of the powder in this method, this method facilitates the surface in situ reaction and reinforcement dispersion, thereby creating flake Nano-Grains (NG) and Ultra-Fine Grains (UFG). It is possible to retain fine microstructures in bulk materials once they are formed. In the case of the destruction of the flake shape of the powder through the powder consolidation process, either a layered configuration or a nano-reinforced body dispersion structure will be formed [10]. This method was also used for ensuring the well-distribution of CNTs in the Al matrix in previous research studies [4,7]. Jiang et al. [11] used the flake powder metallurgy that was developed to fabricate biomimetic $\text{Al}_2\text{O}_3/\text{Al}$ composites. They used nanoflake Al powders with native Al_2O_3 skins as the building blocks to rapidly assemble them into biomimetic nanolaminated structures by compacting and extrusion, thus resulting in strong and ductile composites with the tensile strength of 262 MPa and plasticity of 22.9 %. In recent years, Al-CNT composites with different CNT contents and consolidation methods have been obtained from flake powders [10,12-16]. Of note, the Al-CNT composites fabricated through this method is characterized by superior mechanical properties to those of other composites formed by conventional methods [12,14-16]. In this study, Al-CNT flake powders with different CNT content were fabricated using mechanical milling and then consolidated using uniaxial pressing and sintering at different temperatures. This study also investigated the effects of the CNT content and sintering temperature on densification and mechanical properties.

2. EXPERIMENTAL

In this study, the particle size of the aluminum powder is less than $5\ \mu\text{m}$ with the purity of $> 99.5\%$, and the outer diameter of the CNT powder is $40\ \text{nm}$ with the purity of 99% . Using a planetary mill, monolithic Al and

Al-2 vol. % CNT, Al-4 vol. % CNT, and Al-8 vol. % CNT powders were separately milled for three hours to achieve homogeneous nanotube distribution. Here, the ball-to-powder ratio and rotational speed values were obtained as 10:1 and 200 rpm, respectively, and 12 mm balls, stainless-steel balls, and cups were used. Followed by milling, the samples were compressed at the pressure of 1 GPa and then sintered at the temperature of $600\ ^\circ\text{C}$ under Ar atmosphere for an hour. The inner diameter of the steel mold was 6 mm. The green samples were prepared by compressing the die at the load of 2881.7 Kgf. A D8 Advance Bruker diffractometer and Mira 3-XMU Field Emission Scanning Electron Microscopy (FE-SEM) were employed to investigate the microstructure of the materials. In order to record the XRD patterns using Cu $K\alpha$ radiation, step size of 0.02° were used. Archimedes method was also used to determine the density values, and the compressive test with a speed of 1 mm/min was done to determine the mechanical properties. For compression tests, cylindrical specimens with diameters of 6 mm and heights of 9 mm were used. The obtained results from the three tests conducted on each sample were then averaged and taken into consideration.

3. RESULTS AND DISCUSSION

3.1. Microstructure of the Milled Powders Ation

Figure 1 shows the SEM images of primary Al, three-hour-milled Al and Al-8 vol. % CNT composite powders. High-energy milling repeatedly involves fattening, cold welding, fractureing, and rewelding of powder particles [17]. In the case of a collision between two steel balls, a small amount of CNT powders would be trapped in between Al powders. In addition, plastic deformation of the powder particles occurs as a result of the impact force, hence flattening, hardening, and fracture of particles. By creating new surfaces, the particles can weld together,

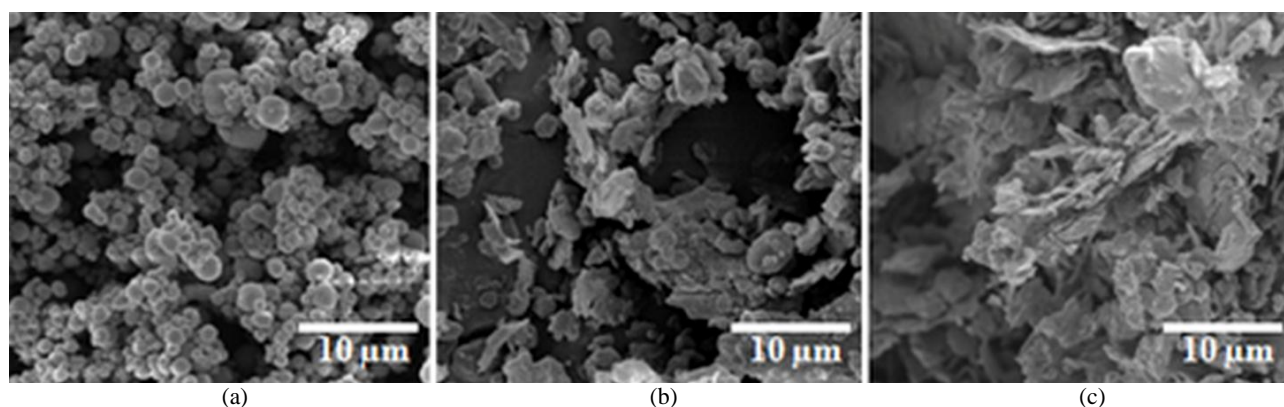
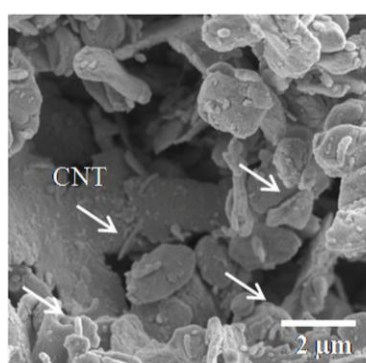


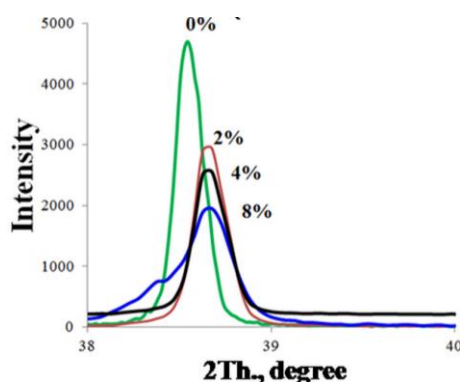
Figure 1. Morphology of (a) as received Al, (b) Milled Al for 3h, and (c) milled Al-8 vol. % CNT for 3h

thus resulting in their larger size [18,19]. In the early stages of milling (three-hour milling time in this study), the Al particles are soft and consequently, they tend to weld together and form large particles. In this stage, the composite particles have a flake structure composed of a mixture of the constituents. Figure 1a shows the Al powder with spherical morphology. Figures 1b and 1c show the Al and Al-8 vol. % CNT powders, respectively, after three hours of milling.

As a result of milling, flake-shaped powders will be formed. High surface area flakes facilitate CNT dispersion. In Figure 2a, high magnification SEM



(a)



(b)

Figure 2. High magnification SEM micrograph of milled Al-2 vol. % CNT powder showing the dispersion of CNTs on flake powders, and (b) XRD patterns of the samples from the (111) planes

3.2. Sintering Behavior and Microstructure

The milled powders were compressed at the pressure of 1 GPa and then sintered at the temperature of 600 °C. Table 1 shows the density and relative density of the samples measured by Archimedes method.

Table 1. Density and grain size of the bulk samples with different CNT contents

Material	Density (g/cm ³)	Relative Density	Grain Size (nm)
Al	2.47	91.4	220
Al-2 vol. % CNT	2.38	88.9	163
Al-4 vol. % CNT	2.32	87.5	161
Al-8 vol. % CNT	2.28	87.6	156

As seen, the density and relative density of Al decreased by adding CNTs which can be attributed to the presence of some CNT agglomerates in the structure and poor bonding of CNTs with the matrix. This behaviour was already reported for Al-CNT composites prepared through other powder metallurgy methods [4].

Figure 3 shows the microstructure of Al-CNT composites with polished and etched surfaces. As observed in this figure, CNT content caused a decrease in the grain size of Al matrix. In addition, the grain size of the Al matrix

micrographs show the dispersion of CNTs on the surface of flake Al powders. According to this figure, CNTs were well dispersed. Figure 2b shows the XRD patterns for samples in the (111) planes. The peak in Al with high CNT content is wider and less intense than the others. During three hours of ball milling, addition of CNT to Al facilitated the microstructural evolution. In the presence of CNT reinforcement, the Al matrix undergoes inhomogeneous local deformation which results in a higher work hardening rate, hence finer crystallite sizes of the milled powder, as already reported in previous studies [1,4,8].

remains in the submicron range where the CNT content is a determining factor.

Based on the SEM micrographs, the average grain sizes of the samples were determined using the intercept method. Table 1 presents the grain size values for different samples. The decrease in the grain size of the composite samples is the result of pinning effect of CNT during sintering [20].

Figure 4 demonstrates the XRD patterns of the sintered samples. Due to the small volume fraction of CNTs and their nanometer size, only the peaks related to FCC Al can be observed. According to this figure, the intensity of the diffraction peaks decreases as the CNT volume % increases.

3.3. Compressive Strength and Strengthening Mechanisms

Figure 5 presents the compressive stress-strain response of the sintered samples with different CNT vol. %. Table 2 lists the properties of the materials based on the stress-strain curves given in Figure 5. Although a small amount of CNTs (less than 8 vol. %) was added to the matrix, the compressive strength was significantly enhanced. Under the same processing, Al-2 vol. % CNT composite showed a 30 % increase in the compressive strength compared to that of monolithic Al.

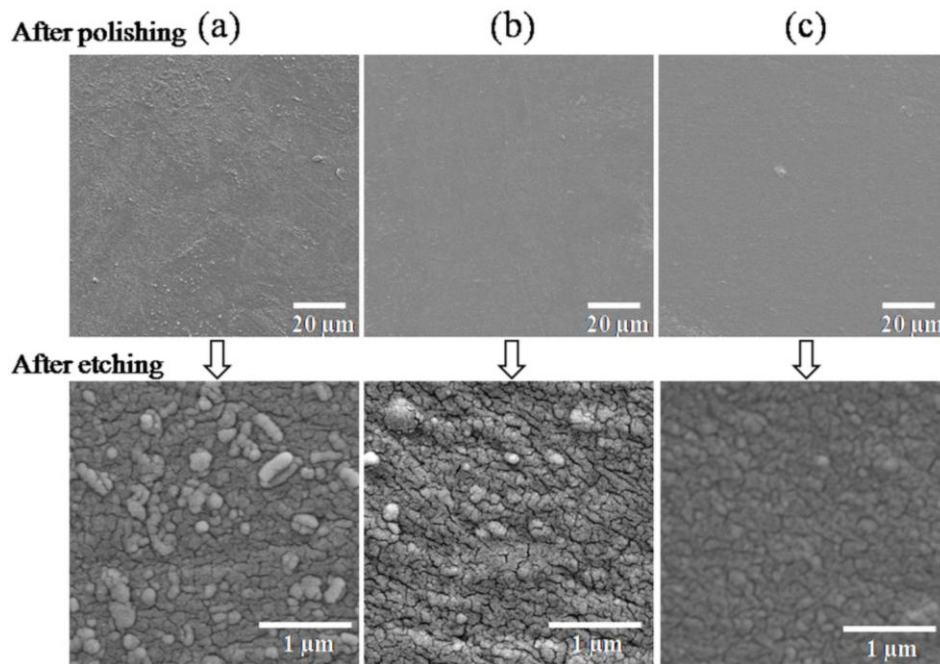


Figure 3. High magnification SEM micrograph of milled Al-2 vol. % CNT powder showing the dispersion of CNTs on flake powders, and (b) XRD patterns of the samples from the (111) planes

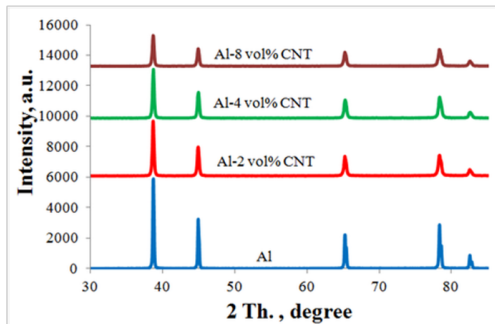


Figure 4. XRD patterns of sintered samples

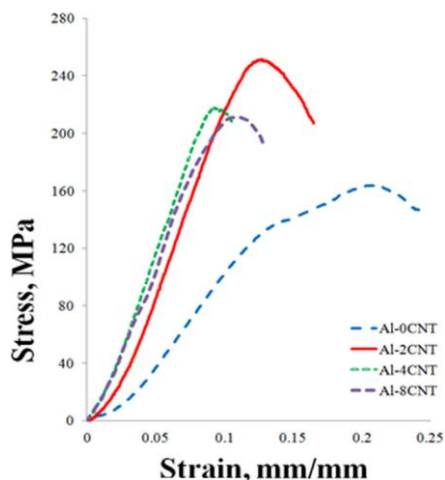


Figure 5. Compressive stress-strain curves of the sintered samples with different CNT vol. %

Table 2. Grain size and strength of the sintered materials

Material	Yield strength (MPa)	Compressive Strength (MPa)	Grain Size (nm)
Al	128.5±8.1	163.8±13.7	220±34
Al-2 vol. % CNT	222.4±14.5	251.3±11.9	163±29
Al-4 vol. % CNT	214.9±11.4	217.6±8.3	161±17
Al-8 vol. % CNT	196.2±16.0	210.9±18.2	156±25

The conventional sintering method resulted in lower strength in samples prepared by flake powders than that of the hot pressed samples [4]. Sintering temperature and time are important factors that determine this difference. hot pressed samples succeeded in obtaining finer structure of the matrix phase due to their lower temperature and shorter soaking time than those of other samples. In addition, the density of the samples plays a role in making this difference. Further, as the compressive strength of the composite increased, its ductility would decrease. Reduced dislocation mobility would lead to decreased ductility.

It should be noted that the CNT content of Al-2 vol. % provided the highest strength. As the CNT content surpassed 2 vol. %, the properties would significantly decrease in comparison to the Al-2 % CNT samples. A decrease in the Al-CNT composite properties at higher CNT contents resulted from the CNT dispersion problems under high reinforcement loads. The results from the Al-CNT samples prepared through different processes [4,21-29] were compared with the optimal results from this study, the summary of which are given in Figure 6. According to the findings, severe plastic

deformations and low temperatures (or short processing times) lead to an improvement in the Al-CNT composite strength mainly due to the refinement of the matrix phase that was altered throughout these processes. In most

methods, strength increases at the expense of ductility. On the contrary, those methods that are based on flake powder metallurgy can ensure less ductility reduction.

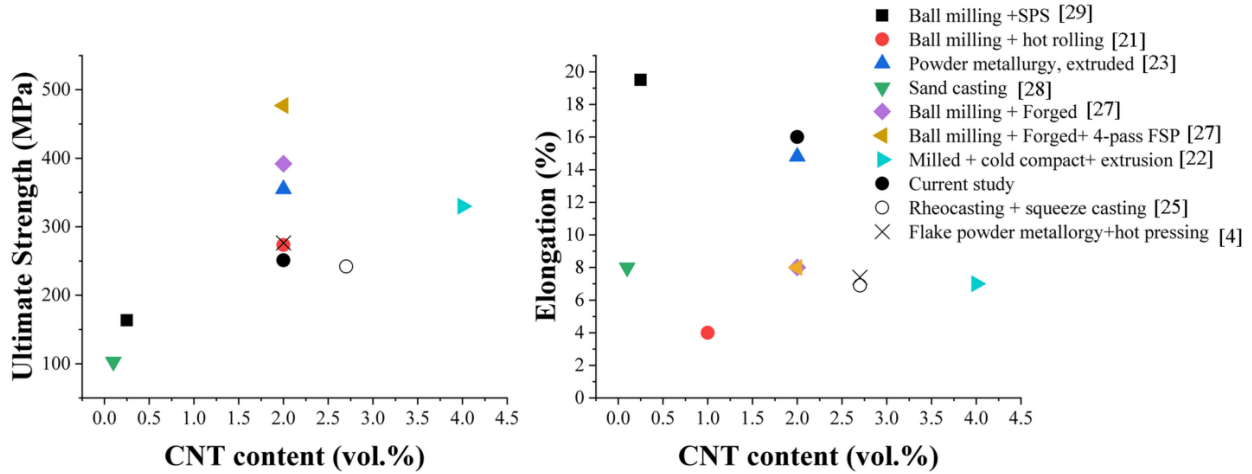


Figure 6. Comparison of strength and ductility of Al-CNT composites prepared by various processes

Two main strengthening mechanisms of load bearing effect and grain size refinement are considered for Al-CNT prepared by the conventional sintering methods. As a result of the high aspect ratio of the CNT, load is transferred from the matrix to the CNTs. According to the Hall-Petch relationship, strength increases as the matrix grain size decreases.

Followed by superimposing two mechanisms contributions, Clyne method calculates yield strength of the composite as follows [30]:

$$\sigma_y = \sigma_0 + \sqrt{\Delta\sigma_{H-P}^2 + \Delta\sigma_{LT}^2} \tag{1}$$

where σ_0 stands for the intrinsic yield stress (9.8 MPa for Al) [4], $\Delta\sigma_{H-P}$ the grain size effect (Hall-Petch effect), and $\Delta\sigma_{LT}$ the load bearing effect of the CNTs. Based on the equations below, the contribution of each mechanism can be determined. In these equations, some parameters were extracted from the references [4,8].

$$\Delta\sigma_{H-P} = \frac{k}{\sqrt{d}} \tag{2}$$

$$\Delta\sigma_{LT} = V_f\sigma_m \frac{A(l+t)}{4l} \tag{3}$$

where V_f represents the volume fraction of CNTs, and σ_m the yield strength of the unreinforced Al matrix (128.5 MPa). In addition, l and t are the dimensions of the particulate parallel to parallel and perpendicular to the

compression direction ($l=4 \mu\text{m}$ and $t=60 \text{ nm}$), respectively. The experimental yield strength of Al is used to determine K where A is the aspect ratio of CNTs (66.66) and b the Burgers vector of Al matrix (0.35 nm).

Table 3 presents the results of theoretical calculations of yield stress for the produced nanocomposites. Based on this Table, reducing the grain size or the Hall-Petch mechanism has a greater theoretical contribution than other mechanisms.

Based on the assumptions in theoretical calculations, it can be concluded that the calculated yield stress shows a relatively better fit with the experimental results in the cases of Al and Al-2 vol. % CNT. It is evident that the difference between the theoretical and experimental values is high for CNT content over 2 vol. %.

It should be noted that the probability distribution of the CNTs and relative microstructure heterogeneity are important factors that can better elaborate this difference.

Table 3. Al-CNT composites yield strength and contribution of different strengthening mechanisms

Material	$\Delta\sigma_{H-P}$ (MPa)	$\Delta\sigma_{LT}$ (MPa)	σ_y (MPa)
Al	149.0	-	159.0
Al-2 vol. % CNT	173.3	43.4	226.6
Al-4 vol. % CNT	170.2	86.9	267.0
Al-8 vol. % CNT	177.9	173.8	360.9

4. CONCLUSIONS

1. In this study, Aluminum reinforced with different volume fractions of CNTs was prepared by flake

powder metallurgy through short time milling and conventional sintering methods.

- The flake powder metallurgy method provided good dispersion of CNTs on the surface of Al particles.
- The Al-2 vol. % CNT sintered at 600 °C obtained the highest relative density.
- The compression test results showed that the CNT volumes less than 2 % led to an increase in the yield and compressive strength values and a higher Young's modulus. However, in higher percentages, the values of these properties would decrease.
- The contributions of the load bearing and grain size refinement strengthening mechanisms to the final strength of the composites were also investigated, and it was found that the grain size refinement was the dominant strengthening mechanism for Al-CNT composites.

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