FABRICATION OF NANO CABONS REINFORCED METAL MATRIX COMPOSITES BY LIQUID PRESSING PROCESS

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Abstract

Nano carbons reinforced aluminium (Al) alloy composites have been fabricated successfully by the surface treatment of nano carbons and the unique casting method, liquid pressing process. CNTs and CNFs have been coated with metal and/or oxide in order to improve the wettability between nano carbons and metallic melts. In liquid pressing process, Al alloy melts have been pressed hydrostatically and infiltrated on nano carbons surfaces. Based on the microstructures of the composites have been analyzed with SEM, the surface treated CNFs were dispersed homogeneously in the matrix. Also, mixed particles with 10 vol. % CNFs and 10 vol. % sub-micron sized SiC reinforced aluminum composite had a sound microstructure. The elastic modulus, compressive strength, and tensile strength of composite were 104 GPa, 585 MPa, and 261 MPa, respectively.

1 Introduction

Nano carbon materials such as carbon nanotubes (CNTs), carbon nanofibers (CNFs), and graphene have been promising reinforcements for light metallic matrices due to their excellent specific strength, specific modulus, and thermal and electrical conductivities. However, fabrication of nano carbons reinforced metal matrix composites (MMCs) is very challenging due to their poor wettability. Because of the difficulties in introducing carbon materials into metal melts, most work used powder metallurgy (PM) techniques [1-2].

When fabricating nano carbons reinforced metal matrix composites (MMCs), they are critical to disperse the nano carbons in metallic melt and to maintain the stability of nano carbons under atmosphere due to strong reactivity of carbon with oxygen. In order to improve the stability of nano carbons and the wettability between nano carbons and metallic melt, Nano carbons have been coated with metal and/or oxide [3]. The squeeze casting process, one of conventional casting methods for composites, has merits such as high productivity and easiness for near-net-shape fabrication, but has shortcomings of poor reliability, requirement of high-pressure loading of 50 MPa or more in order to enhance the wettability between reinforcements and matrix. To effectively fabricate the metal matrix composites reinforced with nano carbons, it is thus necessary to introduce new-concept fabricating processes, one of which is a liquid pressing process [3-4] using low pressure near to the theoretically required minimum loading pressure. In this process, thin-plate-type composites can be readily

fabricated, and the crystallization of the amorphous matrix can be prevented or minimized by rapid cooling of the melt.
In the present study, nano carbons reinforced aluminium (Al) alloy composites have been fabricated successfully by the surface treatment of nano carbons and the unique casting method, liquid pressing process. CNTs and CNFs have been coated with metals and oxides in order to improve the wettability between nano carbons and metallic melts. In liquid pressing process, Al alloy melts have been pressed hydrostatically and infiltrated on nano carbons surfaces. Microstructures of the composites have been analyzed. The surface treated CNTs and CNFs were dispersed homogeneously in the matrix. Their mechanical properties have also been evaluated by tensile and compressive tests.

2 Experimental

The multi-wall carbon nanotubes (CNTs) and the vapor grown carbon nanofibers (CNFs) supplied by Applied Carbon Nanotechnology Co. (Korea) and Showa Denko (Japan), respectively, were used as the reinforcements. Also, submicron sized Silicone carbide (SiC) particles supplied by Nilaco (Japan) were additional reinforcement. Table 1 lists the physical and mechanical properties of CNTs, CNFs, SiC and A356.

Materials	Density	Elastic Modulus [GPa]	Tensile Strength [MPa]	Diameter [nm]	Length [μm]
CNT	1.4	1,000	50,000	10~15	1~3
CNF	2.0	240	3,000	150~200	~10
SiC	3.21	430	-	-	0.5
A356	2.69	72	220	-	-

Table 1. Physical and mechanical properties of CNTs, CNFs, and A356 aluminium alloy.

Titanium oxide (TiO_2) coated CNTs particles were synthesized by Sol-Gel method. First, CNTs were cleaned and dispersed in ethanol by a beam-type ultrasonicator for 10 min. Benzyl alcohol as a surfactant and distilled water were then poured into the well dispersed CNTs/ethanol mixture. Titanium butoxide (TNBT) was used as a precursor of TiO_2. TNBT/ethanol solution was added in CNTs/ethanol mixture slowly by a dropping funnel. After reaction, the mixture was filtered and washed. After Sol-Gel process, the particles underwent heat treatment process for crystalline structure under argon atmosphere.
Nickel coated CNF particles were synthesized by electroless plating. First, CNFs were cleaned and dispersed in ethanol and deionized water, respectively. The pre-treated CNFs were then sensitized and activated in an aqueous solution containing $SnCl_2$, $PdCl_2$, and HCl. The activated CNFs were accelerated in an aqueous solution with sulfuric acid. And then, the CNFs mixture was poured in a nickel plating bath with a mechanical stirrer. After plating process, the particles underwent heat treatment under argon atmosphere. The synthesized particles were observed by for scanning electron microscope (SEM). Their phases were analyzed by X-ray diffraction (XRD).
The composites were fabricated by the liquid pressing process [3-4]. The mold interior was sized by 60×60×6 mm. The prepared particles and A356 master alloy plates were inserted into the mold, degassed, and evacuated by a mechanical vacuum pump. The mold was heated to 870^{o}C, held for 5 minutes, and then pressed under a pressure of approximately 10 MPa. The fabricated composites were sectioned, polished for scanning electron microscope (SEM)

observations. The composites were also machined into sub-sized dog-bone and rectangular shape specimens for tensile and compressive tests.

2 Results and discussion

The minimum infiltration pressure of aluminium alloy melt into preform of nano carbons was interpreted by melt flow theory on the basis of triangle array and square array of reinforcements. Figure 1 show the results of minimum infiltration pressure of aluminium alloy melt into nano carbon preforms. From preliminary experiments, it was confirmed that the volume fraction of the preform of nano carbons increased approximately 55% due to compaction by applied pressure. In case of particle size, the minimum infiltration pressures of CNTs (15nm) and CNFs (150nm) were about 90MPa and 9MPa, respectively. In other words, the infiltration pressure is in inverse proportion to the particle size. As a decrease of wetting angle of 110°, the infiltration pressure decreased from 90MPa to 35MPa.

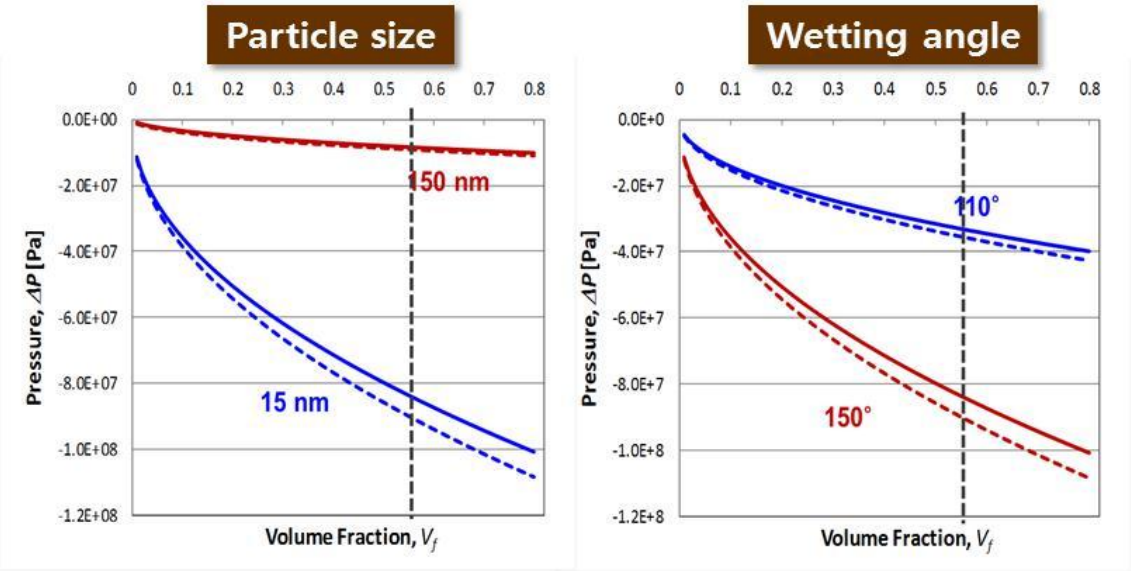

Figure 1. Calculated results of minimum infiltration of aluminium alloy melt into nano carbon preforms as functions of particle size at same wetting angle of 150° and wetting angle at same particle size of 15nm. Preform volume fraction: 55%; Particle size: CNTs of 15nm and CNF of 150nm; Wetting angle: 110° and 150°

Figures 2 (a) and (b) show the as-coated CNTs and heat-treated TiO_2 coated CNTs. The TiO_2 coating layer was uniformly distributed with the typical thickness of approximately 25nm. From XRD analysis, the as-coated layer was amorphous phase and the heat-treated layer under argon atmosphere at 450°C was anatage crystalline phase.

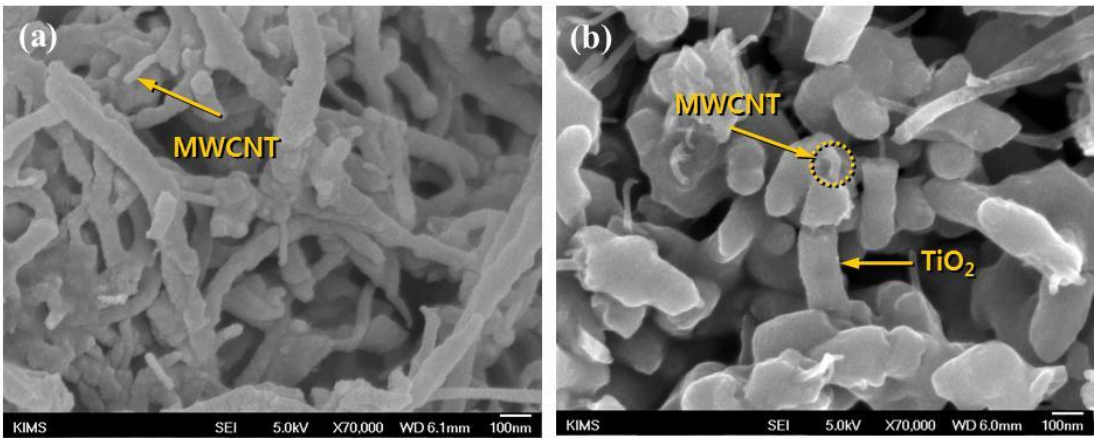

Figure 2. SEM images of (a) as-coated CNTs and (b) heat-treated CNTs

Figures 3 (a) and (b) show the as-received CNFs and nickel plated CNFs. The Ni coating layer was uniformly distributed with the typical thickness of approximately 100nm. The XRD analysis results of as-plated CNFs and heat-treated Ni plated CNFs at 400oC, 600oC, 800oC under argon atmosphere were shown in Fig. 4. The as-plated layer was amorphous phase with small amount of cryatalline phase. After heat-treatment, the amorphous phase was transformed to two major crystalline phases of fcc Ni and Ni_3P. Also, there was small amount of NiO from oxidation.

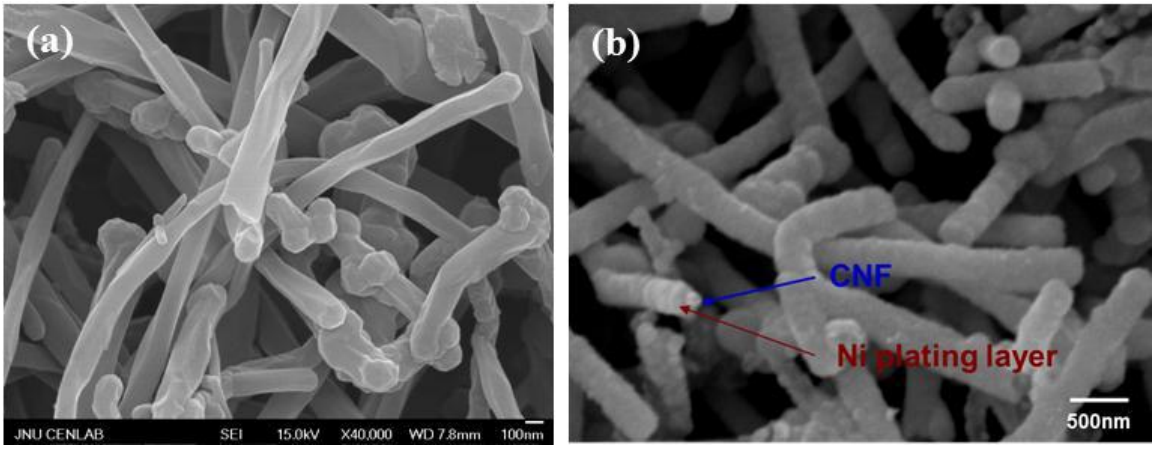

Figure 3. SEM images of (a) as-received CNFs and (b) Ni plated CNFs

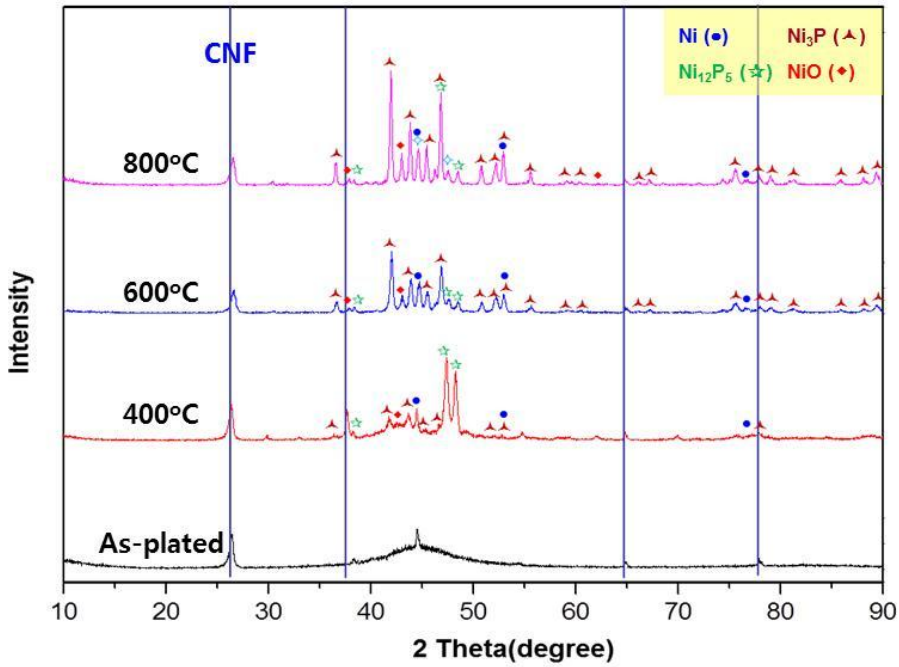

Figure 4. XRD analysis results of as-plated CNFs and heat-treated Ni plated CNFs at 400oC, 600oC, 800oC under argon atmosphere

Figures 5 (a) and (b) show low and high magnification SEM micrographs of only 10 vol. % CNFs reinforced A356 composite. There were lot of CNFs clusters and Most CNFs were not wetted with aluminium alloy even though small amount of alloy melt was infiltrated into some CNFs cluster. Figures 5 (c) and (d) show low and high magnification SEM micrographs

of 10 vol. % CNFs and 10 vol. % SiC reinforced A356 composite. The reinforcements of CNFs and SiC were homogeneously distributed in the aluminium matrix. The defects formed by misinfiltration or reaction products formed by interfacial reaction at fiber/matrix interfaces are hardly found. The physical and mechanical properties were summarized in Table 2. The elastic modulus of composite of 104 GPa is about 50% higher than that of A356 alloy. Also, the compressive strength was also much higher from CNFs and SiC reinforcements, even though the tensile strength was increased slightly.

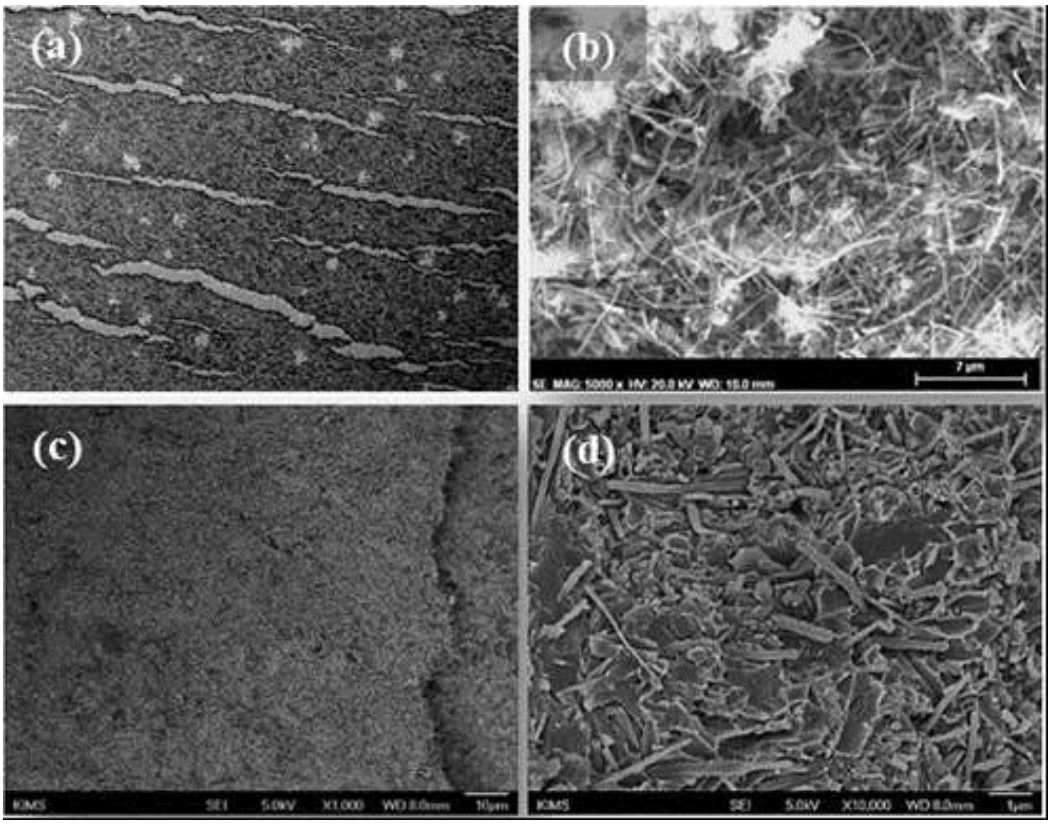

Figure 4. SEM images: (a) and (b) CNFs reinforce A356 composite; (c) and (d) CNFs+SiC reinforced A356 composite

Specimen	Density	Elastic Modulus [GPa]	Tensile Strength [MPa]	Compressive Strength [MPa]
CNF+SiC/ A356	2.69	104	261	585

Table 2. Physical and mechanical properties of 10 vol. % CNFs and 10 vol. % SiC reinforced A356 composite.

Summary

A356 aluminium alloy composites reinforced with nano carbons such as CNTs and CNFs have been fabricated successfully by the surface treatment of nano carbons and the liquid pressing process. CNTs and CNFs have been coated with metal and/or oxide in order to improve the wettability between nano carbons and metallic melts. The Ni plated CNFs and the mixture of CNFs and SiC were dispersed homogeneously in the aluminium matrix. Also, mixed particles with 10 vol. % CNFs and 10 vol. % sub-micron sized SiC reinforced aluminum composite had a sound microstructure and excellent mechanical properties; elastic modulus of 104 GPa, compressive strength of 585 MPa, tensile strength of 261 MPa.

Acknowledgement

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Nano carbons/aluminiu alloy composites been fabricated by the liquid pressing process. CNTs and CNFs have been coated with metal and/or oxide in order to improve the wettability. 10 vol. % CNFs and 10 vol. % SiC reinforced aluminum composite had a sound microstructure and excellent mechanical properties.