

WEAR CHARACTERIZATION OF CNF/Al COMPOSITES FABRICATED BY LIQUID PROCESS

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Abstract

Carbon nanofiber (CNF) reinforced Al matrix composites were fabricated by a liquid process and the wear properties of the CNF/A7xxx composites were investigated using a pin-on-disk tester at dry condition. To enhance the dispersion of CNFs in the A7xxx matrix, the aspect ratio of CNFs were reduced by vibration milling and Cu layer was coated on the surface of CNFs. The effects of the concentration of CNFs and the applied load on wear properties of the CNF/A7xxx composites were studied using the measurement of wear loss and the examination of worn surfaces. The wear loss of the composites showed a steadily decreasing trend with weight fraction of CNF in the composites, which is due to not only the excellent mechanical properties of CNF and the uniform dispersion of CNFs in A7xxx matrix, but also the lubrication effect of the CNFs exposed on the worn surfaces.

1 Introduction

Carbon nanofiber (CNF) with very small diameter of 100~150 nm has been regarded as a promising reinforcement material for composites owing to its outstanding properties of high elastic modulus, high strength, and superior electrical and thermal conductivity [1-2]. However, its entanglement due to Van der Waals interaction holds back its uniform distribution in aluminum matrix. In addition, its poor wetting property on aluminum and difference in density between CNF and aluminum have been great obstacles for manufacturing composites. Up to now, due to the obstacles above, a large number of CNF reinforced metal matrix composites have been fabricated via solid state process i.e. powder metallurgy [3-5]. The powder metallurgy process can yield well dispersed CNF reinforced metal matrix composites, while it cannot be easily applied to large scale products and is more costly than the liquid process. In contrast, the liquid process is simple and cheap, and can fabricate large scale composite billets. However, most investigations have focused on the fabrication process of the composites and the mechanical or thermal properties. Very few results have been reported on wear characteristics of CNF/Al composites.

In this study, A7xxx matrix composites reinforced by CNFs were fabricated by a liquid process such as the melt stirring method. To overcome the entanglement of the CNFs and disperse them uniformly in the Al matrix, we decreased the aspect ratio of the CNFs by vibration milling, and used a surfactant and applied ultrasonication. Also, a Cu layer was coated on the surface of the CNFs to improve the wetting of molten Al on the CNFs and

decrease the floating of the CNFs on the surface of the Al melt. The wear properties of the CNF/A7xxx composites were characterized using a pin-on-disk wear tester. Effects of the concentration of CNFs on wear properties of the composites were investigated using the measurement of wear loss and the examination of worn surfaces.

2 Experimental procedures

Carbon nanofibers (Showa Denko Co., Ltd. in Japan) with diameters of ~130 nm and lengths of ~10 μm were prepared by catalytic chemical vapor deposition. To dissolve the entanglement of CNFs and disperse them uniformly in an Al matrix, vibration milling was used, in which the CNFs were cut to reduce their aspect ratio. To enhance the dispersion of the CNFs, the vibration-milled CNFs were stirred using ultrasonication in distilled water containing poly carbon acid amine as a surfactant. The dispersed CNFs were sensitized and activated in SnCl_2 and PdCl_2 solutions, respectively, at room temperature. And then, the pre-treated CNFs were filtered and washed sufficiently in distilled water. Then, a Cu layer was coated on the surfaces of the pre-treated CNFs in CuSO_4 solutions at 45 °C. After deposition of the Cu layer on CNFs, the Cu-coated CNFs were mixed with pure Al powders using an impeller in ethanol, and then dried in a vacuum oven. From the dried mixed powder (5 wt% CNF + 95 wt% pure Al powders), button-shaped feedstocks of 20 mm in diameter and 15 mm in height were fabricated by vacuum hot press at 560 °C and 25 MPa. The hot pressed CNF/Al feedstocks were inserted into the A7xxx melt of 500g at 700°C. The A7xxx matrix alloy was supplied from Alutec Co., Ltd. (Korea), and its chemical composition is represented in Table 1. After complete melting of the feedstocks, the CNF/A7xxx composite melt was agitated using a mechanical stirrer for 3 min in N_2 atmosphere, and then cast in a water cooled Cu mold of 50 mm in diameter. The concentration of the CNFs in CNF/A7xxx composites was controlled by the number of feedstocks. The cast CNF/A7xxx composites were hot-extruded at 400 °C to align the CNFs in the direction parallel to the extrusion direction using an extrusion ratio of 15 : 1 at a pressure of 110 ton. The extruded CNF/A7xxx composites were solution-treated at 450 °C for 1 hr and quenched in water, and then aged at 120 °C for 32 hr (T6 heat treatment).

The hardness values of the aged specimens were measured by a micro Vickers hardness tester (Matsuzawa Seiki Co., Ltd.) under an indentation load of 100gf. Their wear behavior was characterized using a pin-on-disk wear tester at dry sliding condition [6]. The aged CNF/A7xxx composites were machined as pin specimens with a diameter of 5 mm and a length of 25 mm. The wear tests were carried out for 2 hr at a sliding speed of 100 rpm (0.2094 m/s) under applied loads of 5~20 N using a rotating disk made of SKD 11 steel. The effects of CNF reinforcement on wear resistance of the CNF/A7xxx composites were evaluated from the measurement of wear loss and the examination of worn surfaces. The distribution of CNFs in Al matrix and the morphologies of the Cu coated CNFs and the worn surfaces were observed by a field emission scanning electron microscopy (FE-SEM).

3 Results and discussion

CNF/A7xxx nanocomposites with uniformly dispersed CNFs are difficult to fabricate by liquid processing, since the wettability of Al on the CNF is poor, CNFs float on the surface

Element	Zn	Mg	Si	Fe	Cr	Cu	Al
Wt%	4.69	1.31	0.23	0.21	0.04	0.026	rest

Table 1. Chemical composition of A7xxx alloy (wt%).

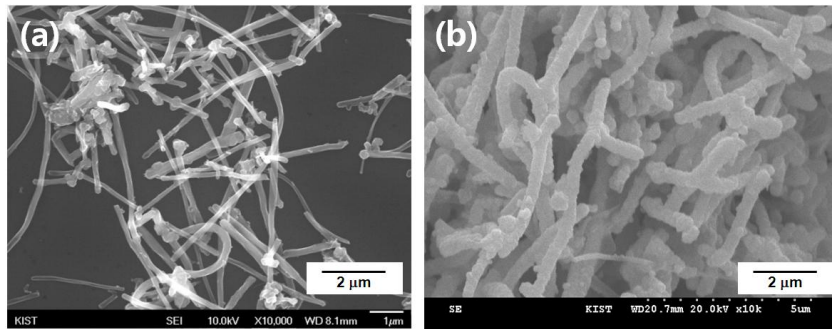


Figure 1. SEM micrographs of (a) vibration-milled and (b) Cu coated CNFs.

of the Al melt due to the difference in their specific gravities, and brittle Al_4C_3 phases emerge by reaction between CNF and Al matrix. In our work, to resolve these difficulties, a Cu layer was coated on the surface of the CNFs. Fig. 1 shows the CNFs before and after Cu coating on CNFs by electroless plating. As shown in Fig. 1(b), uniform Cu layers were coated on CNFs. Fig. 2 represents the process for fabrication of the CNF/A7xxx composites by a liquid processing. The direct injection of Cu-coated CNF powders into the Al melt is very difficult because the thermal convection above the surface of the Al melt disturbs the contact of the Cu-coated CNFs with the Al melt. So, for effective injection of the Cu-coated CNFs into the Al melt, button shaped feedstocks were fabricated by a vacuum hot press using a mixture of Cu-coated CNFs and pure Al powders. Fig. 2(a) and (b) show the mixed powders of Cu-coated CNFs and pure Al powders and the CNF/Al feedstock fabricated with the mixed powders, respectively. By inserting the CNF/Al feedstocks into the Al melt and stirring the melt by impeller as shown in Fig. 2(c), CNF/A7xxx composite billets could be easily fabricated. The cast CNF/A7xxx nanocomposite billets were hot-extruded to align the CNFs in the direction parallel to the extrusion direction.

To confirm whether the CNFs were well dispersed in the A7xxx matrix, the fractured surfaces of the composites were examined using a FE-SEM. Fig. 3 represents the SEM images of the fractured surfaces of the A7xxx alloy and the CNF/A7xxx composites. As shown in Fig. 3, the CNFs in A7xxx matrix are dispersed relatively uniformly in the A7xxx matrix and aligned in the direction parallel to the extrusion direction. This uniform alignment of CNFs was due to not only the vibration milling, the addition of surfactant, the ultrasonic agitation, and the Cu coating on the surface of CNFs, but also the extrusion of the composite billets.

The hardness values of the CNF/A7xxx composites measured by a micro Vickers hardness tester is represented in Fig. 4, in which, a considerable increase in hardness value was observed by addition of CNFs in A7xxx matrix. The remarkable enhancement of hardness by addition of CNFs is originated from the uniform dispersion of the CNF reinforcement in the

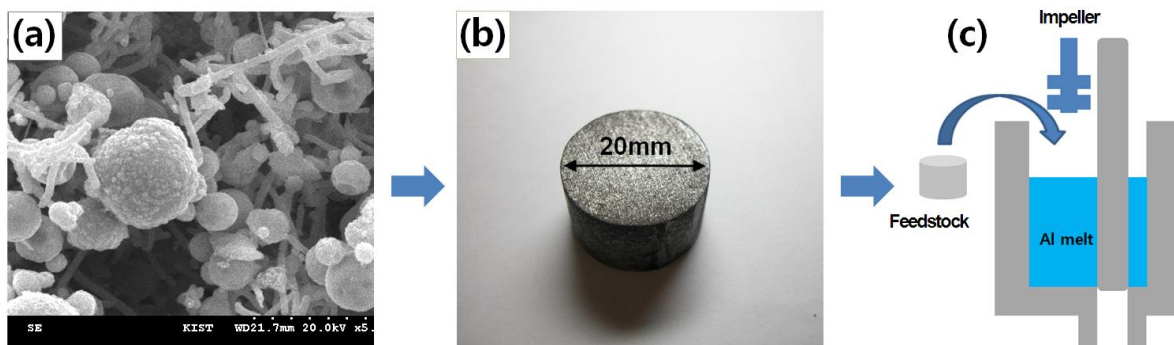


Figure 2. Fabrication process of CNF/A7xxx composites by a liquid processing: (a) SEM image of a mixture of Cu coated CNFs and Al powders, (b) a feedstock fabricated by vacuum hot press, and (C) casting method of the composites by melt stirring.

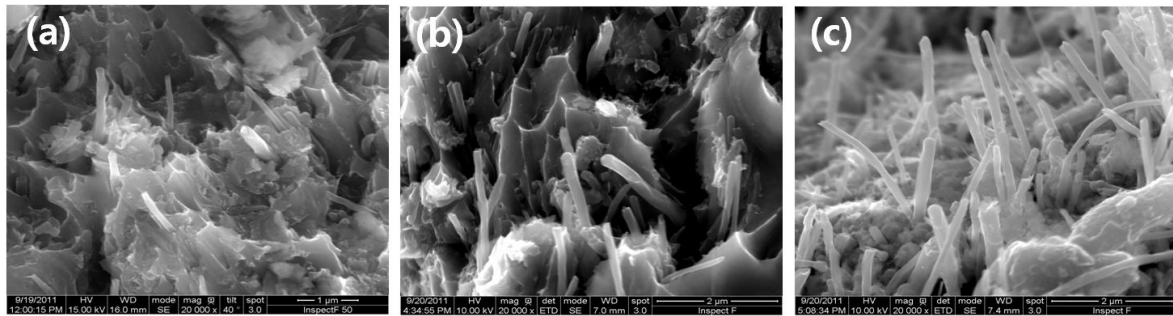


Figure 3. SEM micrographs of the fractured surfaces of (a) 0.43 wt%, (b) 0.59 wt%, and (c) 0.76 wt% CNF/A7xxx composites.

A7xxx matrix. Therefore, the CNFs were very effective in mitigating the movement of dislocations.

Fig. 5(a) and (b) shows the variations of wear loss of the CNF/A7xxx composites with weight fraction of CNF and applied load, respectively. As shown in Fig. 5, the wear loss of the composites showed a steadily decreasing trend with weight fraction of CNF in the composites, and also the wear loss of the composites increased with applied load. It is considered that the favourable effects of CNF on wear resistance of the composites are attributed to its excellent mechanical properties and uniform dispersion of CNFs in Al matrix. At the same time, the CNFs exposed on

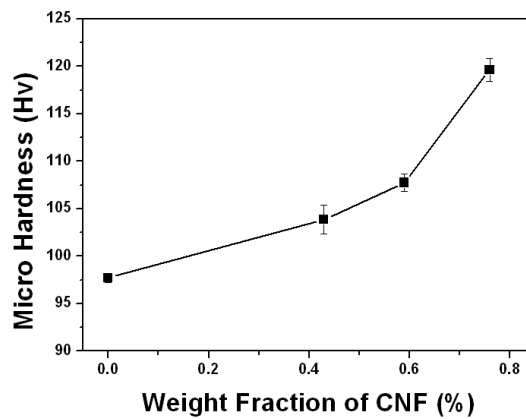


Figure 4. Variation of micro Vickers hardness of CNF/A7xxx composites with weight fraction of CNF.

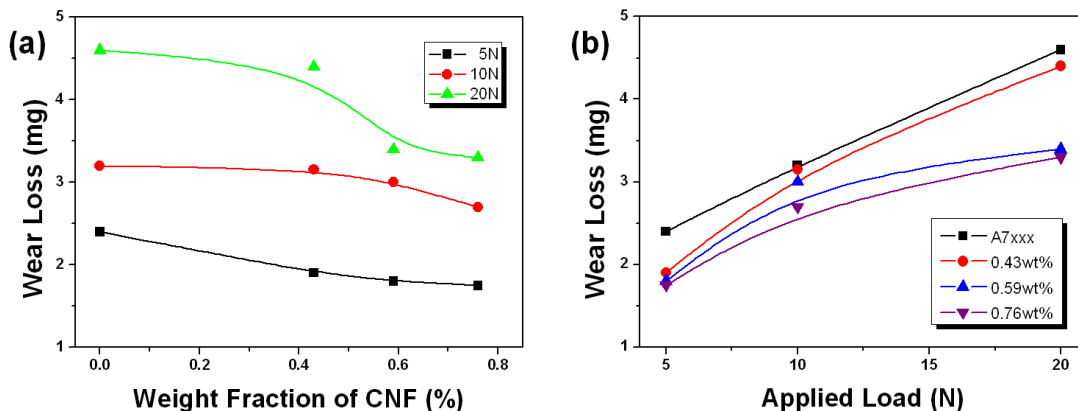


Figure 5. Variations of wear loss of CNF/A7xxx composites with (a) the weight fraction of CNF and (b) the applied load.

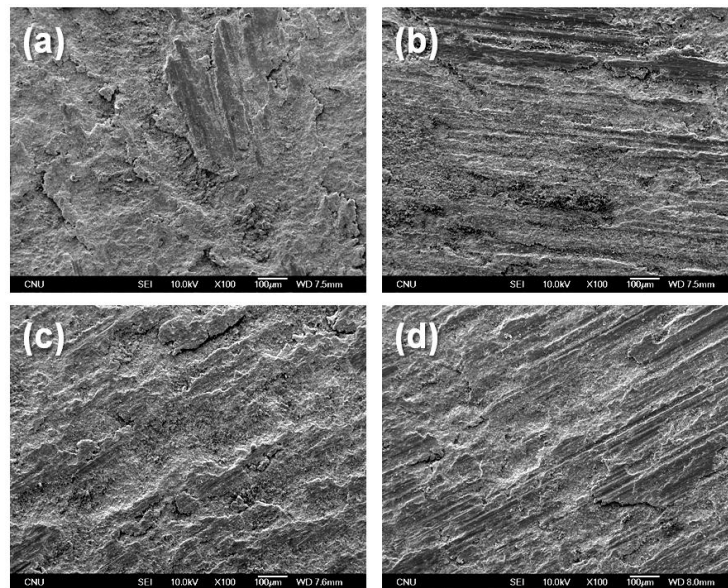


Figure 6. Variations SEM micrographs of worn surfaces of (a) A7xxx alloy and the CNF/A7xxx composites with (b) 0.43 wt% CNF, (c) 0.59 wt% CNF, and (d) 0.76 wt% CNF under load of 20 N.

the worn surfaces during wear test could act as a lubricating agent between the Al matrix and the counter part steel disk. The effects were more remarkable under high applied load than low applied load, i.e., the decrease of wear loss as increasing weight fraction of CNF under applied load of 5N was small, while it was large under applied load of 20N.

Fig. 6 shows the microstructure of the worn surfaces of the A7xxx alloy and the CNF/A7xxx composites with different CNF weight concentration tested under load of 20N, indicating that the A7xxx alloy revealed severe plastic deformation and flake formation. The flake formation occurs when a highly deformed layer formed on the specimen surface during dry sliding wear. However, the addition of CNF into A7xxx matrix showed less plastic deformation and flake formation on the surfaces, which implies the enhancement of the wear resistance of CNF/A7xxx composites due to their strengthening effect [7].

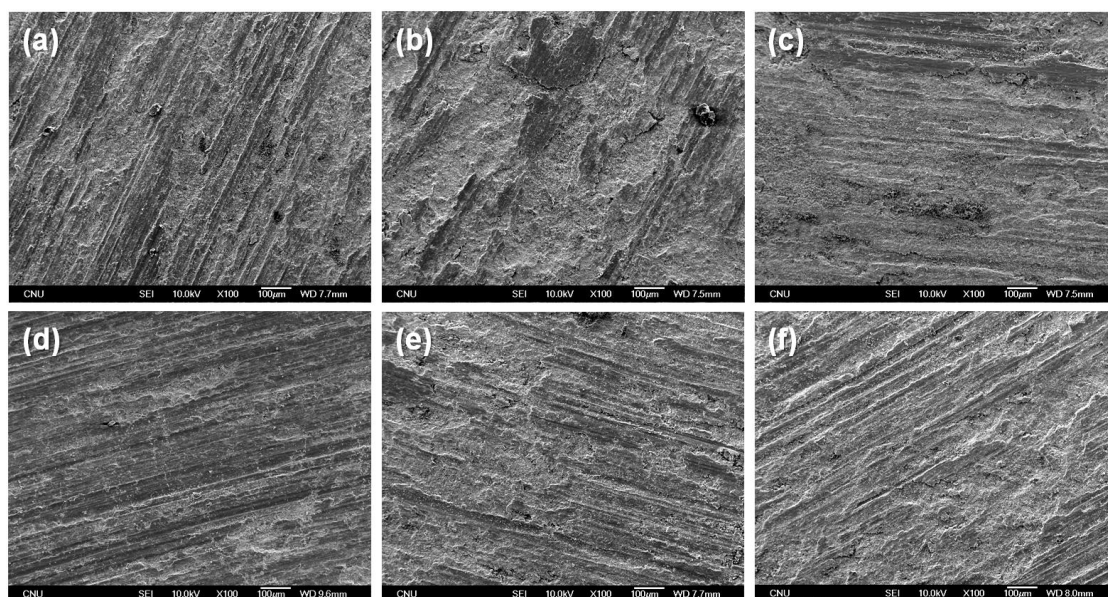


Figure 7. SEM micrographs of worn surfaces of the CNF/A7xxx composites: (a) 0.43 wt% CNF, 5N; (b) 0.43 wt% CNF, 10N; (c) 0.43 wt% CNF, 20N; (d) 0.76 wt% CNF, 5N; (e) 0.76 wt% CNF, 10N; and (f) 0.76 wt% CNF, 20N.

The worn surfaces of the 0.43 wt% and 0.76 wt% CNF/A7xxx composites at load of 5 N, 10 N, and 20 N, respectively, are shown in Fig. 7. When the specimen with lower CNF concentration (0.43 wt%) slides at lower load condition (5 N), a little plastic deformation with grooves and flake formation were observed as shown in Fig. 7(a). For the composite with higher CNF concentration (0.76 wt%), the flake formation was greatly reduced due to its higher hardness (Fig. 7(d)) as indicated in Fig 4. As increasing the applied load from 5 N to 20 N, the flake formation increased in all weight fractions of CNF. The worn surfaces of the composites with lower weight fraction of CNF (0.43 wt%) revealed severe flake formation (Fig. 7(c)) at load of 20 N. However, the flake formation remarkably reduced in the composites with higher weight fraction of CNF (0.76 wt%), which is well coincided with the results shown in Fig. 5.

According to the result of Tu *et al.*[8], the plastic deformation with grooves is the principal wear mechanism for the composites with low weight fraction (up to 2.0 wt% CNT), while the cracking and flake formation were predominant for composites with higher fraction of CNT. However, this study indicates that the flake formation was the principal wear mechanism even for the CNF/A7xxx composites with lower weight fraction of CNF than 0.76 wt%. The reason why the principal wear mechanism for the CNF/A7xxx composites with low concentration of CNF is not the plastic deformation with grooves but the flake formation is not only the higher wear resistance of the A7xxx matrix than Cu but also the uniform dispersion of CNFs in Al matrix.

4 Conclusions

- (1) CNF reinforced A7xxx composites were successfully fabricated using a liquid process, which was mainly due to the dispersion enhancement of CNFs in Al matrix by vibration milling and Cu coating on CNFs. A considerable increase in hardness value by addition of CNFs in A7xxx matrix was observed.
- (2) The wear loss of the composites showed a steadily decreasing trend with weight fraction of CNF in the composites, and also increased with applied load. The favourable effects of CNF on wear resistance of the CNF/A7xxx composites are attributed to its excellent mechanical properties and uniform dispersion of CNF in Al matrix. At the same time, the CNFs exposed on the worn surface during wear test could act as a lubricating agent between the Al matrix and the counter part steel disk. The effects were more remarkable under high applied load than low applied load.
- (3) The flake formation was the principal wear mechanism for the CNF/A7xxx composites with lower weight fraction of CNF than 0.76 wt%.

Acknowledgements

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