Carbon nanotube reinforced aluminium composites fabricated by powder metallurgical and hot-rolling techniques

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Abstract. This research aims to fabricate light weight and high strength materials such as multi-walled carbon nanotubes (CNT) reinforced aluminium composites at relatively low cost. CNT/Al composites have been fabricated in three steps: (1) preparation of CNT dispersion, (2) preparation of CNT/Al based composite material by powder metallurgical slurry method, and (3) strengthening and refining of CNT/Al based composites by rolling processes. After fabrication, the composites have been characterized on dispersibility of CNT, Vickers hardness, three-point bending and tensile properties.

1. Introduction

In recent years, in order to cope with environmental problem issues such as CO₂ emission reduction, replacing various conventional structural materials like heavy metals consisting major parts in automobiles such as iron materials with light metals such as aluminum (Al), magnesium (Mg) and titanium (Ti) have been desirable. However, the weight reduction of structural materials by light metals often leads to decrease in strength, which causes problems threatening securing safety and sustainability. Attempts to develop carbon nanotube (CNT)/Al-based composites with enhanced strength have been highly attractive. However, at present there are very few practical examples due to difficulty to obtain uniform dispersion and wetting of CNTs with the matrix [1,2].

Fillers usually need some surface treatment to improve dispersion and wetting, but it is difficult to uniformly treat nanomaterials such as CNTs. Further, it has been reported that there is a possibility that the properties of the CNT may be impaired by the surface treatments. It was proposed that in order not to reduce the properties of CNT, dimethylacetamide was used as a solvent, and potassium carbonate which is an inorganic salt as a dispersing agent. Dispersion of CNT in the solvent was performed by sonication [3].

In this study, CNTs with high modulus and strength have been uniformly dispersed in Al matrix phase, and high-performance light-weight CNT / Al-based composites have been fabricated by combining powder metallurgy and hot rolling techniques. In addition, the relation between microstructures and mechanical properties have been investigated based on examination of electron microscope images, micro Vickers hardness, three-point bending and tensile test results.
2. Experiments

2.1 Fabrication

Al powder with a particle size of 30 (μm) and multi-walled carbon nanotubes (MWNT) with a diameter of 10-15 (nm) and an aspect ratio of 1000 were mixed. Potassium carbonate was used as dispersant, and dimethylacetamide was used as solvent. The CNT powder and potassium carbonate were put in dimethyl acetamide and sonicated with an ultrasonic equipment. The mixture was filtered using filter paper, and the CNT powder was taken out. A mixed powder was prepared from the CNT and Al powders, poured into a mold and dried using a heat gun. After that, pressure molding was performed at 150 kPa using a press machine. Then, it was sintered at 500 °C for 2 hours in an electric furnace.

2.2 Hot rolling

The test pieces prepared were heated in an electric furnace at 400 °C for 15 minutes, and repeated hot rolling was performed with a reduction ratio of 3 % for a rolling process at the beginning. After ten time rolling, the reduction ratio was changed to 4 to 5 % for a rolling process. Then, the rolling was repeatedly conducted to total reduction ratio of 30 %. Fig.1 shows the repeated processes of hot rolling.

![Figure 1](image)

Figure 1. Repeated hot rolling processes

2.3 Characterization

Evaluation of dispersibility of CNT in the composites, Vickers hardness, three-point bending and tensile tests were carried out. Cross-sectional observations in CNT/Al composites was performed using a scanning electron microscope (SEM) and energy dispersive X-ray spectrometry (EDX).

For Vickers hardness test, the cross sections of the test pieces were polished, and the Vickers hardness test (on five points) was performed. For three-point bending tests, the test pieces were prepared by cutting out edge sides from as-rolled samples. Displacement rate was set at 1.0 mm/ min in the bending tests. The support span is 25mm. For tensile tests, Instron-type tensile equipment was used, and the strain rate was set at 5.6x10⁻⁶/ sec. Elastic modulus, tensile strength and maximum tensile strain were estimated. Anisotropy was also examined.
3 Results and Discussion

The SEM backscattered electron image and EDX image of the cross section of CNT/ Al composites are shown in Fig.2.

![SEM image and EDX image of CNT/Al composites](image)

Figure 2. Cross-sectional Observation of CNT/Al Composites (Ultrasonic Treatment for 1 hour, Carbon Nanotube Content of 1.0 mass .%) (a) SEM image, (b) EDX image

CNTs are seen to be relatively uniformly dispersed. Some aggregation of CNT can be also observed.

![Sample apparatus before and after rolling](image)

Figure 3. Sample apparatus (a) before and (b) after rolling

Fig.4 shows the relationship between bending stress and strain (extracted from three-point bending test results) for sonication time of 0h, 3h, and 4h (hour). The content of CNT was 0.5 mass %.

![Stress-strain diagram for CNT/Al composites](image)

Figure 4. Stress-strain diagram for CNT (0.5 mass %) / Al composites from three-point bending tests
Fig. 5 shows the relationship between sonication time and flexural modulus. The CNT content was 0.5 mass %. The flexural modulus obtained from the bending tests are lower than the values in literature. It can be confirmed that the flexural modulus increases as sonication time increases to sonication time of 3h. In case of 4h, the flexural modulus sharply drops to the value which is the almost same as that for 0h. It can also be seen that ductility is significantly impaired for 3h. The reason for the low value of flexural modulus at 0h is due to the least dispersion of CNTs forming CNT aggregation and lumps. Some voids exist, which leads to decrease in flexural modulus.

**Figure 5.** Relationship between flexural modulus and sonication time.

Next, we move to the results of tensile test. Fig. 6 shows stress-strain curves for pure Al and CNT/ Al composites with sonication time of 0h, 1h, 2h, 3h and 4h. The CNT content was 0.5 mass%.

**Figure 6.** Stress-strain diagram for CNT (0.5 mass %)/ Al composites.

The Young’s modulus and tensile strength as a function of sonication time are shown in Fig.7 (a) and (b). The CNT content of the composites was 0.5 mass%.
Figure 7. Relationship between (a) tensile strength, (b) Young’s modulus and sonication time.

Elastic modulus of pure Al is seen around 90 GPa, and average values of elastic modulus of the composites are between 30 and 92 GPa. Maximum value is achieved for the composites with sonication time of 2h, and minimum is for the composites with sonication time of 4h. There can be highly scattering data in elastic modulus of the composites with sonication time of 2h. For tensile strength, with sonication time increasing, tensile strength increases. The composites with sonication time of 4h shows highest value of the tensile strength. It is also seen in Fig.7 (b) that the composites with sonication time of 4h achieved highest tensile strength and deformation ability.

Fig.8 shows Vickers hardness test results. The CNT content was 0.5 mass%.

Figure 8. Relationship between Vickers hardness and sonication time

The literature value of Vickers hardness of pure Al (1000 series) is around 30 (HV). In this study, the rolled Al shows more than 4 times of the hardness, because work hardening occurs as plastic deformation undergoes by rolling. It is also seen that the influence of sonication time is not so large, and CNT does not have large effect on Vickers hardness.
4 Conclusions

1. CNT/Al based composites were fabricated by a technique combining powder metallurgy and repeated hot rolling.
2. The tensile strength of the composites was enhanced by dispersing of CNTs in Al matrix, which was achieved with potassium carbonate in dimethyl acetamide by sonication in an ultrasonic bath.
3. It was found that the sonication time had a great influence on dispersing state of CNT.
4. Vickers hardness of the composites was not highly influenced by sonication time and CNT content.
5. Three-point bending test results demonstrated that flexural modulus increased with sonication time increasing up to 3h.
6. Tensile test results demonstrated that sonication time did not highly affect elastic modulus, while tensile strength increased as sonication time increased.

References

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