

분자수준 혼합공정을 이용한 탄소나노튜브/Cu 나노복합재료의 제조 및 특성평가

김경태*, 차승일*, 홍순형*

Fabrication and Characterization of Carbon Nanotube/Cu Nanocomposites by Molecular Level Mixing Process

Kyung Tae Kim*, Seung Il Cha* and Soon Hyung Hong*

Key Words: Carbon Nanotube, Cu, nanocomposites, molecular level mixing, characterization

Abstract

Since the first discovery of carbon nanotube (CNT) in 1991, a window to new technological areas has been opened. One of the emerging applications of CNTs is the reinforcement of composite materials to overcome the performance limits of conventional materials. However, because of the difficulties in distributing CNTs homogeneously in metal or ceramic matrix by means of traditional composite processes, it has been doubted whether CNTs can really reinforce metals or ceramics. In this study, CNT reinforced Cu matrix nanocomposite is fabricated by a novel fabrication process named molecular level mixing process. This process produces CNT/Cu composite powders whereby the CNTs are homogeneously implanted within Cu powders. The CNT/Cu nanocomposite, consolidated by spark plasma sintering of CNT/Cu composite powders, shows to be 3 times higher strength and 2 times higher Young's modulus than Cu matrix. This extra-ordinary strengthening effect of carbon nanotubes in metal is higher than that of any other reinforcement ever used for metal matrix composites.

1. Introduction

Several researchers have attempted to fabricate CNT reinforced metal or ceramic matrix composite materials by means of traditional powder metallurgy process,¹⁻³ which consists of mixing CNTs with matrix powders followed by sintering or hot pressing. However, these attempts have failed to fabricate CNT/metal or CNT/ceramic composites with homogeneously dispersed CNTs in the matrix. This is mainly due to strong agglomeration of CNTs in powder forms: the van der Waals forces between CNTs cause them to mutually attract each

other rather than homogeneously disperse. Furthermore, if CNT/metal or CNT/ceramic nanocomposites are manufactured by the conventional process, most of the CNTs are located on the surfaces of the metal or ceramic powders after mixing.¹⁻³ The conventional process inhibits the diffusion of matrix materials across or along the powder surfaces; hence, sintering cannot proceed without damaging the CNTs or removing them from the powder surfaces. Even if sintering is successful, CNTs are mostly located at grain boundaries of the matrix and are insignificant in improving material performance. At the same time, the most important processing issue is the interfacial strength between carbon nanotubes and the matrix. In the case of CNT/polymer nanocomposites, the interfacial

* 한국과학기술원 신소재공학과

strength between the CNTs and the polymer matrix is strong because they interact at molecular level.⁴ In the case of CNT/metal or CNT/ceramic nanocomposites, however, the interfacial strength cannot be expected to be strong because the CNTs and the matrix are merely blended.¹

2. Experimental procedures

The strategy for developing a novel fabrication process for CNT/Cu nanocomposite basically involves molecular level mixing of the reinforcement(CNT) and the matrix material in a solution instead of the conventional powder mixing. This new process produces CNT/Cu composite powders where the CNTs are mainly located within the Cu powders rather than on surfaces of them; the chemical bonding between the CNTs and the Cu ions provide homogeneous distribution of CNTs as well as high interfacial strength between CNT and Cu.

The suggested process for fabricating CNT/Cu composite powders consists of molecular level mixing of CNTs with metal ions. A salt containing Cu ion is dissolved in the CNT suspension. Additional sonication treatment assists the dispersion of Cu ions among the suspended CNTs and promotes the reaction between Cu ions and the functional groups on CNT surfaces. The third step is to dry the solution by heating in the air. During this process, the solvent and ligands are removed and the Cu ions on CNTs are oxidized to form powders (Fig 1(a)). The fourth and final step is the calcination and reduction processes to obtain chemically stable crystalline powders. The powders obtained in the third step are generally mixture of Cu oxide materials. These powders are changed into stable CNT/CuO by heating in air and then reduced into CNT/Cu composite powders by a reduction process under hydrogen atmosphere. Finally obtained CNT/Cu composite powder has the schematic shape, which CNT is implanted in Cu matrix, shown in Fig. 1(b).

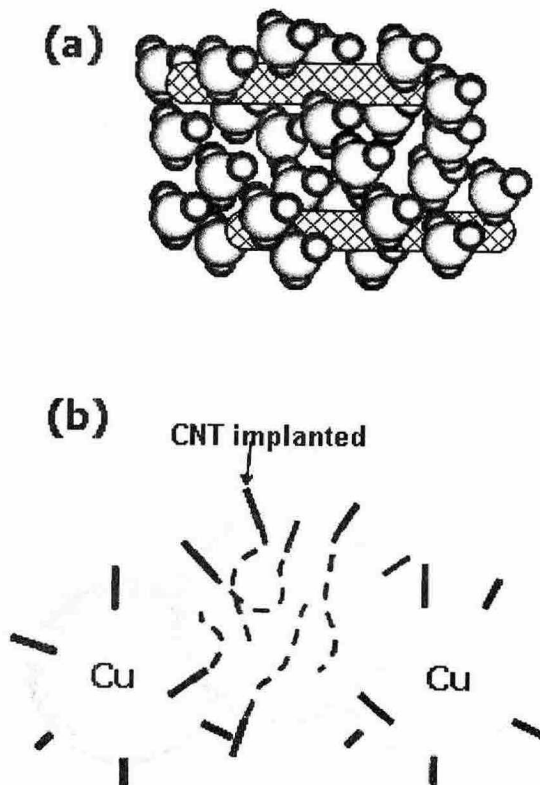


Fig. 1.(a) dissolution of Cu salt and attaching Cu ions to functional group on the surface of carbon nanotube, (b) vaporization of solvent by heating. (c) Schematic figure of CNT/Cu composite powder

3. Results

3. 1 Microstructure of CNT/Cu nanocomposites

The above 4-step process gives homogeneously dispersed CNTs within Cu powders as shown in Fig. 2. The most important feature of this process is that CNTs and Cu ions are mixed each other at molecular level. That is, the CNTs are located within the powders rather than on the powder surfaces. The morphologies of the CNT/CuO and CNT/Cu powders show an ideal composite microstructure, which displays spherical morphologies with CNTs implanted in the powders (Fig. 2(a), 2(b)).

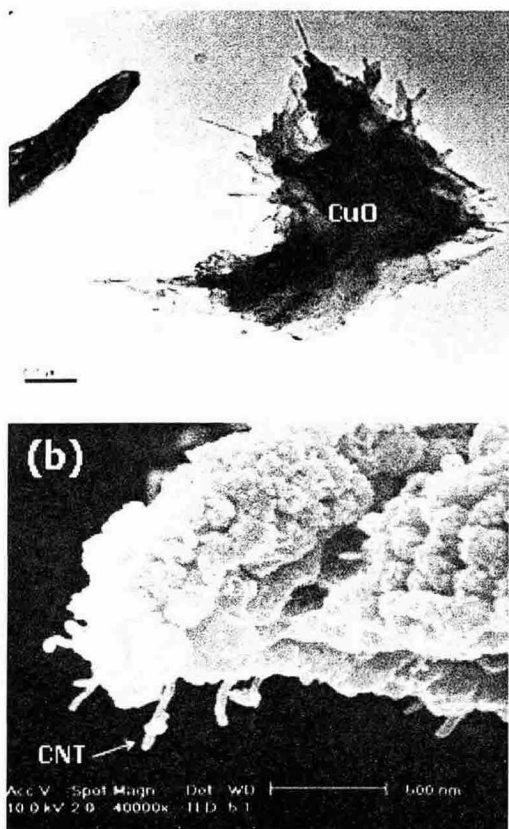


Fig. 2. (a) TEM micrographs of CNT/CuO composite powders, showing that CNTs are implanted within the matrix (b) SEM micrographs of CNT/Cu composite powders,

The CNT/Cu composite powders fabricated by the above-explained molecular level mixing process was consolidated into bulk CNT/Cu nanocomposite with full densification by spark plasma sintering, which can produce a high heating rate of 100°C/min and rapid consolidation through high joule heating and spark plasma generated between powders. The schematics of microstructure of CNT/Cu nanocomposite is shown in Fig. 3(a). The consolidated CNT/Cu nanocomposite shows homogeneous distribution of carbon nanotubes within the Cu matrix, which had not been obtained until now for CNT/metal or CNT/ceramic nanocomposites(Fig. 3(b)).

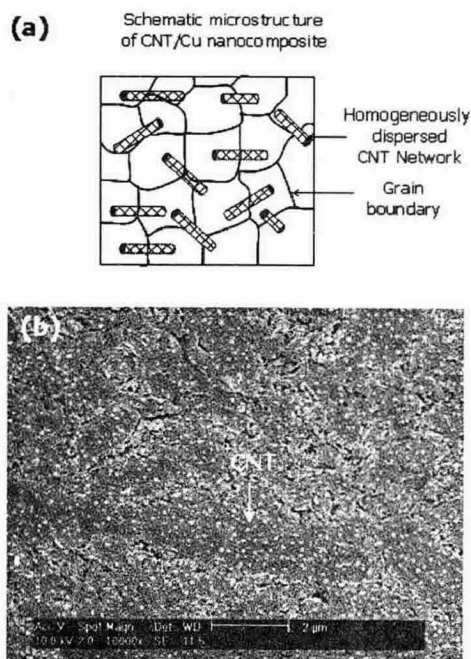


Fig. 3 (a) Schematics of microstructure of CNT/Cu nanocomposite. (b) SEM micrographs showing homogeneous distribution of carbon nanotubes, which are revealed after chemical etching of CNT/Cu nanocomposite with 5vol.% of carbon nanotubes.

3. 2 Mechanical properties of CNT/Cu nanocomposites

The mechanical properties of CNT/Cu nanocomposite were characterized using a compressive test. As shown in Fig. 4(a), the compressive yield strengths of CNT/Cu nanocomposites were much higher than that of Cu matrix, which is fabricated by the same process without adding CNTs. 5 volume percent CNT reinforced Cu matrix nanocomposite shows yield strength of 360MPa, which is 2.3 times higher than that of Cu. In the case of 10 volume percent CNT reinforced Cu, the yield strength is 485MPa, which is more than 3 times higher than that of Cu. Moreover, Young's modulus of CNT/Cu nanocomposite increases as the volume fraction of

carbon nanotubes is increased, as shown in Fig. 4(b).

Such remarkable strengthening by CNT reinforcement is due to the high load transfer efficiency of CNTs in metal matrix. High load transfer efficiency is caused by strong interfacial strength between CNT and Cu, which is originated from chemical bonding formed during molecular level mixing process.

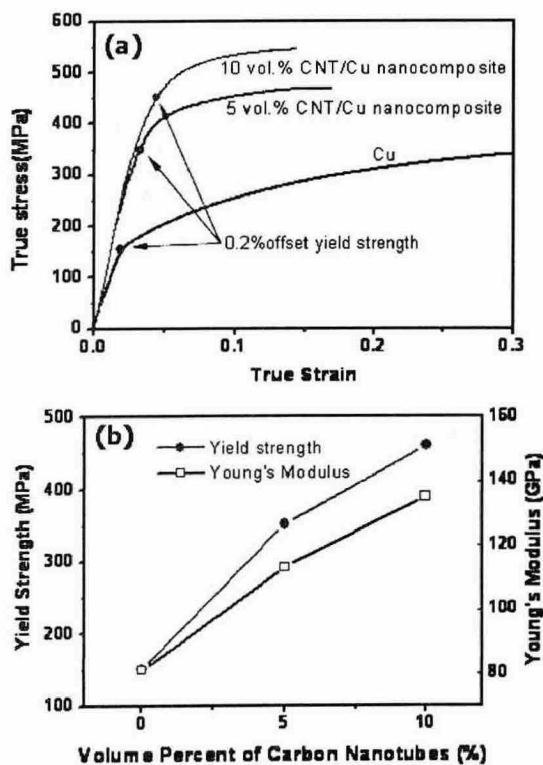


Fig. 4. Mechanical properties of CNT/Cu nanocomposites. (a) the stress-strain curves of CNT/Cu nanocomposites obtained by compressive test and (b) yield strength and Young's modulus of CNT/Cu nanocomposites according to the volume percentage of CNTs.

4. Conclusion

CNT/Cu nanocomposites with homogeneously dispersed CNTs within the Cu matrix can be

fabricated by means of the molecular level mixing process, which consists of mixing Cu ions with functionalized CNTs in solvent. The yield strength of CNT/Cu nanocomposite is shown to be 3 times higher than that of Cu. The carbon nanotube shows the most effective strengthening efficiency among all reinforcement materials: 8 times higher than that of SiC particle and 3 times higher than that of SiC whisker.

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