Nondestructive Sensing Evaluation of Ni Nanowire Strands and Carbon Nanotube/Epoxy Composites Using Electro-Micromechanical Techniques

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Electro-Micromechanical 시험법을 이용한 Ni Nanowire Strands 및 Carbon Nanotube 강화 에폭시 복합재료의 비파괴 감지능 평가

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KEY WORDS: nondestructive sensing evaluation, Ni nanowire strands, carbon nanotube, electro-micromechnical technique

ABSTRACT

Nondestructive damage sensing and load transferring mechanism of Ni nanowire strands and multi-wall carbon nanotube (MWCNT)/epoxy composites were investigated using electro-micromechanical techniques. MWCNT composite was especially prepared for high volume contents, 50 vol % of reinforcement. Electro-micromechanical techniques were applied to measure apparent modulus and contact resistance of Ni nanocomposites with their alignment and different diameters, and adding contents. Applied cyclic load affected on apparent modulus and electrical properties on nanocomposites due to various inherent properties of each CNMs. Contact resistivity on humidity sensing was a good indicator for monitoring as for multifunctional applications. Further study on actuation as well as sensing will be investigated for the following work continuously.

Nomenclature

$\Delta\rho$	: Change in electrical resistivity
ρ_v	: Electrical volume resistivity
ρ_c	: Electrical contact resistivity
L_{ec}	: Voltage contact length
A	: Cross sectional area
A_c	: Electrical contact area
R_c	: Electrical contact resistance

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1. INTRODUCTION

Recently, carbon nanomaterials (CNMs) such as single-wall carbon nanotube (SWCNT), multi-wall carbon nanotube (MWCNT), and carbon nanofiber (CNF) reinforced polymeric matrix composites have attracted with considerable attention in the research and industrial field due to their unique mechanical and electrical properties [1-2]. High aspect ratio, sub-micron and nanoscale Ni metal filaments are also recently produced commercially as 'nanostrand' [3]. Ni nanowire strands is easy to alignment for magnetic field. Ni nanowire strands composites good electrical conductivity. Carbon nanocomposites have high stiffness, strength and good electrical conductivity at relatively low concentrations of reinforcing CNM. Electrical and mechanical properties of CNM or Ni nanowire strands reinforced polymer composites depend on many factors seriously, such as inherent properties of CNM and Ni nanowire strands, the degree of dispersion, orientation, interfacial adhesion, aspect ratio, fiber shape and adding content, etc. Especially, the degree of dispersion is well known as one of the important factors to control many uniform properties. Experimentally-observed percolation threshold values strongly depend on the aspect ratio of the reinforcement. Generally CNT and Ni nanowire strands have high electrical conductivity and large aspect ratio. CNMs or aligned buckypapers could be used for achieving the desirable mechanical orientation in composite materials [4-6]. The electro-micromechanical technique [7-9] has been studied as an economical nondestructive evaluation (NDE) method for damage sensing, the characterization of interfacial properties, and nondestructive behavior because conductive fiber can act as a sensor in itself as well as a reinforcing fiber. In this work, contact resistance and sensing effect were evaluated for Ni nanowire and CNT reinforced epoxy as function of diameter, content and temperature by electrical resistance measurement. In addition, feasible humidity sensing was also evaluated for CNT composites.

2. EXPERIMENTAL

2. 1. Materials

Ni nanowire strands (Metal matrix Composites, inc.) and CNT (Iljin Nanotech Co., Korea) as reinforcing and sensing materials were used and their average diameters were 20 nm, respectively. Epoxy resin (YD-128 and YD-127, Kukdo Chemical Co., Korea) based on diglycidyl ether of bisphenol-A was used as a matrix. Flexibility of the epoxy matrix was controlled by changing the ratio of Jeffamine (polyoxypropylene diamine, Huntsman Petrochem. Co.) D400 *versus* D2000 in the curing mixture. As another curing agent, KH-100 (aromatic amine, Kukdo Chemical) was used for high modulus properties.

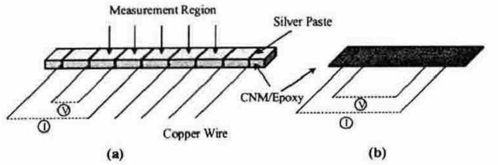


Fig. 1 Experimental scheme for the electrical volume resistivity measurement (a) Ni nanowire strands and (b) CNT composites

2. 2. Specimen preparation

2.2.1. Preparation of Testing Specimens

Detailed dispersion process was performed for uniform mechanical mixing. Ni nanowire strands were dispersed in epoxy. Ni nanowire strand were cured at pre-curing temperature, 80 °C for 2 hours and then post-curing at 120 °C for 2 hours. Compressed CNT sheet by roller were dispersed in acetone-based epoxy solution by mechanical mixing and then infiltrated into CNT. Acetone solvent was evaporated under low temperature in 35 °C for 2 hours and then cured at 150 °C for 4 hours.

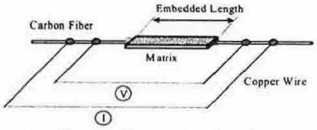


Fig. 2 Schematic figure for cyclic loading test

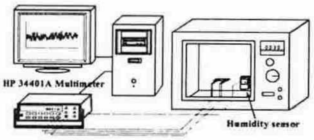


Fig. 3 Scheme of humidity test in oven

2. 3. Electrical resistivity measurement

Figure 2 shows the experimental scheme for the electrical volume resistivity measurement under uniform cyclic loading. Figure 3 shows the scheme for measuring the electrical volume resistivity on the humidity. Electrical resistance of Ni nanowire strands nanocomposites and CNT with volume fraction was measured by four-point probe method. Electrical contact points were located with regular distance using copper wire and silver paste as a glue. Electrical volumetric resistivity was obtained from the measured electrical volumetric resistance, cross-sectional area of the Ni nanowire strands nanocomposites, A_v, and electrical contact length, L_{ec} of the testing specimen connecting to copper wire. Testing speed and load cell were 0.5 mm/min. and 100 N, respectively. After a testing specimen was fixed into the UTM grip, the composite and the multi-meter were connected electrically using a very thin copper wire. While 5 cyclic loads were applied, the electrical resistance of the microcomposites was measured simultaneously with stress/strain changes. The relationship between electrical volume resistivity, ρ_v and resistance, R_v is as follow:

$$\rho_V = \left(\frac{A_V}{L_{ec}}\right) \times R_V \quad (\Omega \cdot cm) \tag{1}$$

The electrical contact resistivity, ρ_c is as follow:

$$\rho_C = A_C \times R_C \quad (\Omega \cdot cm^2) \tag{2}$$

where, A_c and R_c are electrical contact area and resistance, respectively.

3. RESULTS AND DISCUSSION

3. 1. Ni nanowire strands/epoxy composites

Ni nanowire strands have high aspect ratio and conductivity and inherently metal materials with high density as disadvantage, whereas Ni nanowire strands have high conductivity and can be easily aligned under magnetic field.

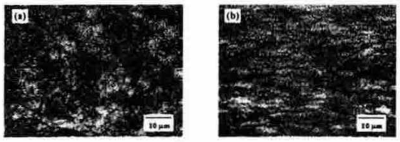


Fig. 4 Ni nanowire strands with (a) random state and (b) aligned state under magnetic field

Fig. 5 SEM image for Ni nanowire strands with (a) 100-300 nm, (b) 300-800 nm, (c) 1-3 μm.

Figure 4 shows Ni nanowire strands with random state and distinct aligned state under magnetic field using reflective optical microscope. Figure 5 shows SEM photos of three different aspect ratios of Ni nanowire strands with some of certain variation range statistically.

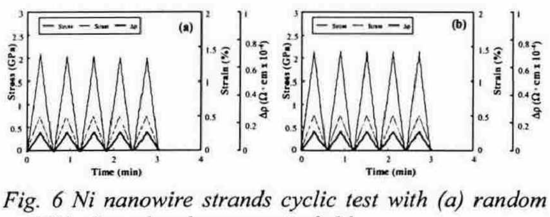


Fig. 6 Ni nanowire strands cyclic test with (a) random and (b) aligned under magnetic field

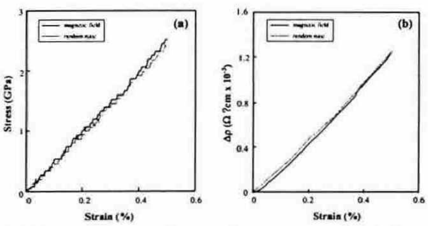
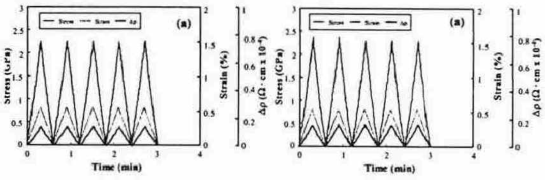


Fig. 7 Direct comparison of apparent modulus and ρ-strain curve of Ni nanowire strands/epoxy composite with random and aligned states

Figure 6 shows the cyclic test result of random and aligned states of Ni nanaowire strands. Electrical resistance was responded well for uniformed applied five cyclic loading. Reaching time and stress were similar to each other. Figure 7 shows the comparison of apparent modulus and ρ-strain curve of Ni nanowire strands/epoxy composite with random and aligned under magnetic field. Apparent modulus and electrical resistance of two cases were almost similar to each other within error range.



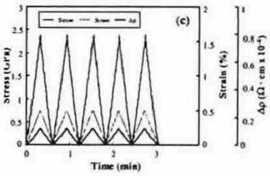


Fig. 8 Cyclic test with different diameters of Ni nanowire strands (a) 100-300 nm, (b) 300-800 nm, (c) 1-3 μm.

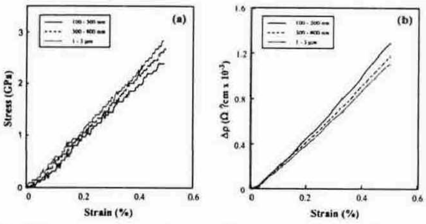
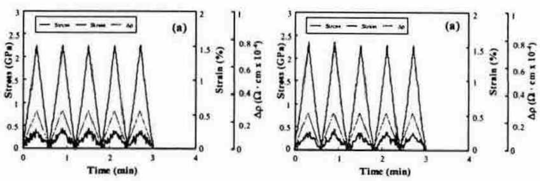


Fig. 8 Direct comparison of apparent modulus and ρ-strain curve of Ni nanowire strands/epoxy composite with different diameter (a) 100-300 nm, (b) 300-800 nm, and (c) 1-3 μm.

Figure 8 shows the direct comparison of apparent modulus and ρ-strain curve of Ni nanowire strands/epoxy composites with 3 different diameters. Thin diameter Ni strands showed higher apparent modulus and smaller electrical resistance. It can be due to more opportunity of electrical contact in case of same volume content with short aspect ratio.



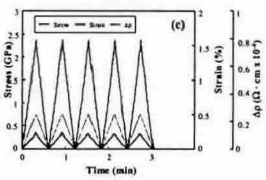


Fig. 9 Contact resistivity with different concentration of Ni nanowire strands (a) 0.3 vol%, (b) 0.5 vol%, (c) 2.0 vol%.

Figure 9 shows contact resistivity with three different concentrations of Ni nanowire strands. Electrical resistance responded well except the sensing degree.
Figure 10 shows the change in electrical contact resistivity for Ni nanowire strands. As added content increased, contact resistance decreased due to increased percolation concentration. Sensing response became also

improved with increasing volumetric concentration by reducing detecting noise.

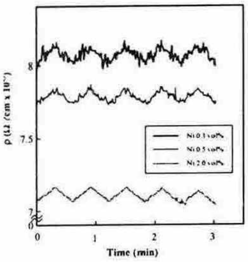


Fig. 10 The change in electrical contact resistivity for Ni nanowire strands.

3. 2. Carbon Nanotube/epoxy composites

As a high CNT volume composites, added CNT volume fraction was over 50wt%. Figure 11 shows the comparison of tensile properties for neat epoxy matrix and CNT. Tensile strength and modulus of CNT composites were much higher then neat epoxy case.

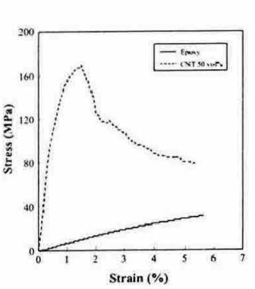


Fig. 11 Comparison of tensile properties for CNT composites and epoxy matrix.

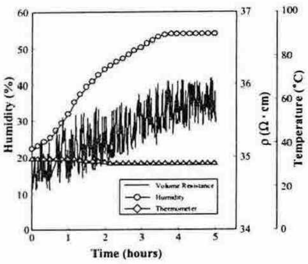


Fig. 12 The change in humidity for CNT of volume resistance.

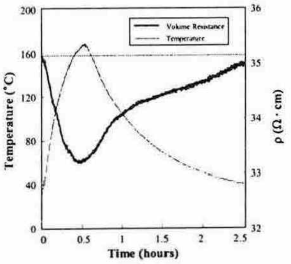


Fig. 13 The change in electrical contact resistivity for CNT composite of volume resistance in increasing temperature.

Figure 12 shows the change in humidity for electrical volume resistivity of CNT. When the humidity increases, also the resistance increases. Electrical resistance on humidify will be evaluated continuously further for humidify sensor for different CNMs.

Figure 13 shows the electrical resistance with changing temperature using CNF nanocomposite. Electrical resistance was responded reversely well with temperature due to inherent thermal properties of CNT and internal residual stress as described in our previous works [9].

4. CONCLUSIONS

Electro-micromechanical technique was applied to thermal treated CNMs composites to investigate sensing and stress transferring properties for Ni nanostrands and CNT/epoxy composites. Under magnetic field, Ni nanowire strands were rearranged well compared to random case, although their apparent modulus was similar to each other. Comparing different diameter effect of Ni nanowire composites, thin diameter of Ni nanowire strands showed larger apparent modulus value than thicker diameter case. With increasing adding content of Ni nanowire contents, sensing response became improved. Higher volume content of CNT composites were prepared and showed significantly higher tensile strength and modulus than neat epoxy case. CNT composite was evaluated for humidity sensing and showed generally proportion, whereas CNT composites showed disproportion on temperature. In this work one of important main point of CNT composites is to get such a high volumetric content, over 50 vol%, than conventional 3-5 vol% CNT/epoxy composites due to high viscosity. Some new information on temperature sensing and stress transfer effect of Ni nanostrands and carbon nanocomposites could be obtained from the electrical resistance measurement as a feasible new concept of the nondestructive evaluation.

ACKNOWLEDGMENT

This work was financially supported by Korea Research Foundation Grant (KRF-2004-002-D00192).

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