Sensing and Interfacial Evaluation of Ni Nanowire Strands/Polymer Composites using Electro-micromechanical Technique

Sung-Ju Kim*, Jin-Gyu Jung*, Joung-Man Park*,†

Electro-Micromechanical 시험법을 이용한 Ni Nanowire Strands 강화 고분자 복합재료의 Sensing과 계면 물성 평가

김성주*• 정진규*•박종만*,†

KEY WORDS: nondestructive sensing evaluation, Ni nanowire strands, electro-micromechnical technique, humidity sensing, temperature sensing

ABSTRACT

Sensing and interfacial evaluation of Ni nanowire strands/polymer composites were investigated using electro-micromechanical technique. Electro-micromechanical techniques can be used as sensing method for micro damage, loading, temperature of interfacial properties. Using Ni nanowire strands/silicone composites with different content, load sensing response of electrical contact resistivity was investigated under tensile and compression condition. The mechanical properties of Ni nanowire strands with different type/epoxy composites were measured using uniformed cyclic loading and tensile test. Ni nanowire strands/epoxy composites showed humidity and temperature sensing within limited ranges, 20 vol% reinforcement. Some new information on temperature and humidity sensing plus loading sensing of Ni nanowire strands/polymer composites could be obtained from the electrical resistance measurement as a new concept of the nondestructive interfacial evaluation.

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

1. INTRODUCTION

Recently, metal nanowire strands of nickel have been previously studied as conductive additives for polymer resins and fiber reinforced composites. Nanowirestrands are identified a sub-micron diameter filaments of pure metal, their length maybe from tens of microns to several millimeters. Typical aspect ratios are about 500:1 very high. Adding the Ni nanowire strands to the polymer is the process with the most widespread potential for many applications, due to the many industrially accepted practices of making polymers conductive by simply adding conductive filler. Ni nanowire strands have been successfully mixed in to epoxies, urethane, acrylics, elastomers and thermoplastics, rending all of them conductive [1]. Ni nanowire strands is easy to alignment for magnetic field. Ni nanowire strands composites good electrical conductivity. Ni nanocomposites have high stiffness, strength and good electrical conductivity at relatively low concentrations of reinforcing Ni nanowire strands. Electrical and mechanical properties of Ni

---

*School of Materials Science and Engineering/
Polymer Science and Engineering,
Engineering Research Institute,
Gyeongsang National University

nanowire strands reinforced polymer composites depend on many factors such as inherent properties of Ni nanowire strands, the degree of dispersion, orientation, interfacial adhesion, aspect ratio, fiber shape and content, etc. Especially, the degree of dispersion is well known as one of the most important factors in electrical properties [2]. Experimentally-observed percolation threshold values strongly depend on the aspect ratio of the reinforcement. Generally Ni nanowire strands have high electrical conductivity and large aspect ratio. Ni nanowire strands could be used for improving the mechanical properties and increasing fracture energy in composite materials [3,4]. The electro-micromechanical technique 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. Fiber damage in electrical insulator such as polymer matrix could not be detected after the first fiber fracture occurred, whereas in electrical conductive matrix fiber fracture as well as matrix deformation could be detected continuously by electrical resistance measurement [5,6]. In this work, mechanical, interfacial properties and sensing effect were evaluated for Ni nanowire strands/polymer composites using electrical resistance measurement.

2. EXPERIMENTAL

2. 1. Materials

Ni nanowire strands (Metal Matrix Composites, inc., Midway, Utah) as reinforcing and sensing materials were used. This reinforcement is three different diameter ranges, i.e., type A: 100-300nm, type B: 300-800nm, type C: 1000-3000nm. Type B was used as main sensor in this work. Conventional carbon fiber (Taekwang Co., TZ307, Korea) with average diameter 8 μm was used as a reinforcement and epoxy resin (YD-128, 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 matrix silicone (KE-1300, SinEtsu chemical Co.,Ltd, Japan) was used.

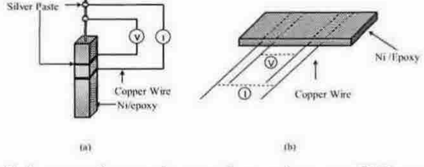

Fig. 1 Scheme of experimental specimens of Ni nanowire strands for (a) loading sensor by the electrical contact resistivity measurement; and (b) humidity and temperature sensors

2. 2. Specimen preparation

2.2.1. Preparation of Testing Specimens

Ni nanowire strands were dispersed in epoxy and silicone. Dispersion process was processed gently mixed using mechanical mixing. Ni nanowire strand /epoxy composites were curing pre-curing temperature in 80 °C for 2 hours and then post-curing temperature in 120 °C for 2 hours. Ni nanowire strands/Silicone composites were curing temperature in 60 °C for 1 hours. Figure 1 (b) shows shape of specimen for humidity and temperature sensing. Concentration of Ni nanowire strands/epoxy composites is 20 vol%. For the Ni nanowire strands/silicone composites case, Size of loading sensing specimens was 10mm in width, 10mm in length, 2mm in thickness, respectively.

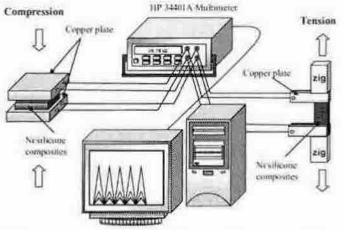

Fig. 2 Scheme of experimental sensing test under compression and tensile loading

2. 3. Electrical resistivity measurement

Figure 1 (a) shows the experimental scheme for the electrical volume resistivity measurement. And Figure 2 shows the scheme for sensing of loading under compression and tensile test. Electrical resistance of Ni nanowire strands nanocomposites was measured by four-point probe method. Electrical contact points were located with regular distance using copper wire and silver paste. Electrical volume resistivity was obtained from the measured electrical volume resistance, cross-sectional area of the Ni nanowire strands composites, 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. Electrical resistivity was obtained from the measured electrical resistance, cross-sectional area of the conductive fiber, A, and electrical contact length, L_{ec} of the testing fiber connecting to copper wire. The relationship between electrical volume resistivity, ρ_v and resistance, R_v is as follow:

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

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/silicone composites

Mixing process is very important to get uniform properties and to prevent mechanical damage of brittle crystalline Ni nanowire strands. Figure 4 show FE-SEM photographs for fractured surface of 20 vol% Ni nanowire strands/silicone composites. Despite high content dispersion is observed very well in the silicone.

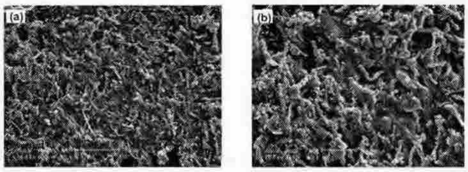

Fig. 4 FE-SEM photographs for (a) well-dispersion; and (b) fractured surfaces of Ni content 20vol%/silicone composites

For the Ni nanowire strands/silicone composites case, Silicone matrix is possible for loading sensing under tension and compression. Figure 5 and Figure 6 shows sensing on applied loading.

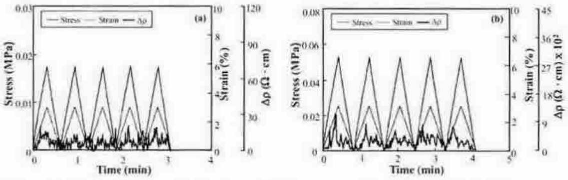

Fig. 5 Contact resistivity of Ni nanowire strands /silicone composites under tension; Ni content of (a) 10 vol% and (b) 20 vol%

Figure 5 shows contact resistivity of Ni nanowire strands/silicone composites under tension. Sensing response of resistivity is change with change content of Ni nanowire strands.

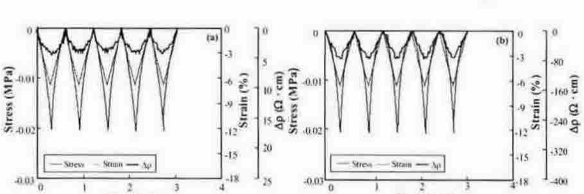

Fig. 6 Contact resistivity of Ni nanowire strands/silicone composites under compression; Ni content of (a) 10 vol% and (b)20 vol%

Figure 6 shows contact resistivity of Ni nanowire strands/silicone composites under compression. For the tensile case, as the stress and strain increased resistivity increased. On the other hand for the compression case, as the stress and strain increased the resistance decreased.

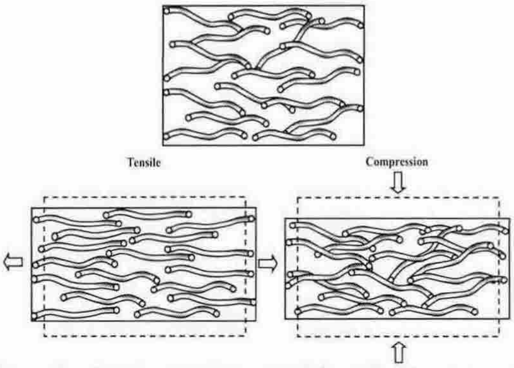

Fig. 7 Load sensing model of Ni nanowire strands/silicone composites under tension and compression

Figure 7 shows loading sensing model Ni nanowire strands/silicone composites under tension and compression.

Since under compressive load, contact probability among dispersed nanowire strands in silicone matrix increases, conductivity increased and thus resistivity decreased.

On the other hand, since under tensile loading, the orientation can occur to a certain degree and thus electrical contact probability can reduce, the conductivity decreases and thus the resistivity increases.

3. 2. Ni nanowire strands/epoxy composites

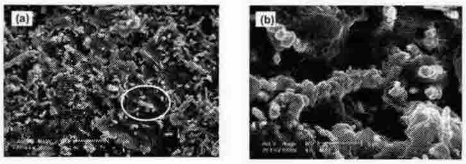

Fig. 8 FE-SEM photographs for (a) well dispersion and (b) fractured surfaces of Ni content of 20 vol%/epoxy composites

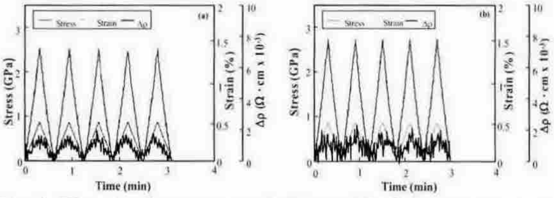

Fig. 9 Electrical contact resistivity with different type Ni nanowire strands/epoxy composites (a) A type: 100-300nm, (b) B type: 300-800nm

Figure 8 shows FE-SEM photographs for the fractured surface of 20 vol% Ni nanowire strands/epoxy composites. Good dispersion condition was observed despite poor bonding between Ni nanowire strands and epoxy matrix. Pull-out pattern of Ni nanowire strands crystalline was observed.

Figure 9 shows Electrical contact resistivity with different type Ni nanowire strands/epoxy composites. For the B type case, maximum stress was larger than Ni A type, although Ni A type loading sensing response of electrical contact resistivity was better than Ni B type.

Figure 10 shows the direct comparison of apparent modulus and mechanical properties curve of Ni nanowire strands/epoxy composites with 3 different diameters.

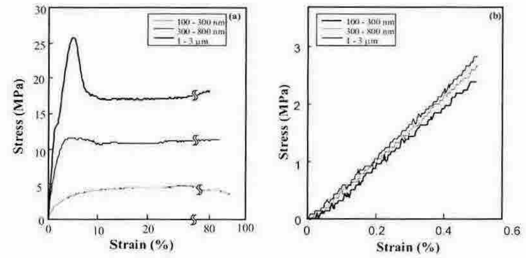

Fig.10 Comparison of (a) tensile properties and (b) apparent modulus of Ni nanowire strands/epoxy composites for 3 different types

The thickest type among 3 different types shows higher apparent modulus and tensile mechanical properties. It can be because other two thinner types were broken into smaller crystalline sizes due to their brittleness during mixing process, whereas the thickest type can keep the crystalline shape without breakage, which can result in stress transferring mechanisms and keep reinforcing effect.

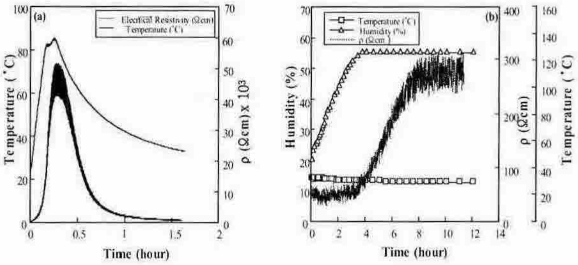

Fig. 11 Ni nanowire strands/epoxy composites showing (a) the change of electrical resistivity with changing temperature (b) the change in humidity for volume resistance; 20 vol% content.

Figure 11 shows humidity and temperature sensing of Ni nanowire strands/epoxy composites. In figure 10 (a), shows the change of electrical resistivity with changing temperature using Ni nanowire strands/epoxy composites. With increasing the temperature, electrical resistivity also increased and *vice versa.* And error range increased with increasing temperature. It is because mean free path due to thermal perturbation decreased and conductivity increases and thus resistivity decreased. In figure (b), the change in the humidity for the electrical volume resistivity of Ni nanowire strands/epoxy composites under constant temperature, 30 ℃. As the humidity increased, the resistivity increased. As humidity became saturated, the resistivity was responded to be consistent as expected.

4. CONCLUSIONS

Electro-micromechanical techniques were to obtain sensing response of Ni nanowire strands polymer composites by measuring electrical properties and interfacial evaluation. For the Ni nanowire strands/silicone composites with different content case, loading sensing response of electrical contact resistivity was monitored under tension and compression condition. Electrical contact resistivity of Ni nanowire strands with two different diameter /epoxy composites was evaluated using electro pull-out test. Maximum stress of large diameter Ni nanowire strands/epoxy composites was larger than thin diameter Ni nanowire strands/epoxy composites. Apparent modulus of three different type of Ni nanowire strands/epoxy composites was consistent with mechanical properties each other. Ni nanowire strands/ epoxy composites responded humidity sensing with increasing humidity under constant temperature. Temperature sensing also were responded well, their sensing error range increased with increasing temperature. Some new information on temperature and humidity sensing plus loading sensing of Ni nanowire strands/polymer could be investigated from the electrical resistivity and indirect interfacial properties 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).

REFERENCES

(1) G. Hansen, *SAMPE journal*, 42(2), 2005, pp. 24-33.

(2) O. Lourie, H.D., Wagner, *Compos Sci Technol.*, 1999, Vol. 59, pp.975-987.

(3) J. Fraysse, A. I. Minett, O. jaschinski, G. S. Duesberg, S. Roth, *Carbon*, 40, 2002, pp. 1735-1739.

(4) S. Roth, R. H. Baughman, *Current App. Physics*, 2, 2002, pp. 311-314.

(5) S. Wang, S. I. Lee, D. D. L. Chung and J. M. Park, *Compos. Interf.*, 8(6), 2001, pp. 435-441.

(6) J. M. Park, S. I. Lee, J. H. Choi, *Compos Sci Technol.*, 2005, Vol.65, No.2, pp571-580.