論文

Micromechanical 시험법과 전기저항 측정을 이용한 탄소섬유 강화 Epoxy-AT-PEI 복합재료의 비파괴적 손상 감지능 및 계면물성 평가

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Interfacial Evaluation and Nondestructive Damage Sensing of Carbon Fiber Reinforced Epoxy-AT-PEI Composites using Micromechanical Test and Electrical Resistance Measurement

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ABSTRACT

Interfacial properties and damage sensing for the carbon fiber/epoxy-amine terminated (AT)-polyetherimide (PEI) composite were performed using microdroplet test and electrical resistance measurements. As AT-PEI content increased, the fracture toughness of epoxy-AT-PEI matrix increased, and interfacial shear strength (IFSS) increased due to the improved fracture toughness by energy absorption mechanisms of AT-PEI phase. The microdroplet in the carbon fiber/neat epoxy composite showed brittle microfailure mode. At 15 phr AT-PEI content, ductile microfailure mode appeared because of improved fracture toughness. After curing, the change in electrical resistance (ΔR) with increasing AT-PEI content increased gradually because of thermal shrinkage. Under cyclic stress, in the neat epoxy case the reaching time until same stress was faster and their slope was higher than those of 15 phr AT-PEI. The result obtained from electrical resistance measurements under curing process and reversible stress/strain was correspondence well with matrix toughness properties.

초 록

Microdroplet 시험법과 전기저항 측정을 이용하여 탄소섬유강화 epoxy-AT-PEI 복합재료의 손상 감지능 및 계면물성평가에 대한 연구를 수행하였다. AT-PEI 함량이 증가함에 따라 기지재료의 파괴인성은 증가하였으며, 이로 인한 에너지 흡수 메커니즘에 의해서 계면전단강도 역시 증가하였다. Microdroplet 시험에서 순수 에폭시는 취성파괴 현상을 그리고 15 phr AT-PEI의 경우에는 파괴인성의 증가로 인해 연성 파단 현상을 관찰할 수 있었다. 경화 후에 열 수축에 의한 전기저항 변화는 AT-PEI 함량 증가에 따라 증가하였으며, 가변하중 하에서 순수 에폭시에 함침된 탄소섬유의 같은 응력까지의 도달시간과 기울기는 15 phr AT-PEI의 경우보다 더 빠르고 높았다. 경화과정과 가역적인 하중 하에서의 전기저항 측정으로부터 얻은 결과는 기지재료의 파괴인성과 잘 일치하였다.

Key Words: 손상감지능(Damage sensing), 전기저항도(Electrical resistivity), 계면전단강도(Interfacial shear strength (IFSS)), 파괴인성(Fracture toughness), 잔류응력(Residual stress)

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1. Introduction

Toughened epoxy matrix using liquid reactive rubber has been reported widely[1,2]. However, the improved toughness in most of rubber modified thermosetting systems results in a significant decrease in the glass transition temperature, Tg, stiffness and strength of the cured thermosetting resin. High performance thermoplastics, such as poly(ethersulfone) (PES), plyetherimide (PEI), polycarbonate and poly(phenyleneoxide) (PPO), or a combination of rubber and thermoplastics, are commonly added to thermosetting resins as processing modifiers. They increased the fracture toughness without reducing thermal and mechanical properties[3-5].

The electro-micromechanical technique had been studied as an economical and new nondestructive evaluation (NDE) method for curing monitoring, stress or strain sensing, characterization of interfacial properties, and nondestructive behavior because conductive fiber can act as a sensor in itself as well as a reinforcing fiber[6,7]. Residual stress in fiber-reinforced composite occurred during curing process due to the thermal contraction of the matrix or the difference in thermal expansion coefficient (TEC) between fiber and matrix. The effect of residual stress is reflected in the mechanical performance of cured composite[8].

In this work, interfacial properties, microfailure modes and cure monitoring of carbon fiber reinforced amine terminated (AT)-PEI toughened epoxy matrix composite were investigated using micromechanical test and electrical resistance measurement. The changes of electrical resistance under cyclic stress/strain and during curing process were correlated with matrix toughness.

2. Experimental

2.1 Materials

Two kinds of carbon fibers as reinforcing materials were used, and their average diameters were about 18 ㎛ (Mitsubishi, Chemical Co., Japan) and 8 ㎛ (Taekwang Co., TZ-307, Korea), respectively. A difunctional epoxy resin (YD-128, Kukdo Chemical Co., Korea), diglycidylether of bisphenol-A (DGEBA) was used as a main matrix resin and nadic methyl anhydride (NMA, Kukdo Chemical Co., Korea) was used as a curing agent. Synthesized AT-PEI using a commercial grade of PEI (Ultem 1000, General Electric Co.) was used as a thermoplastic modifier.

2.2 Methodologies

2.2.1 Preparation of Specimen

The fracture toughness of the cured epoxy-AT-PEI matrix was measured by three point bending test based on ASTM E 399[9] using universal testing machine (UTM, Lloyd Instrument Co., U.K.). The specimens for fracture toughness test were precured for each 2 hours at 80℃ and 120℃ in a vacuum oven. After eliminating vacuum, it was postcured finally for 12 hours at 140℃. The crosshead speed was 1.3 mm/minute and the span length was 40 mm. The fracture toughness, K_{IC} was calculated using the following equation:

$$K_{IC} = \left(\frac{FS}{BW^{3/2}}\right)\cdot f\left(\frac{a}{W}\right) \tag{1}$$

where F is the load, S is the span length, B and W are specimen thickness and width. And a is the crack depth and $f(a/W)$ is a geometrical factor of the specimen.

2.2.2 IFSS Measurement

The carbon fiber with 18 ㎛ diameter was fixed with regularly separated distance in a steel frame. Microdroplets of neat epoxy and epoxy-AT-PEI matrix were formed on each fiber axis using carbon fiber of 8 ㎛ in diameter. Microdroplet specimens were cured with above same curing steps.

In microdroplet test, the shear force at the interface was developed by applying the load. A microdroplet specimen was fixed by the microvise using a specially designed micrometer. The IFSS, τ was calculated from the measured pullout force, F using the following equation,

$$\tau = \frac{F}{\pi D_f L} \tag{2}$$

where D_f and L are fiber diameter and fiber embedded length in the matrix resin, respectively.

2.2.3 Electrical Resistance Measurement

Fig. 1, 2 show the scheme for the electrical resistance measurement of carbon fiber/epoxy-AT-PEI composite during curing process and under cyclic loading, respectively. While the curing was in progress and cyclic load was applied, the electrical resistance was measured using a digital multimeter (HP34401A). For the electrical resistance measurement under cyclic load, strain and stress were measured by mini-UTM (Hounsfield Test Equipment Ltd., U.K.). Testing speed and load cell were 0.5 mm/minute and 100 N, respectively. The calculation method of electrical resistivity, r is as follows:

$$\rho = \frac{L_{ec}}{A} \cdot R \tag{3}$$

where R is the electrical resistance, A is the cross-section area of conductive fiber, and L_{ec} is the electrical contact length between voltage contacts.

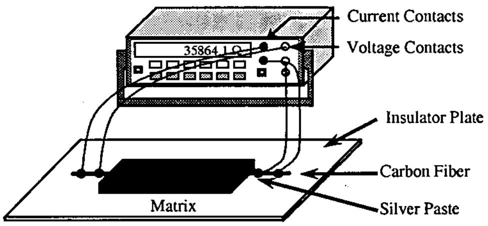

Fig. 1 Schematic diagram for electrical resistance measurement during curing process.

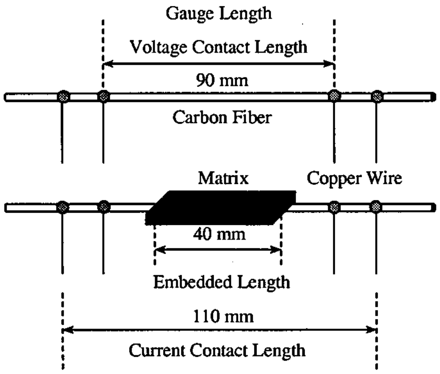

Fig. 2 Schematic diagram for electrical resistance measurement under cyclic loading.

3. Results and discussion

3.1 Mechanical Properties of Matrix

Mechanical properties of epoxy-AT-PEI matrix with AT-PEI content were measured by tensile and three-point short beam tests. Fig. 3 shows scanning electron microscopy (SEM) photographs for epoxy-AT-PEI matrix with AT-PEI content for (a) 5 phr AT-PEI, (b) 10 phr AT-PEI and (b) 15 phr AT-PEI. Epoxy-AT-PEI matrix appeared many AT-PEI phases in the epoxy matrix. In our previous work[5], pure PEI particles without amine group were microspherical shape and interface was clear, whereas AT-PEI particles were irregular microspherical shape and interface between AT-PEI particle and epoxy matrix was not observed clearly. The results might be because the difference in surface energy between AT-PEI and epoxy was lower than that of pure PEI case. AT-PEI was more hydrophilic compared to pure PEI due to the existing amine group. AT-PEI microspheres were spread uniformly in epoxy resin, and both relative contents and size of AT-PEI microsphere increased with increasing initial blending AT-PEI content. Several parameters, i.e. the content, size and shape of AT-PEI particle might affect on mechanical properties of epoxy-AT-PEI matrix, such as toughness, tensile strength and modulus.

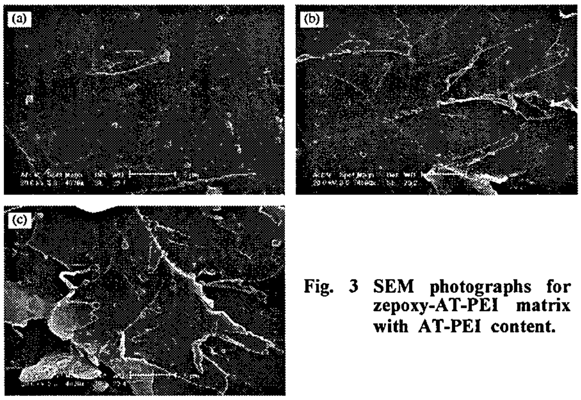

Fig. 3 SEM photographs for zepoxy-AT-PEI matrix with AT-PEI content.

Mechanical properties of AT-PEI modified epoxy matrix were compared to the matrix toughness. Fig. 4 shows the mechanical properties of epoxy-AT-PEI matrix and their stress-strain curves. Tensile strength and elongation were improved with increasing AT-PEI content by the general rule of mixture, whereas Young's modulus decreased. Fig. 5 shows the matrix fracture toughness of epoxy-AT-PEI matrix. The fracture toughness was improved gradually with increasing AT-PEI content. The results were consistent with stress-strain curves in Fig. 4. Microcracks could be propagated easily through brittle matrix, whereas ductile matrix could blunt crack propagation. At 15 phr AT-PEI, the fractured surface appeared tougher than the case of 5 phr AT-PEI content showing more likely smooth surface. The morphological change of fracture surface indicated the improved fracture toughness by adding AT-PEI.

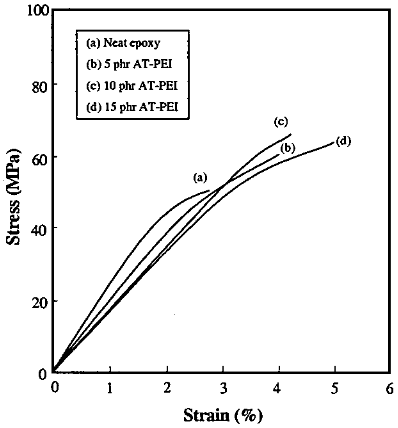

Fig. 4 Stress-stain curves of epoxy-AT-PEI matrix with AT-PEI content.

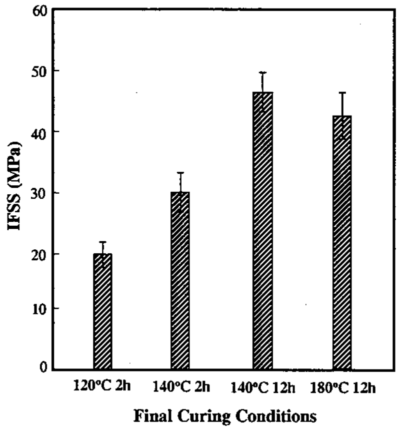

Fig. 6 IFSS of carbon fiber/epoxy-AT-PEI composites with curing conditions.

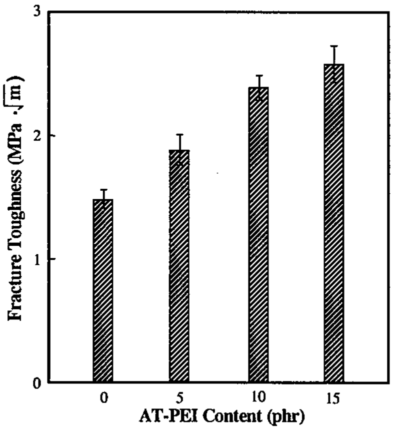

Fig. 5 Fracture toughness of epoxy-AT-PEI matrix with AT-PEI content.

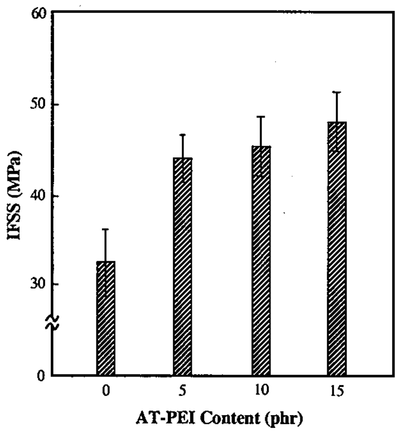

Fig. 7 IFSS of carbon fiber/epoxy-AT-PEI composites with AT-PEI content.

3.2 IFSS and Microfailure Modes

Interfacial properties of carbon fiber reinforced AT-PEI modified epoxy matrix with curing condition and AT-PEI content were measured by microdroplet test, and then they were correlated with microfailure modes. Fig. 6 shows IFSS of carbon fiber reinforced epoxy-AT-PEI composite with final postcuring conditions. IFSS between carbon fiber and epoxy-AT-PEI matrix cured at too low or high temperature might be small because of the low cross-linking density and thermal damage in the interface. Optimum cure condition obtained from IFSS measurement was that carbon fiber/epoxy-AT-PEI composite was precured for each 2 hours at 80℃ and 120℃, and then it was postcured finally for 12 hours at 140℃. Fig. 7 shows IFSS of carbon fiber reinforced epoxy-AT-PEI composite with AT-PEI content. IFSS increased with adding AT-PEI content due to the enhanced fracture toughness and energy absorption mechanisms. In this fiber and matrix system, matrix fracture toughness might be directly proportional to IFSS.

Fig. 8 shows SEM photographs of typical microfailure modes for the carbon fiber reinforced epoxy-AT-PEI composite with AT-PEI content after the microdroplet test for (a) neat epoxy and (b) 15 phr AT-PEI. Neat epoxy microdroplet appeared brittle microfailure mode, whereas high content of AT-PEI microdroplet exhibited more likely plastic deformation and ductile microfailure mode. Failure mode of fracture surface was generally changed from smooth to rough and the brittle nature became tougher with adding AT-PEI content.

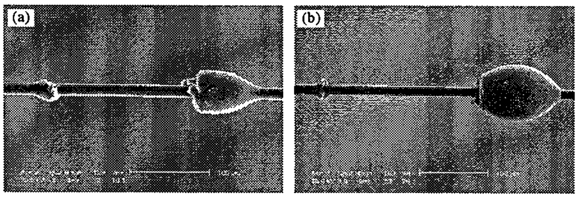

Fig. 8 Typical microfailure modes of carbon fiber/epoxy-AT-PEI composite for (a) neat epoxy and (b) 15 phr AT-PEI.

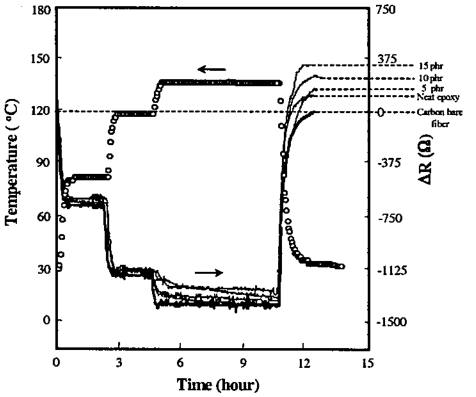

Fig. 9 The changes in electrical resistance for carbon fiber/epoxy -AT- PEI composite.

3.3 Cure Monitoring and Damage Sensing

Fig. 9 shows the change in electrical resistance (ΔR) for carbon fiber/epoxy-AT-PEI composite with AT-PEI content during curing process. Electrical resistance for carbon bare fiber without matrix was changed little compared to initial value. As AT-PEI content increased, ΔR after curing process increased gradually. ΔR after curing may be affected by matrix thermal shrinkage or residual stress determined by matrix toughness. External stress induced by cure and thermal shrinkage applied to carbon fiber through matrix. The higher fracture toughness, the higher thermal and cure shrinkage. ΔR of neat epoxy after curing was the smallest. It might be because neat epoxy had the highest modulus and the lowest matrix fracture toughness among four matrix series.

In the same stress, the reaching time and strain of carbon fiber/epoxy-AT-PEI specimens relating to the measurement of electrical resistivity appeared differently with their mechanical properties of matrix. Fig. 10 shows the changes in electrical resistivity (Δρ) and strain for carbon fiber reinforced (a) neat epoxy and (b) 15 phr AT-PEI specimens under cyclic stress. In the same stress, the reaching time for neat epoxy were faster than that of 15 phr AT-PEI. The result could be related to their matrix modulus. Mobility of carbon fiber in neat epoxy matrix might be small due to high matrix modulus, whereas in 15 phr AT-PEI case, the mobility in the interface might be high due to high matrix toughness and low modulus. Neat epoxy with high modulus need faster reaching time until the same stress. The change of stress and stain was correspondence with the behavior of electrical resistivity.

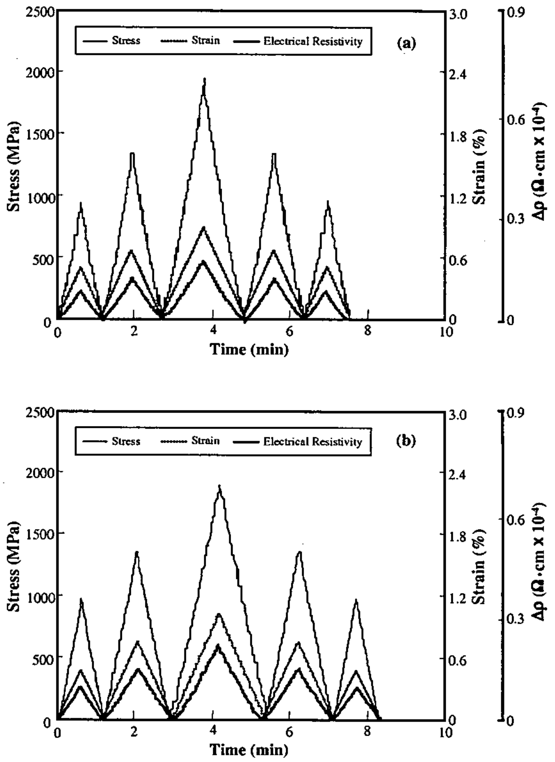

Fig. 10 The change of strain and electrical resistivity for single carbon fiber/epoxy-AT-PEI composite under changable stress.

Fig. 11 shows (a) strain-stress and (b) change of electrical resistivity-stress curves for carbon bare fiber or carbon single fiber/epoxy-AT PEI composite with AT-PEI content. Apparent modulus of 15 phr AT-PEI with high fracture toughness was lower than that of neat epoxy. The slope of strain-stress curves was apparent modulus that increased with improving matrix modulus. The apparent modulus means the fiber modulus embedded in the matrix in stress-strain curve comparing to a bare fiber modulus in itself[10]. The tendency of apparent modulus was consistent well with the results of electrical resistivity measurement.

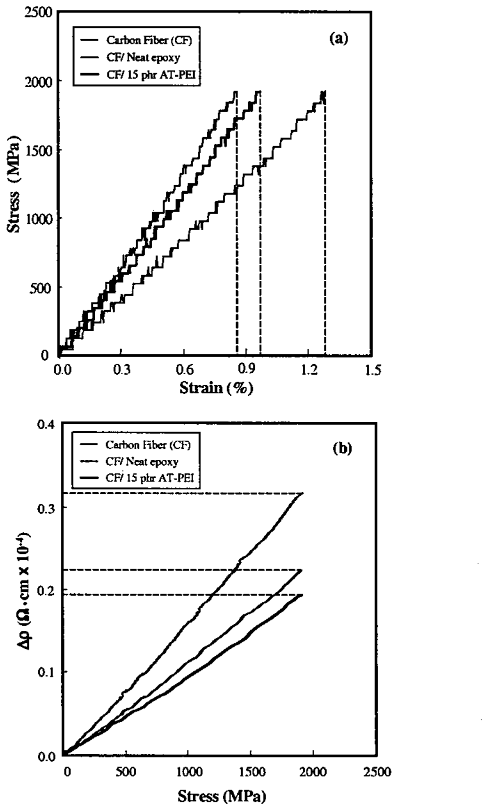

Fig. 11 (a) Stress-strain and (b) change in electrical resistivity-stress curves for carbon fiber/epoxy-AT-PEI composite.

4. Conclusions

Interfacial and electrical properties for the carbon fiber reinforced epoxy-AT-PEI composites were performed using microdroplet test and electrical resistance measurement. With adding AT-PEI content, the fracture toughness of epoxy-AT-PEI matrix increased, and IFSS was improved due to the improved matrix fracture toughness. The microdroplet with the carbon fiber and neat epoxy system showed rather brittle microfailure pattern. For higher AT-PEI content, ductile microfailure mode appeared. The changes in electrical resistance (ΔR) after curing increased gradually with adding AT-PEI. The matrix fracture toughness was directly proportional to IFSS and ΔR. Under cyclic stress, the reaching time of neat epoxy case was faster than that of 15 phr AT-PEI. It might be because apparent modulus of neat epoxy was higher than that of 15 phr AT-PEI case. The results obtained from measuring electrical resistance during curing process, and measurement of electrical resistivity under cyclic stress were correspondence well with the matrix mechanical properties such as modulus and toughness.

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