Interfacial Evaluation and Damage Sensing of Carbon Fiber/Epoxy-AT-PEI Composite using Electro-Micromechanical Techniques

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Electro-micromechanical 시험법을 이용한 탄소섬유 강화 Epoxy-AT PEI 복합재료의 손상 감지능 및 계면물성 평가

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Key Words : fracture toughness, electro-micromechanical test, interfacial shear strength (IFSS), thermal expansion coefficient (TEC), electrical resistivity (Δρ), stress/strain sensing

ABSTRACT

Interfacial evaluation and damage sensing of the carbon fiber/epoxy-amine terminated (AT)-polyetherimide (PEI) composites were performed using micromechanical test and electrical resistance measurement. As AT-PEI content increased, the fracture toughness of epoxy-AT-PEI matrix increased, and thus their interfacial shear strength (IFSS) was improved due to the improved toughness. After curing process, the changes in electrical resistance (ΔR) with increasing AT-PEI contents increased gradually because of the changes in thermal expansion coefficient (TEC) and thermal shrinkage of matrix. Matrix fracture toughness was correlated to the IFSS, residual stress and electrical resistance. The results obtained from the electrical resistance measurement during curing process, reversible stress/strain, and durability test were consistent with modified matrix toughness properties.

Nomenclature

τ	: Interfacial shear strength (IFSS)
σ_R	: Residual stress
K_{IC}	: Fracture toughness
ΔR	: Change of electrical resistance
$\Delta\rho$	: Change of electrical resistivity
L_{ec}	: Electrical Contact length
TEC	: Thermal expansion coefficient

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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 (T_g), stiffness and strength of the cured thermosetting resin. High performance thermoplastics, such as poly(ethersulfone) (PES), poly(etherimide) (PEI), polycarbonate (PC) and poly(phenyleneoxide) (PPO), or a combination of rubber and thermoplastics, are commonly added to thermosetting resin as a processing modifier. They increased the fracture toughness without reducing thermal and mechanical properties [3-5]. The electro-micromechanical technique has been studied as an economical and new nondestructive evaluation (NDE) method for curing monitoring, stress/strain sensing, characterization of interfacial properties, and nondestructive behavior because conductive fiber can act as an inherent sensor in itself as well as a reinforcing fiber [6,7]. Residual stress in the fiber reinforced composite occurs during curing process due to the thermal contraction of the matrix and/or the difference in thermal expansion coefficient (TEC) between fiber and matrix. The effect on residual stress is reflected in the mechanical performance of cured composites [8]. In this work, interfacial properties, residual stress and cure monitoring of carbon fiber reinforced amine terminated (AT)-PEI toughened epoxy matrix composites were investigated using micromechanical test and electrical resistance measurement. The change in electrical resistance (ΔR) during curing process, under cyclic changeable stress/strain and durability test were correlated with matrix toughness.

2. EXPERIMENTAL

2. 1. Materials

Two kinds of carbon fibers were used and their average diameters were about 18 μm (Mitsubishi, Chemical Co., Japan) and 8 μm (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. Specimen Preparation and IFSS Measurement: The carbon fiber with 18 μm diameter was fixed with regularly separated distance in a steel frame. Microdroplets of neat epoxy and epoxy-AT-PEI matrix were formed on each carbon fiber of 18 μm using carbon fiber of 8 μm in diameter as a tip pin. Microdroplet specimens were cured with same curing stages. A microdroplet specimen was fixed by the microvice using a specially designed micrometer. The IFSS, τ was calculated from the measured pullout force, F using the following equation,

$$\tau = \frac{F}{\pi D_f L} \tag{1}$$

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

2.2.2. Electrical Resistance Measurements: Figure 1 shows the scheme of electrical resistance measurement (a) during curing and (b) under cyclic loading. During curing process and under cyclic loading, the electrical resistance was measured using a HP34401A digital multimeter. For durability test the specimen was soaked in 98°C boiling water for 3 hours and absorbed moisture was evaporated during measuring the electrical resistance. In compressive Broutman test, the change of electrical resistance was measured relating to acoustic emission (AE, Mistras 2001 system, Physical Acoustic Co.) parameters. For the electrical resistance measurements under cyclic load, strain-stress curves 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 the electrical resistivity, ρ is as follows:

$$\rho = \left(\frac{A}{L_{ec}}\right) \times R \tag{2}$$

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.

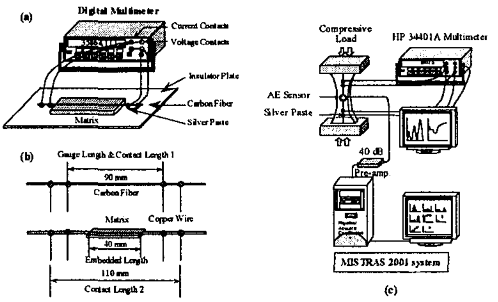

Fig. 1 Scheme of electrical resistance measurements: (a) curing; (b) cyclic loading: (c) compressive testing.

Residual Stress and FEM Analysis: It is known that residual stress occurred by TEC between fiber and matrix during curing and subsequent cooling process. The residual stress in the fiber, σ_{Rf} was calculated from following equation,

$$\sigma_{Rf} = \frac{E_f E_m V_m (\alpha_m - \alpha_f) \cdot \Delta T}{(V_m E_m + V_f E_f)} \tag{3}$$

where E_f is the modulus of fiber, E_m is the modulus of matrix, α_f is the TEC of fiber, α_m is the TEC of matrix, V_f is the volume fraction of fiber, V_m is the volume fraction of matrix, and ΔT is the temperature change.

The shrinkage of the epoxy-AT-PEI matrix is higher than that of the carbon fiber. The residual stress of matrix in single fiber composite (SFC) was calculated simply from TEC of each components and the modulus of SFC specimen. Because the volume fraction of matrix is too high in the SFC, the residual stress of matrix can generally be estimated by the following equation,

$$\sigma_{Rm} = (\alpha_m - \alpha_f) \cdot \Delta T \cdot E(\varepsilon) \tag{4}$$

where $E(\varepsilon)$ is the modulus which obtained the measured stress-strain curve of the specimen.

FEM was performed by means of commercial ANSYS 5.5. Nonlinear approach in FEM was used under the multi-linear curve resulted from true stress-strain curve. The relationship between stress components and equivalent residual stress was designated by von Mises criterion.

3. RESULTS AND DISCUSSION

3.1. Matrix Fracture Toughness and IFSS: Interfacial properties of carbon fiber reinforced AT-PEI modified epoxy matrix were compared to matrix fracture toughness. Figure 2 shows the fracture toughness of epoxy-AT-PEI matrix and IFSS of carbon fiber/epoxy-AT-PEI composite with AT-PEI content. The fracture toughness was improved gradually with increasing AT-PEI content. 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 improving fracture toughness by adding AT-PEI content. IFSS increased with increasing AT-PEI content due to the enhanced fracture toughness and energy absorption mechanisms.

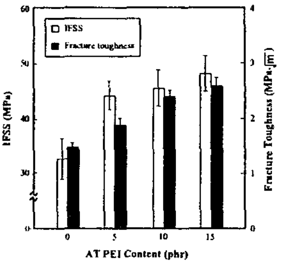

Fig. 2 Matrix fracture toughness and IFSS of carbon fiber/epoxy-AT-PEI composite.

Figure 3 shows SEM photographs of typical microfailure modes for the carbon fiber reinforced (a) neat epoxy and (b) 15 phr AT-PEI composite. Neat epoxy microdroplet appeared brittle microfailure mode, whereas high content of AT-PEI microdroplet exhibited more likely plastic deformation and ductile microfailure mode. Typical failure mode of fracture surface was generally changed from smooth to rough and the brittle nature became tougher with adding AT-PEI content.

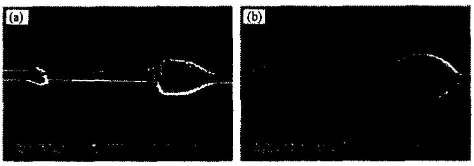

Fig. 3 Typical microfailure modes of carbon fiber/epoxy-AT-PEI composites.

3.2 Electrical Resistance and Residual Stress: Figure 4(a) shows the change of electrical resistance for carbon fiber/epoxy-AT-PEI composite with AT-PEI content during curing process. As AT-PEI content increased, electrical resistance after curing process increased gradually. The changes in electrical resistance (ΔR) during curing may be affected by matrix thermal shrinkage or residual stress determined by matrix toughness. The higher fracture toughness, the higher cure shrinkage. The change of electrical resistance of neat epoxy after curing was the lowest. It might be because neat epoxy had the highest modulus and the lowest matrix fracture toughness among four matrix series. Figure 4(b) shows the changes of electrical resistivity (Δρ) for carbon fiber/epoxy-AT-PEI composite under durability test. The electrical resistance increased gradually, and then saturated. It could be because evaporating moisture from matrix resulted in the recovering matrix modulus. Tables 1 and 2 show mechanical, interfacial, thermal, electrical properties and residual stress of carbon fiber/epoxy-AT-PEI composite. As AT-PEI content increased, modulus decreased, whereas fracture toughness, IFSS, TEC, electrical resistance and residual stress increased gradually. Residual stress obtained by calculation method was consistent well with FEM results.

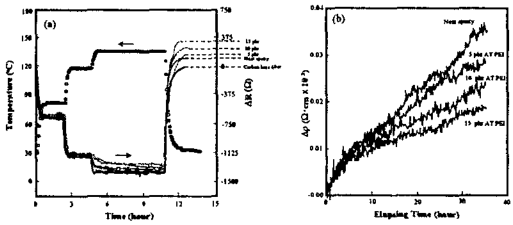

Fig. 4 Changes of electrical resistance for carbon fiber/epoxy-AT-PEI composite.

Table 1 Mechanical, thermal and electrical properties of carbon fiber/epoxy-AT-PEI composite and matrix

AT-PEI Content (phr)	Modulus (GPa)	K_{IC} (MPa·$\sqrt{m}$)	IFSS (MPa)	TEC (℃$^{-1}$)	ΔR[2] (Ω)
0	2.0 (0.4)[1]	1.4 (0.1)	32.7 (7.5)	81.4	140
5	1.8 (0.2)	1.8 (0.1)	44.2 (4.5)	57.1	164
10	1.7 (0.1)	2.3 (0.1)	46.8 (6.5)	69.1	247
15	1.6 (0.2)	2.6 (0.2)	48.6 (6.6)	76.6	278

1) Standard deviation (SD)
2) After curing

Table 2 Residual stress of carbon fiber/epoxy-AT-PEI composite.

AT-PEI Content (phr)	Residual Stress (MPa)			
	Calculation		FEM Analysis	
	Fiber	Matrix	Fiber	Matrix
0	1596	16.1	1617	15.6
5	1120	10.2	1134	9.8
10	1355	11.6	1372	11.3
15	1502	12.1	1521	11.8

Figure 5 shows equivalent stress on (a) the fiber and (b) the matrix obtained from FEM. The equivalent stress of the fiber was low at both ends of specimen and it was high at the center region, whereas that of matrix was high at both ends of specimen and it was low at the center region. Both residual stresses built on the fiber and matrix increased with adding AT-PEI content. Compressive stress on fiber induced because TEC of the matrix is higher than that of the fiber.

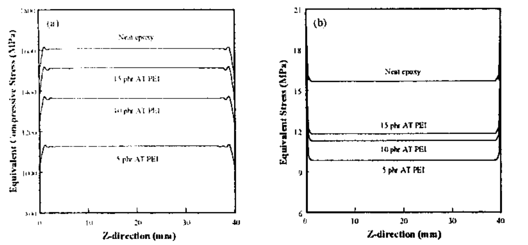

Fig. 5 Equivalent stress of (a) carbon fiber and (b) epoxy-AT-PEI matrix by FEM.

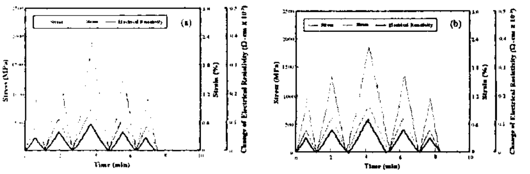

Fig. 6 Changes of strain and electrical resistivity under changeable stress.

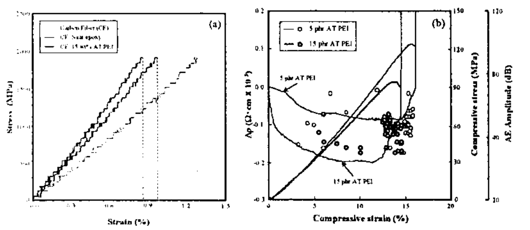

Fig. 7 (a) Apparent modulus and (b) Δρ, strain-stress curve and AE amplitude.

3.3. Strain/Stress Sensing: Under the same stress, the strain of carbon fiber/epoxy-AT-PEI specimens for measuring electrical resistivity appeared differently with their mechanical properties of matrix. Figure 6 shows the changes of strain and electrical resistivity for (a) neat epoxy and (b) 15 phr AT-PEI under changeable stress. The strain and electrical resistivity of neat epoxy were higher than those of 15 phr AT-PEI, and the reaching time until the same stress was faster in neat epoxy case. Compressive stress of carbon fiber in neat epoxy matrix might be small due to high matrix modulus, whereas in case of 15 phr AT-PEI, thermal shrinkage might be high due to high matrix toughness and low modulus. This tendency could be also related to their apparent modulus, which provides the interfacial information: i.e., apparent modulus means the fiber modulus embedded in the matrix in stress-strain curve comparing to the carbon bare fiber modulus in itself [9]. Figure 7(a) strain-stress curves with AT-PEI content. The slope in curve was apparent modulus that increased with improving matrix modulus. Apparent modulus was consistent well with the changes in electrical resistivity (Δρ). Figure 7(b) shows Δρ, strain-stress curve and AE amplitude of carbon fiber/ epoxy-AT-PEI composite in compressive Broutman test. In initial state, Δρ decreased steadily and then increased with AE signals detected from fiber fracture. The change of Δρ of 15 phr AT-PEI was higher than those of 5 phr AT-PEI case.

4. CONCLUSIONS

Interfacial and electrical properties for the carbon fiber/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 fracture toughness. The changes in electrical resistance (ΔR) during curing and TEC increased gradually with increasing AT-PEI content. The matrix fracture toughness was directly proportional to IFSS, TEC, residual stress and electrical resistance. In changeable stress test, the strain and electrical resistivity of neat epoxy were higher than those of 15 phr AT-PEI, and the reaching time until the same stress was faster in neat epoxy case. It might be because of increasing apparent modulus of matrix. The results obtained from measuring electrical resistance during curing process, under cyclic stress/strain, compressive Broutman and durability tests were correspondence well with matrix mechanical properties including modulus and toughness.

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