Microfailure Mechanisms of Single-Fiber Composites Using Tensile/Compressive Fragmentation Techniques and Acoustic Emission

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인장/압축 Fragmentation 시험법과 음향방출을 이용한 단 섬유 복합재료의 미세파괴 메커니즘

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KEY WORDS : Tensile fragmentation test, Compressive Broutman test, Acoustic Emission (AE), Interfacial Shear Strength (IFSS), Surface Treatments

ABSTRACT

Interfacial and microfailure properties of carbon fiber/epoxy matrix composites were evaluated using both tensile fragmentation and compressive Broutman tests with acoustic emission (AE). Amino-silane and maleic anhydride polymeric coupling agents were used via the dipping and electrodeposition (ED), respectively. Both coupling agents exhibited higher improvements in interfacial shear strength (IFSS) under tensile tests than compressive cases. However, ED treatment showed higher IFSS improvement than dipping case under both tensile and compressive test. The typical microfailure modes including fiber break, matrix cracking, and interlayer failure were observed during tensile test, whereas the diagonal slippage in fiber ends was observed during compressive test. For both the untreated and treated cases AE distributions were separated well under tensile testing. On the other hand, AE distributions were rather closer under compressive tests because of the difference in failure energies between tensile and compressive loading. Under both loading conditions, fiber breaks occurred around just before and after yielding point. Maximum AE voltage for the waveform of carbon or basalt fiber breakage under tensile tests exhibited much larger than those under compressive tests.

Nomenclature

τ_t , τ_c : Interfacial Shear Strength (IFSS) under Tensile and Compressive loading

σ_{fu} , σ_{fc} : Tensile and Compressive Strength of The Fiber

l_c : Critical Fragment Length

d : Fiber Diameter

1. INTRODUTION

Interfacial properties of the treated carbon fiber/epoxy matrix composites were evaluated using both the tensile and compressive fragmentation tests with an aid of AE. The commonly used tensile fragmentation test was interested in characterizing the fiber/matrix interfacial properties [1-3]. Transversal interfacial properties of the fiber/matrix were obtained by the single-fiber Broutman test to investigate the interface debonding and buckling behavior while subjecting to a transverse compressive stress [4,5]. During both testing, AE test monitored the fracture signals of microfailure sources simultaneously, and correlated with the interfacial shear strength (IFSS). IFSS can be improved by an introduction of chemical functional groups via the oxidation of fiber, plasma or commercial coupling agents. The electrodeposition (ED) is a process that a film is deposited on a conductive carbon fiber from a dispersion of colloidal particles (or ions) in aqueous solution with the optimized treating processes such as desired thickness [3].

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2. EXPERIMENTAL

2. 1. Materials

Carbon fiber was supplied from Tae Kwang Co. (TZ-307). Carbon fiber has a density of 1.8 g/cm^3 and average diameter of 7.9 μm, respectively. 98 μm basalt fiber were also used for the comparison of diameters. Epoxy resin based on diglycidyl ether of bisphenol-A was used as a matrix. Polyoxypropylene diamine (Jeffamine) was used as curing agents to provide optimum condition. γ-aminopropyl triethoxysilane (APS) and polybutadiene maleic anhydride (PBMA) coupling agents were used via the dipping and ED treatments, respectively.

2. 2. Methodologies

2. 2. 1.Single Fiber Strength and IFSS Measurements: Single carbon fiber tensile strength was obtained using about fifty specimens for statistical mean value. The fragmentation test was carried out to obtain IFSS using UTM and a specially designed mini-tensile fixture. Ultimate fragment lengths were measured, and subsequent failure process was observed via a polarized-light microscopy.. The relationship among fiber tensile strength σ_f, aspect ratio l_c/d, and IFSS, was given by Kelly-Tyson [6] equation and Weibull statistics:

$$\tau_t = \frac{\sigma_{fu} \cdot d}{2 l_c} \qquad (1)$$

where σ_{fu} is the tensile strength of the fiber at average critical length l_c, and d is the fiber diameter. The compressive stress on a fiber can be transferred perfectly across the break from one the fiber fragment to the other due to the fact that the fragments are still in contact with each other. A critical fragment length, as defined by the tensile load transfer model, does not exist in compressive system. According to the compressive profile, τ_c, based also on the force balance,

$$\tau_c = \frac{\sigma_{fc} \cdot d}{2 l_c} \qquad (2)$$

where critical length l_c is the original length of the fiber ($l_c = l_1$). σ_{fc} is the fiber stress at the point where the interfacial stress is insufficient to induce further fragmentation.

2. 2. 2. Preparation of Microspecimens: Tensile and Compressive Broutman microspecimens were made single fiber embedded in epoxy matrix in silicone mould as shown in figure 1.

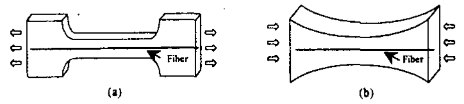

Fig. 1. Schematic illustrations showing for (a) tensile; and (b) compressive Broutman specimens

2. 2. 3. Fiber Surface Treatment by ED and Dipping: The carbon fibers acted as an anode in itself and the cathode was made of an aluminum plate. PBMA coupling agents were diluted to the suitable concentration in the deionized water. After anode frame and cathode bar was immersed into aqueous electrolyte solutions, voltage was supplied to both electrodes by power source. Typical immersing time and applied voltage were 10 minutes and 3 V, respectively. APS coupling agent was diluted to the required 0.5 wt.% concentration in aqueous solution for coating on carbon and basalt fiber surface for comparison, respectively.

2. 2. 4. AE Measurement: Micro-specimen was placed on the UTM for tensile/compressive tests. AE sensor was attached in the center of the specimen using a couplant. AE signals were detected using a miniature sensor (Resonance Type model, PICO by PAC) with peak sensitivity of 54 Ref V/(m/s) and resonant frequency at 500 kHz. The sensor output was amplified by 40 dB at preamplifier and passed through a band-pass filter with a range of 200 kHz to 750 kHz. The threshold level was set to 30 dB. Using in-built software AE waveforms were analyzed.

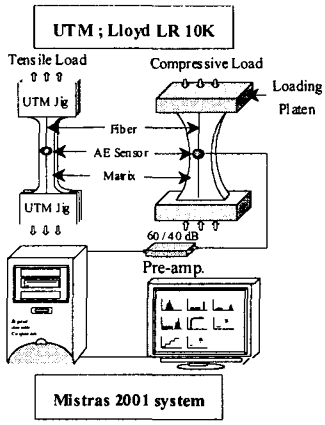

Fig. 2. Schematic diagram of instrumentation for AE

3. RESULTS and DISCUSSION

3. 1. Microfailure Modes of Tensile/Compressive Specimens

Figure 3 shows photographs for tensile and compressive microfailure. Untreated carbon fiber exhibited the debonding around fiber fracture and APS treated case exhibited the debonding and cone-shaped fiber fracture. ED treated carbon fiber case exhibited cone-shaped matrix crack, whereas in compressive specimen there were the diagonal slippage based on transversal tensile stress, characteristic of the transverse properties of the interface. Especially, in compressive test basalt fiber composites exhibited fiber fracture in center region of specimens.

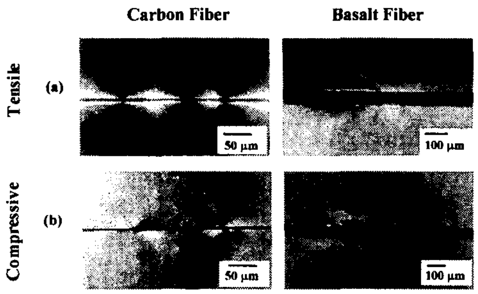

Fig. 3. Polarized-light photographs of the failure modes for carbon and basalt fiber under applied load: (a) Tensile; (b) Compressive.

3. 2. Comparison of IFSS Using Two Coupling Agents

Cases using two coupling agents show significant improvements in IFSS under both tensile and compressive fragmentation tests as shown in figure 4. It may be due to the primary and the secondary chemical bonding and physical interdiffusion between coupling agent and epoxy matrix. In the tensile and compressive tests ED treated coupling agent exhibited higher IFSS improvement than APS coupling agent case. It may be because better wetting effect and more uniform coating can contribute to affect favorably IFSS. On the other hand, the tensile test exhibited higher IFSS improvement compared to the case in the compressive test. It may be because of the different effectiveness of the stress transfer mechanisms between the longitudinal tensile load and the transversal shear stress.

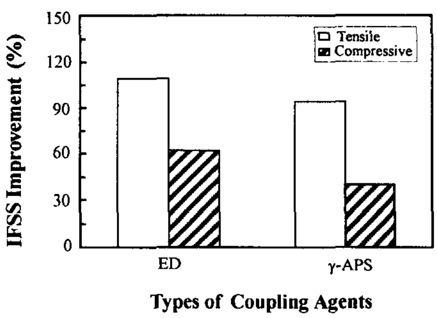

Fig. 4.IFSS improvements on two coupling agents

3. 3. AE Analysis with Microfailure Mechanisms

AE results showed the microfailure mechanisms for carbon and basalt fiber/composites in figure 5. They are separated well in tensile tests for both the untreated and the treated cases in carbon and basalt fiber composites. Whereas AE distributions are rather closer separated in compressive tests. Fracture energy in tensile failure may be much higher than the case in compressive test. It is because of the difference in fracture energies between the tensile and the compressive tests. For both untreated and treated cases, carbon and basalt fiber breaks occurred until just before yielding point under tensile test. Beyond yielding point, however, much more AE events occurred from the interlayer failure in both the ED and APS treated carbon cases, whereas basalt fiber composite exhibited matrix and interlayer failure around before and just after yield point. Ultimate stress in compressive test exhibited much higher than that of tensile test. All microfailures including fiber break, matrix cracking, and interlayer fracture can be correlated with their inherent materials properties. Basalt fiber composites exhibited significantly higher amplitude events than those of the carbon fiber composites due to higher fracture occurring from thicker fiber under tensile and compressive tests.

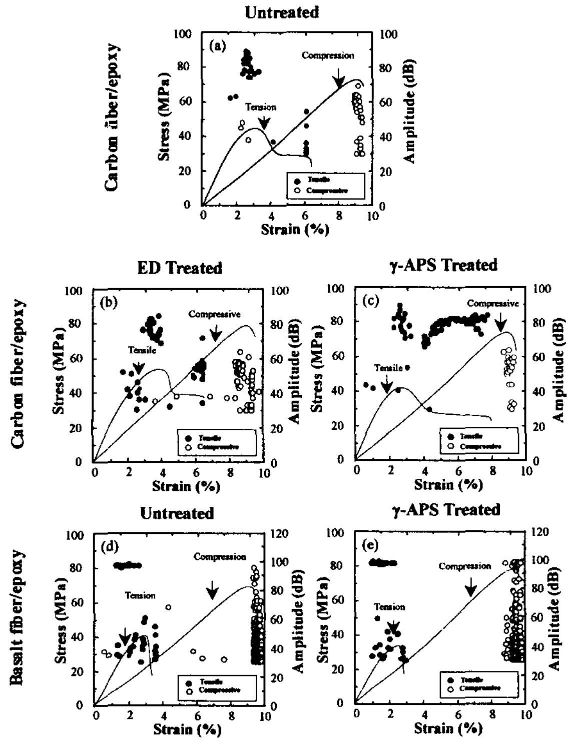

Fig. 5. Stress-strain curves and AE amplitudes for composite using tensile and compressive fragmentation tests: (a) Untreated, (b) ED treated carbon fibers, (c) 0.5 wt.% APS treated carbon fiber; (d) Untreated, (e) 0.5 wt.% APS treated basalt fiber.

Figure 6 shows AE waveforms in the untreated, ED treated and γ-APS treated carbon fiber/epoxy composites, respectively. In the case of tensile fragmentation test, there were so many intermediate size waveforms coming from the interlayer failure in the treated conditions. In case of compressive Broutman test, the interlayer failure with intermediate waveform overlapped with carbon fiber fracture signals. The maximum AE voltages coming from the fiber break waveform under tensile tests were much larger than those under compressive tests. Under tensile test ED and APS treated fiber waveform exhibited larger than the untreated case. In compressive test ED treated fiber waveform exhibited larger voltage than the untreated case and even than APS treated case. It may be due to the microfailure types and differing failure energies in compressive tests for both fibers.

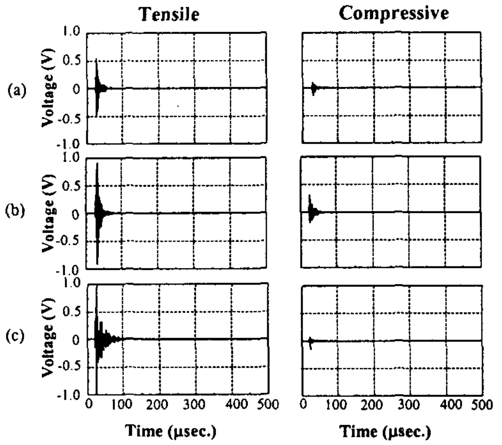

Fig. 6. AE waveforms in carbon fiber/epoxy composites: (a) Untreated, (b) ED treated fiber fracture, (c) 0.5 wt. % γ-APS treated fiber fracture under tensile and compressive tests.

Figure 7 shows AE waveform of the untreated and APS treated carbon fiber/epoxy composites, respectively. Tensile fiber fracture signal showed much larger signal voltage than the case of compressive test, as in the carbon fiber composites. However, signal size of basalt fiber were much larger compared to the signal of carbon fiber fracture, due to much larger diameter size. There were no significant differences in signal size between the untreated and APS treated cases. Especially, interlayer failure waveforms were not observed as carbon fiber case. Matrix signal was hard to distinguish from the debonding signal because of the overlapped outcome.

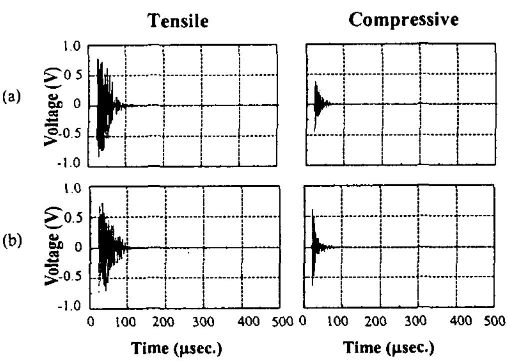

Fig. 7. AE waveforms in basalt fiber/epoxy composites: (a) Untreated, (b) 0.5 wt. % γ-APS treated fiber fracture under tensile and compressive tests.

4. CONCLUSIONS

Using fragmentation tests, IFSS was improved by using PBMA by ED and APS coupling agent by the dipping. ED treatment showed higher IFSS improvement due to the interlayer with the compact and uniform surface coating. In compressive test for carbon and basalt fiber composites, there were diagonal slippages based on transverse tensile stress characteristic of the transversal properties of the interface. AE test monitored the fracture signals of microfailure sources, such as fiber break, matrix cracking, especially diagonal slippage in the broken fiber ends, too. For both the untreated and treated cases AE events were separated well under tensile testing whereas AE distributions were rather closer under compressive tests, due to the difference in fracture energies between two tests. For all tests, carbon and basalt fiber breaks occurred around the yielding point. Beyond yielding much more AE events occurred from the interlayer failure in carbon fiber tensile cases, whereas basalt fiber did not show such distinct interlayer signals. Ultimate stress in compressive loading exhibited much higher than that of tensile loading. Maximum AE voltage for the waveform of two-fiber breaks under tensile tests exhibited much larger than those under compressive tests.

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