Interfacial and Nondestructive Evaluation of Single Carbon Fiber/Epoxy Composites by Fiber Fracture Source Location using Acoustic Emission

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Acoustic Emission 의 섬유파단 Source Location 을 이용한 Carbon Fiber/Epoxy Composites 의 계면특성 및 비파괴적 평가

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KEY WORDS: nondestructive evaluation (NDT), source location, tensile fragmentation, acoustic emission (AE), electrodeposition (ED), interfacial shear strength (IFSS)

ABSTRACT

Fiber fracture is one of the dominant failure phenomena to determine total mechanical properties in composites. Fiber fracture locations were measured by optical microscopic method and acoustic emission (AE) as functions of matrix toughness and surface treatment by the electrodeposition (ED), and then two methods were compared. Two AE sensors were attached on the epoxy specimen and fiber fracture signals were detected with elapsed time. The interfacial shear stress (IFSS) was measured using tensile fragmentation test and AE system. In ED-treated case, the number of the fiber fracture measured by an optical method and AE was more than that of the untreated case. The signal number measured by AE were rather smaller than the number of fragments measured by optical method, since some fiber fracture signals were lost while AE detection. However, one-to-one correspondence between the x-position location by AE and real break positions by optical method was generally established well. The fiber break source location using AE can be a valuable method to measure IFSS for semi- or nontransparent matrix composites nondestructively (NDT).

Nomenclature

- τ : Interfacial shear strength (IFSS)
- α : Scale parameters in Weibull distribution
- β : Shape parameters in Weibull distribution
- Γ : Gamma function
- l_c : Critical length
- C_L : Wave velocity
- Δt : Difference arriving time
- D : Distance of sensor
- V : Propagation velocity of a wave

1. INTRODUCTION

IFSS between fiber and matrix is one of the important factors to determine mechanical performance in fiber reinforced composites. Measurement of IFSS requires special micromechanical techniques. Some techniques to measure the IFSS are the single fiber composites test which is called as the fragmentation test [1], the single fiber microdroplet test which is also called as the single fiber full-out test [2] and the microindentation test.

AE technique as the nondestructive evaluation (NDE) has been widely used to study the fracture behavior of composite materials. AE is a sensitive method for detecting active damages inside composites through elastic wave propagation. Many works [3-5] have been reported on damage evaluation, identification of AE sources, the source location, the correlation between AE parameter and fracture mechanics in composites.

Among them, the source location is to calculate a spatial source location based on the arriving time difference of AE signal. It was possible to calculate the source location along a line between two sensors if the distance between the sensors and the velocity of wave propagation are known using called 'linear location method' [6,7].

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In this work, the interfacial properties of the untreated and ED treated carbon fibers/epoxy composites with curing agent ratios were studied, and fiber break source location by AE method was compared to real measured position by optical microscope.

2. EXPERIMENTAL

2. 1. Materials

Carbon fiber (Taekwang Co., TZ-307, Korea) has a density of 1.8 g/cm^3 and average diameter of 7.9 μm, whereas tensile strength and modulus are 3727 MPa and 245 GPa, respectively. Epoxy resin (Kukdo Chemical Co. YD-128, Korea) is based on diglycidyl ether of bisphenol-A (DGEBA). Polyoxypropylene diamene (Jeffamine D400 and D2000, Huntzman Pertochemical Co.) was used as curing agents. Various specimens were obtained by adjusting the relative proportion of D400 *versus* D2000 to control the epoxy toughness.

2. 2. Methodologies

2.2.1. Surface Treatment of Carbon Fiber: Fifty untreated carbon fibers with 7.9 μm diameter were fixed a suitable distance apart in a rectangular acrylic frame. The carbon fiber acted as the anode, whereas and aluminum plate works as the cathode. PBMA was diluted with 0.5 wt% concentration, and then the anode and cathode were immersed into aqueous electrolyte solution. 3 voltages were supplied to both electrodes by a power supply and the typical coating time was for 10 minutes. After ED treatment, the carbon fibers were dried at room temperature without further thermal treatment.

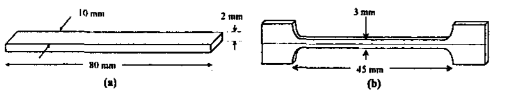


Fig. 1 Dimensional scheme of (a) calibration of matrix specimen and (b) fragmentation testing specimen

2.2.2. Preparation of Testing Specimens: Two-type specimens were used for the test of fiber break source location using AE. Figure 1(a) exhibits rectangular rod testing specimen to evaluate wave velocity of epoxy matrix by pencil-lead-break method with 3 curing agent ratios. The dimension was 80 mm in gauge length, 10 mm in width and 2 mm in thickness. Figure 1(b) shows the single carbon fiber composite to measure the IFSS for fiber break source location. The dogbone-shape specimen has the dimension of 2 mm in thickness, 3 mm in width, and 45 mm in gauge length.

2.2.3. AE Test: To evaluate IFSS and fiber fracture source location, fragmentation test was carried out using man-made mini-universal test machine (UTM). Ultimate fragment lengths were measured by a polarized-light microscope and in-built AE program. The specimens were tested by UTM (load sell of 1kN, speed rate of 1 mm/second) while AE monitoring. Two AE sensors were attached on the near curve-neck of the specimen using vacuum grease couplant.

AE signals were detected using a miniature sensor (Resonance Type, 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 gain. The threshold level was set up as 45 dB. The signal was fed into an AE signal process unit (MISTRAS 2001), where AE parameters were analyzed using in-built software. The typical AE parameters such as hit rate, peak amplitude, and event duration were investigated for the time and the distribution analysis. Schematic AE diagram of the linear location method is shown in Fig. 2.

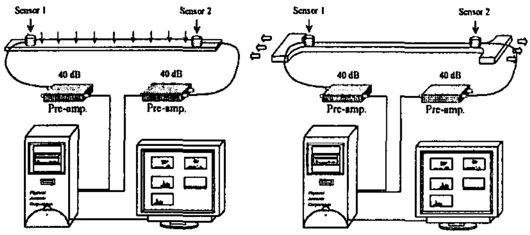


Fig. 2 AE system of (a) calibration of matrix wave velocity; and (b) linear source location testing

2.2.4. Wave Velocity Measurement of Matrix: Wave velocity of epoxy matrix was measured by two AE sensors attached with 45 mm distance apart in one dimensional plate specimen in Figure 2(a). AE signal was generated by pencil-lead-break. The break points obtained by in-built AE source location program were compared with real break points given by pencil-lead-breaks. By trial and error, the wave velocity of epoxy specimens were determined when the real break points showed a good correspondence to the point of AE signals. The source location using difference arriving time is accurate method. The difference arriving time (Δt) is given as

$$\Delta t = \frac{D}{V} \tag{1}$$

where D is the distance of sensor, V is propagation velocity of a regular wave and Δt is arriving time difference. The critical location, d is given as

$$d = \frac{1}{2}(D - \Delta t \cdot V) \tag{2}$$

where d is distance according to the first arriving sensor.

2.2. 5. IFSS Measurement: To measure IFSS, a specially designed mini-tensile testing machine was used combined with AE system and a polarized-light microscope. After the testing specimen was fixed in the mini-tensile testing machine, the composite was strained incrementally and the fiber was broken into small fragments embedded in the matrix until no longer fiber fracture occurred.

IFSS was determined using Drzal equation [8] that was modified from Kelly-Tyson equation [9]. By introducing the Weibull distribution for the aspect ratio, IFSS was in the following equation as

$$\tau = \frac{\sigma_f}{2 \cdot \alpha} \cdot \Gamma[1 - 1/\beta] \tag{3}$$

where α and β are scale and shape parameters of Weibull distribution for aspect ratio (l_c/d), and Γ is the gamma function.

3. RESULTS AND DISCUSSION

3.1. Wave Velocity of Epoxy Matrix: Figure 3 shows calibration curve of epoxy matrix with curing ratios by pencil-lead-break method. Wave velocity of matrix was calculated by arriving time difference using in-built AE program by breaking the pencil lead with impact interval of 5 mm in the rod specimen. The impact point of matrix was nearly correspondence well with AE detect point. Average error range of epoxy matrix was between 0.2 % and 1 %.

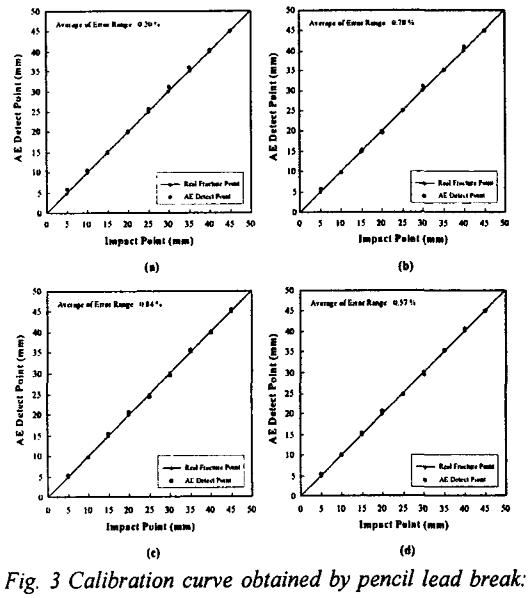


Fig. 3 Calibration curve obtained by pencil lead break: (a) acid anhydride (b) 2.5:0.5, (c) 2.7:0.3, (d) 3.0:0.0

Table 1 shows density and wave velocity of epoxy matrix depending on different curing agent ratios. Wave velocity was measured by the pencil lead break which is method comparing real distance of impact interval of 5 mm to the difference arriving time measured from two senses. As the curing agent content of D2000 decreased, the wave velocity measured by AE increased, whereas epoxy resin matrix was more brittle. In case of epoxy matrix using acid anhydride, the wave velocity was 1580.

Table 1 Wave velocity of epoxy matrix as curing agent content

Type of Materials	Curing Agent	Density (g/cm³)	Wave Velocity (c_L)	Error Range (%)
Epoxy (YD-128)	D400:D2000 (2.5 : 0.5)	1.073	1480	0.89
	D400:D2000 (2.7 : 0.3)	1.078	1560	0.78
	D400:D2000 (3.0 : 0.0)	1.098	1660	0.65
	Acid Anhydride	1.216	1580	0.81

- Interval of impact point: 5 mm
- Distance of sensors: 50 mm
- Pencil lead break method

3.2. Source Location Outcomes: If a failure occurs the inside of the specimen, the first hit and the source location were designated by difference of the arriving time in Figure 4(a) and 4(b). Figure 4(c) and 4(d) are magnified Figure 4(a) and (b). The arriving time difference (Δt) at the fracture point (a) is shorter than that at the fracture point (b)

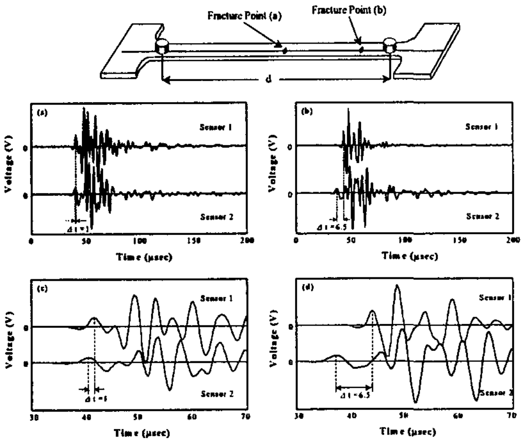
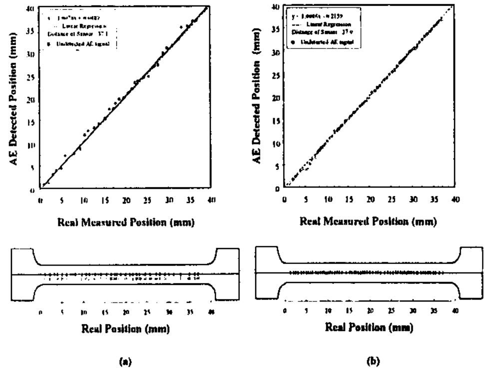


Fig. 4 Source location using arriving time difference (D400:D2000 = 2.5:0.5)

Figure 5 and 6 showed source location results of the untreated and ED-treated carbon fiber/epoxy composites with curing agent ratios. AE detected fracture point signals, whereas real fracture points were observed by optical method. Epoxy matrix became more brittle as curing agent content of D2000 decreased. However, according to fragment number determining IFSS, the 2.7:0.3 ratio might be known to be the optimum compared to either more ductile 2.5:0.5 ratio or more brittle 3.0:0.0 ratio.

Fragment number of ED-treated case increased compared to the untreated case. It might be because ED-treated carbon fiber with many functional groups on fiber surface enhanced chemical bonding with epoxy resin.



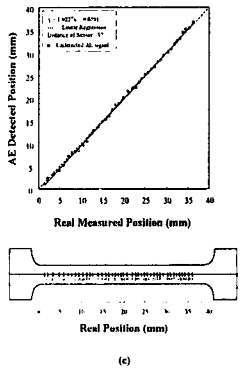


Fig. 5 Source location (the untreated): (a) 2.5:0.5, (b) 2.7:0.3, (c) 3.0:0.0

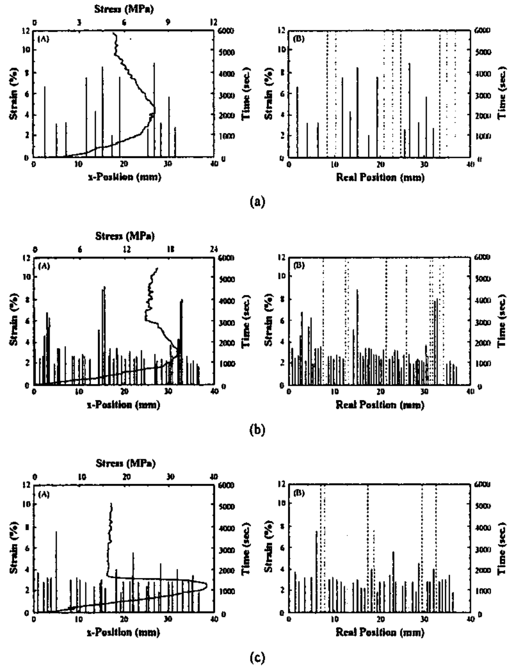
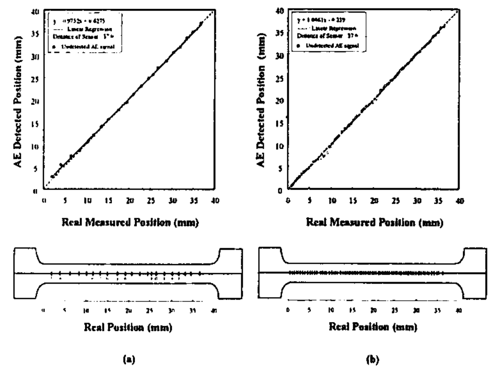


Fig. 7 (A) AE detected position and (B) real measured position as a function of time, strain and stress (the untreated carbon fiber): (a) 2.5:0.5, (b) 2.7:0.3, (c) 3.0:0.0



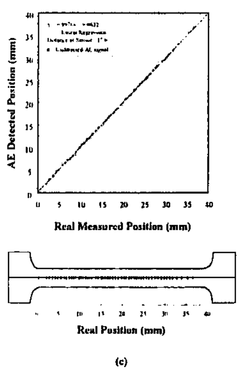


Fig. 6 Source location (ED-treated): (a) 2.5:0.5, (b) 2.7:0.3, (c) 3.0:0.0

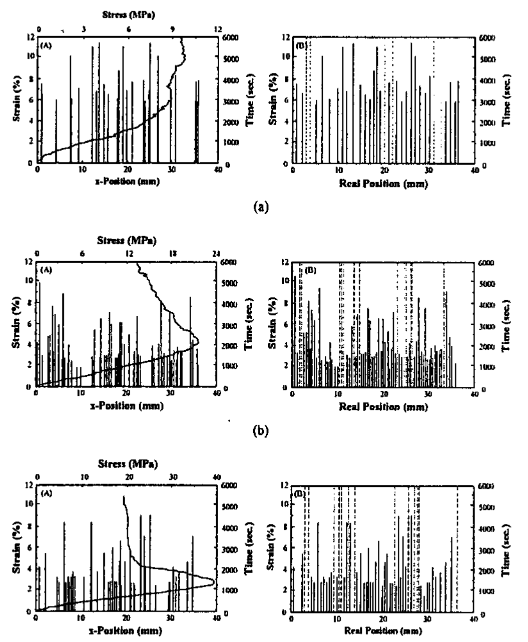


Fig. 8 (A) AE detected position and (B) real measured position as a function of time, strain and stress (ED-treated carbon fiber): (a) 2.5:0.5, (b) 2.7: 0.3, (c) 3.0:0.0

Figure 7 and 8 showed (A) AE detected position and (B) real measured position with elapsed time, stress-strain in the untreated and ED-treated composites with 3 curing agent ratios. A number of signal measured by AE system were rather smaller than fragment number measured by optical method. AE system could not detect some fracture signals of carbon fiber probably due to low carbon fiber signals and damping effect of epoxy matrix. Dot lines means lost signals.

3. 3. Microfailure Modes and IFSS with Curing Agent Ratios: Figure 9 showed the photographs of microfailure modes of the untreated carbon fiber composite with curing agent ratio under tensile test. The fiber fractures of 2.5:0.5 ratio exhibited a round shape because epoxy matrix was more ductile. In case of 2.7:0.3, a fiber facture was a sharp cone shape. The fiber failure modes of ductile 3.0:0.0 content appeared only debonding after the fiber fractured. The interfacial failure mode of carbon fiber showed good bonding in 2.7:0.3 ratio compared to ductile 2.5:0.5 and brittle 3.0:0.0 ratios. In a same magnification, 2.5:0.5 and 3.0:0.0 contents were observed only one fiber fracture, whereas in 2.7:0.3 ratio, two fiber fractures appeared.

(a) (b) (c)

Fig. 9 Microfailure modes on the untreated carbon fiber/epoxy composite in tensile test: (a) 2.5:0.5, (b) 2.7:0.3 and (c) 3.0:0.0

Table 2 IFSS of the untreated and ED-treated carbon fiber/ epoxy composites

Types	Curing Agent	Average Fragment Length (μm)	Aspect Ratio (l_c/d)	Fiber Strength (MPa) 1)	Scale Parameter (α) 2)	Shape Parameter (β) 2)	IFSS (MPa) 3)
Untreated	D400:D2000 (2.5:0.5)	1696	212	3777	236	3.68	10.1
	D400:D2000 (2.7:0.3)	571	71	4613	78	4.90	34.7
	D400:D2000 (3.0:0.0)	829	104	4307	113	4.65	24.4
Treated	D400:D2000 (2.5:0.5)	1088	136	4098	150	3.97	16.8
	D400:D2000 (2.7:0.3)	474	59	4772	64	5.26	42.8
	D400:D2000 (3.0:0.0)	623	78	4538	87	3.26	34.1

1) Fiber strength of critical fragment length: l_c
2) Values from aspect ratio
3) Drzal Eq., $\tau = (\sigma_f/2 \cdot \alpha) \cdot \Gamma(1-1/\beta)$

Table 2 showed interfacial properties and statistical Weibull parameters for the untreated and ED-treated composites with curing agent ratios. The average fragment length and aspect ratio, l_c/d, of the untreated carbon fiber/epoxy composite were larger than that of ED-treated case. The scale parameter of ED-treated case was smaller than that of the untreated case, whereas the shape parameter did not show a significant difference between both cases.

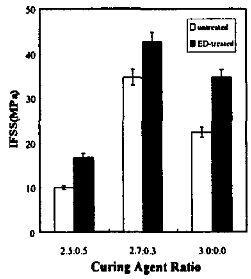


Fig. 10 IFSS of the untreated and ED-treated carbon with curing agent ratio

4. CONCLUSIONS

Fiber fracture locations were measured by optical microscopic method and AE with curing agent ratios for changing matrix modulus and the surface treatment by the ED. The IFSS by tensile fragmentation test and AE were compared. In ED-treated case, the number of the fiber fracture measured by an optical microscope and AE was more than that of the untreated case. The signal number measured by AE were rather smaller than the number of fragments measured by optical microscope, since some fiber fracture signals were missed while AE detection, due to too low carbon fiber fracture signals and ductile epoxy modulus. However, one-to-one correspondence between the x-position location by AE and real break position by optical microscope was generally established well. The fiber break source location using AE can be a valuable method to measure IFSS for nontransparent matrix composites nondestructively (NDT).

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