

Nondestructive Evaluation and Interfacial Damage Sensing of PVDF embedded Polymer Composites using Micromechanical Techniques and Acoustic Emission

Jin-Woo Kong^{*}, Joung-Man Park^{*,†}, Ki-Bok Kim^{**}, Dong-Jin Yoon^{**}

Micromechanical 시험법과 AE 를 이용한 PVDF 함침 고분자 복합재료의 계면손상감지능 및 비파괴적 평가 연구

공진우^{*} · 박종만^{*,†} · 김기복^{**} , 윤동진^{**}

KEY WORDS: nondestructive evaluation (NDE), damage sensing, polyvinylidene fluoride (PVDF), lead-zirconate-titanate (PZT), micromechanical technique, acoustic emission (AE)

ABSTRACT

Conventional piezoelectric lead-zirconate-titanate (PZT) sensor has high sensitivity, but it is very brittle. Recently polymer films such as polyvinylidene fluoride (PVDF) have been used as a sensor. The advantages of PVDF are the flexibility and mechanical toughness. Simple process and possible several shapes are also additional advantages. PVDF sensor can be directly embedded and attached to a structure. In this study, PVDF sensor was embedded in single glass fiber/epoxy composites whereas PZT sensor with AE was attached to single fiber composites (SFC). Piezoelectric sensor responds to interfacial damage of SFC. The signals measured by PVDF sensor were compared to PZT sensor. PZT sensor detected the signals of fiber fracture, matrix crack, interfacial debonding and even sensor delamination, whereas PVDF sensor only detected fiber fracture signals so far, because PZT sensor is much more sensitive than current PVDF sensor. Wave voltage of fiber fracture measured by PVDF sensor was lower than that of PZT sensor, but the results of fast Fourier transform (FFT) analysis were same. Wave velocity using two PZT sensors was also studied to know the internal and surface damage effect of epoxy specimens.

Nomenclature

Δt : Difference arrival time
 V : Wave velocity
 D : Distance between two sensor
 τ : Interfacial shear stress (IFSS)
 σ_f : Single fiber tensile strength
 L_c : Critical fragment length
 d : Fiber diameter

1. INTRODUCTION

AE is well known as one of the important nondestructive evaluation (NDE) methods. Acoustic

emissions are transient elastic waves generated by abrupt deformation within materials or structures. The elastic wave can be detected and monitored by sensors to provide information about AE source location and AE source characteristics, which in turn can aid structural damage assessment [1,2]. A very useful method for evaluation AE has been to correlate AE signal energy with AE source physical process that essentially is rapid release of elastic energy, such as fracture. The AE can monitor the fracture behavior of composite materials, and can characterize many AE parameters to understand the type of microfailure sources during the fracture progressing. When tensile loading is applied to a composite, AE signal may occur from fiber fracture, matrix cracking, and debonding at the fiber-matrix interface [3-5].

Piezoelectric lead-zirconate-titanate (PZT) has been used for such as buzzer, speakers and ultrasonic generators. Especially, PZT sensor has an excellent sensitivity and a wide application of the structure, but it is very brittle. Recently polymer films such as polyvinylidene fluoride (PVDF) have come into

^{*}Department of Polymer Science and Engineering, Research Center for Aircraft Parts Technology, Gyeongsang National University

^{**}NDE Group, Center for Safety Measurement Korea Research Institute of Standards and Science

[†]To whom correspondence should be addressed.

increasing use as a sensor. PVDF sensor has low density and good sensitivity, and is mechanically tough. PVDF sensor can be directly attached or embedded to a structure without disturbing its mechanical motion [6-8].

In this study, interfacial microfailure properties glass fiber/epoxy composites were measured by PVDF sensor and PZT sensor using AE. PVDF sensor was embedded into the epoxy matrix whereas AE sensor was attached on epoxy matrix surface.

2. EXPERIMENTAL

2.1. Materials

E-glass fiber of 30 μm was supplied from Dow Corning Co. The elastic modulus of glass fibers were 58.6 GPa. Epoxy resin (Kukdo Chemical Co. YD-128, Korea) is based on diglycidyl ether of bisphenol-A (DGEBA). Polyoxypropylene diamine (Jef-famine D400 and D2000, Huntzman Pertochemical Co.) was used as curing agents. Lab-made PVDF (Measurement Specialties Inc.) sensor was supplied from NDT laboratory of Korea Research Institute of Standards and Science (KRISS).

2.2. Methodologies

2.2.1. Preparation of Damage Testing Specimens

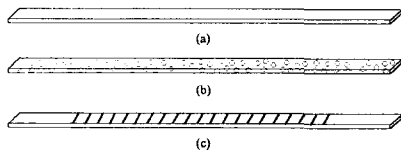


Fig. 1. Dimensional sachem of calibration of epoxy resin specimens; (a) no air bubble, (b) air bubble; and (c) surface damage

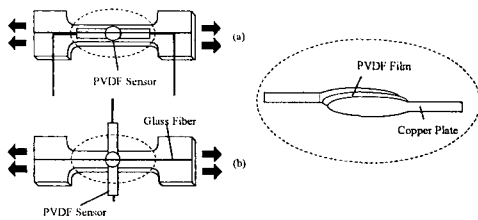


Fig. 2. Test specimen of single glass fiber composites with embedded PVDF sensor: (a) parallel; (b) vertical.

Above specimens were used for tensile test, damage sensing and AE test. Figure 1 exhibits a testing specimen to evaluate wave velocity as a function of various conditions. The specimen of Figure 1(a) has no damage and no air bubble, whereas Figure 1(b) has an air bubble and Figure 1(c) has some surface damage. Figure 2 is the SFC specimens for the measurement of AE signals by PVDF sensor and PZT sensor. AE signals measured by PVDF were compared to those measured by PZT sensor.

2.2.2. AE Test:

To measure wave velocity of various condition epoxy resins and the signals of internal and surface damages, AE was tested. Two AE sensors were attached on the side of the specimen using vacuum grease couplant.

AE signals were detected using a miniature PZT sensor (Resonance type, PICO by PAC) and lab-made PVDF sensor. PZT sensor was attached on the surface of the specimen whereas PVDF sensor was embedded in the specimen. PZT sensor has the peak sensitivity of 54 Ref V(m/s) and resonant frequency at 500 kHz. The outputs of two sensors were amplified by 40 dB at preamplifier gain. The threshold levels were set up as 30 dB for PZT sensor and as 35 dB for PVDF sensor, respectively. Because of the noise of PVDF sensor, the threshold level was rather higher than that of PZT sensor. 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 calibration of wave velocity and measurement of interfacial damages is shown in Figure 2.

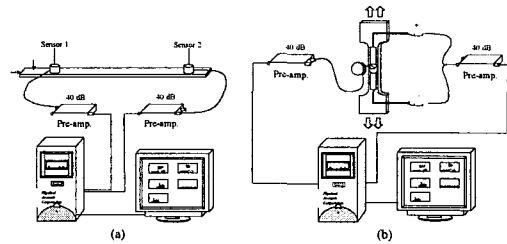


Fig. 2. AE system for (a) calibration of wave velocity and (b) measurement of interfacial damages

2.2.3. Wave Velocity Measurement of Matrix

Wave velocity of epoxy matrix was measured by two PZT sensors attached with 100 mm distance apart in one dimensional plate specimen in Figure 2(a). AE signal was generated by pencil-lead-break method. The impact points were either upper or side directions near to sensor 1. By trial and an error, the wave velocity of epoxy specimens was determined. The wave velocity was calculated by the measurement of the difference arriving time, Δt . Δt is given as

$$\Delta t = \frac{D}{V} \quad (1)$$

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

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

where d is distance according to the first arriving sensor

2.2. 4. IFSS Measurement

To measure IFSS, AE and a polarized-light microscope were used. After the testing specimen was fixed on 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, τ was determined using Kelly-Tyson equation [9] as

$$\tau = \frac{\sigma_f \cdot d}{2 \cdot L_c} \quad (3)$$

where d is the fiber diameter, σ_f is the single fiber tensile strength at the critical fragment length L_c .

3. RESULTS AND DISCUSSION

3.1. Wave Velocity of Epoxy Matrix

Table 1 Wave velocity of epoxy specimen as damage condition of air bubble

Material type	Impact position	Δt ($\mu\text{sec.}$)	Weve velocity (m/sec.)
No air bubble	upper	60.8 (3.44 ¹)	1646
	side	57.4 (0.84)	1733
Air bubble	upper	684 (1.53)	1472
	side	66.3 (2.49)	1509

¹: Standard Deviation

Table 1 shows the wave velocity of epoxy resin specimen with and without air bubbles. Wave velocity was calculated by the measurement of the arriving time difference. The difference in arriving time was measured by pencil-lead-break method. Wave velocity of air bubble specimen was lower than that of no air bubble specimen, because when an interfacial damage signal was propagated, air bubble hindered and scattered the propagation of signal. The velocity of wave propagation for the specimen inside damage is faster than that on a surface, because of difference in the wave mode.

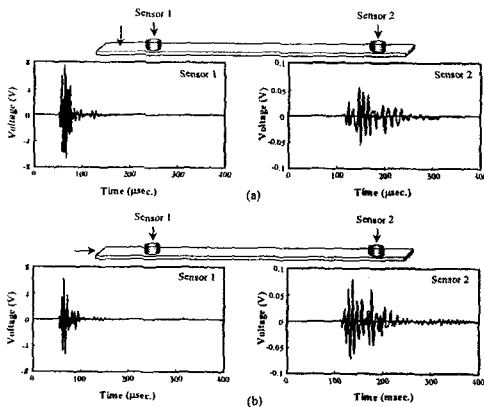


Fig. 3 Waveforms of epoxy resin without air bubble: (a) upper impact and (b) side impact

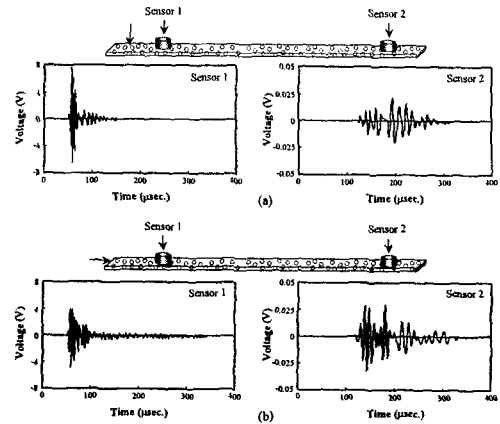


Fig. 4 Waveforms of epoxy resin with air bubble: (a) upper impact and (b) side impact

Figures 3 and 4 show waveforms of epoxy resin with various damage and impact conditions. In the specimen with air bubbles, the amplitude of waveform was smaller. In case of side impact, an extensional mode has high voltage and long time than those of upper impact.

3. 2. Sensitivities of PZT sensor and PVDF sensor

Figure 5 shows the damage signals measured by PZT sensor and PVDF sensor. The PZT sensor detected the signals of fiber fracture, matrix crack and interfacial debonding. These signals were distributed into mainly two groups, i.e., the first group of fiber fracture signal and the second group of matrix crack with interfacial debonding. However, PVDF sensor only detected the fiber fracture signal. Horizontal PVDF sensor did not detected after 300 μsec (8% strain), because a contact point between PVDF film and copper line was disconnected.

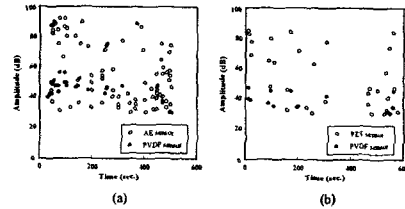


Fig. 5 Sensitivity of PZT sensor and PVDF sensor; (a) horizontal and (b) vertical

Figures 6 and 7 show waveforms and their fast Fourier transform (FFT) of fiber fracture and matrix crack signals measured by PZT sensor and PVDF sensor. The wave voltage of fiber fracture measured by PVDF sensor was lower than that of PZT sensor, because PZT sensor is more sensitive than PVDF sensor. Despite the results of FFT analysis were not same. The fiber fracture signal showed high voltage waveform and high frequency ranges compared to matrix crack. This is because glass fiber is more brittle than epoxy matrix.

PVDF sensor not detected matrix crack signal.

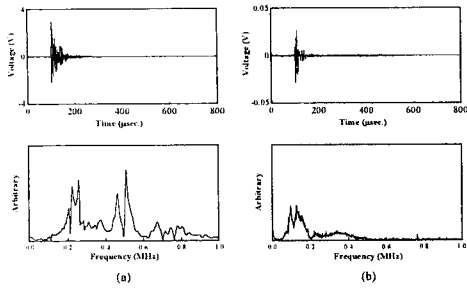


Fig. 6 Waveforms and their FFT analysis of fiber fracture signal; (a) PZT sensor and (b) PVDF sensor

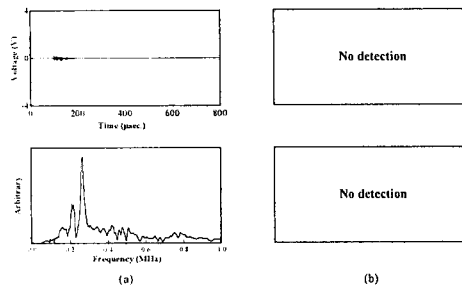


Fig. 7 Waveforms and their FFT analysis of matrix crack signal; (a) PZT sensor and (b) PVDF sensor

3.3 Failure modes and IFSS

Figure 8 shows the specimen shapes of before and after tests of PVDF embedded single glass fiber/epoxy composites. When the embedded PVDF sensor was horizontal, the specimen was cracked near neck parts as shown in Figure 8(a). Thus a contact point between PVDF film and copper line was broken and disconnected. The vertical PVDF sensor detected up to high elongation compared to the horizontal PVDF sensor.

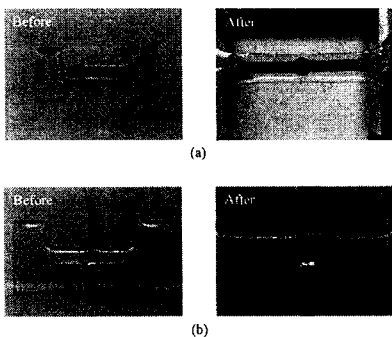


Fig. 8 Failure modes of single glass fiber/epoxy composite with embedded PVDF sensor; (a) horizontal and (b) vertical

Figure 9 shows IFSS of single glass fiber/epoxy composites measured by optical microscope, PZT sensor, and PVDF sensor. The signal numbers of fiber fracture measured by AE were rather smaller than the number of fragments measured by optical method, since some fiber

fracture signals were lost during AE detecting. IFSS measured by PVDF sensor was smaller than that of PZT sensor, because PZT sensor was much more sensitive than PVDF sensor.

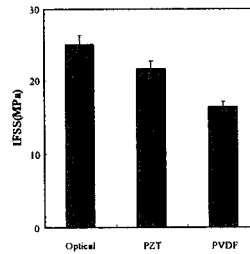


Fig. 10 IFSS measured by optical microscope, PZT sensor and PVDF sensor

4. CONCLUSIONS

The wave velocities of epoxy resin specimen with and without an air bubble as well as surface scratching damage were compared. The wave velocity of air bubble specimen was lower than that of specimen without air bubble and the velocity of wave propagation for inside damage specimen is higher than that on a surface. The PZT sensor detected the signals of fiber fracture, matrix crack and interfacial debonding, but PVDF sensor only detected the fiber fracture signal. And the vertical PVDF sensor detected up to high elongation compared to the horizontal PVDF sensor. The wave voltage of fiber fracture measured by PVDF sensor was lower than that of PZT sensor, because PZT sensor is more sensitive than PVDF sensor, although the results of FFT analysis were same. The study for more improve sensitivity of PVDF sensor will be progressed and the properties will be evaluated.

ACKNOWLEDGMENT

This work was financially supported by Korea Research Institute of Standards and Science (KRISS) through Engineering Research Institute (ERI), GNU.

REFERENCES

- (1) J. Bohse, *Compos. Sci. Tech.*, 60, 2000, p. 1213.
- (2) G. N. Morshcer and A. L. Gyekenyesi, *Compos. Sci. Tech.*, 62, 2002, p. 1171
- (3) Q. Q. Ni and E. Jinen, *Eng. Fracture Mechanics*, 56, 1997, 779
- (4) J. M., Park, J. W. Kim and D. J. Yoon, *Compos. Sci. Tech.*, 62, 2002, p. 743.
- (5) J. M., Park, J. W. Kim and D. J. Yoon, *Compos. Sci. Tech.*, 247, 2002, p. 231.
- (6) F. Teston, C. Chenu, N. Felix and M. Lethiecq, *Mater. Sci. Eng.*, 21, 2002, p. 177.
- (7) R. S. C. Monkhouse, P. D. Wilcox and P. Cawley, *Ultrasonics*, 35, 1997, p. 489.
- (8) A. J. Tuzzolino, *Adv. Space Res.*, 17, 1996, p. 123.
- (9) A. Kelly and W. R. Tyson, *Mech. And Phys. Solids*, 13, 1965, P. 329.