NASA White Sands Test Facility

Task
In-Situ NDE Characterization of Kevlar® and Carbon Composite Micromechanics for Improved COPV Health Monitoring

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Objectives
This project is a subtask of a multi-center project to advance the state-of-the-art by developing NDE techniques that are capable of evaluating stress rupture (SR) degradation in Kevlar/epoxy (K/Ep) composite overwrapped pressure vessels (COPVs), and damage progression in carbon/epoxy (C/Ep) COPVs.

In this subtask, acoustic emission (AE) data acquired during intermittent load hold tensile testing of K/Ep and C/Ep composite tow materials-of-construction used in COPV fabrication were analyzed to monitor progressive damage during the approach to tensile failure. Insight into the progressive damage of composite tow was gained by monitoring AE event rate, energy, source location, and frequency. Source location based on arrival time data was used to discern between significant AE attributable to microstructural damage and spurious AE attributable to background and grip noise. One of the significant findings was the observation of increasing violation of the Kaiser effect (Felicity ratio < 1.0) with damage accumulation.

Approach
Programmed tensile stress schedules were applied using an Instron® 5569 Series Electromechanical Test Instrument equipped with a 50 kN (11,200 lb_f) capacity load cell. To minimize excessive AE during loading and unloading ramps, a 20 N/min (4.5 lb_f/min) loading/unloading rate was used, consistent with ASTM E 1118 [**1**] recommendations. All K/Ep and C/Ep specimens had a gage length of approximately 25 cm (10 in.). AE measurements were taken using a DWC FM-1 (Digital Wave Corp., Centennial, CO) system equipped with broadband, high fidelity B1080 piezoelectric sensor with a frequency range of 1 kHz to 1.5 MHz. Sensor sensitivity was checked using pencil lead breaks performed midway between adjacent sensors, according to guidelines described in ASTM E 976 [**2**].

Materials—Unidirectional 4560 denier Kevlar 49 composite strands (date of manufacture: ca. 1987, received from Texas Research Institute [TRI] Austin, TX) had a nominal ultimate tensile strength (UTS) of 1312 ± 67 N (295 ± 15 lb_f). One denier = 1 g/9000 m of fiber. Each K/Ep tensile specimen was prepared per ASTM D 2343 [**3**] and had an elliptical cross-sectional area of 0.347 mm^2 (0.000544 in^2). Specimens were mounted vertically with sensors positioned approximately 5 cm (2 in.) from each other and the cardboard tabs (Fig. 1). Unidirectional 3817 denier Torayca® T1000G and 3775 denier HexTow™ IM7 composite strands were received from Toray (Santa Ana, CA) and Hexcel (Stamford, CT), respectively, and had UTS values of 1550 ± 67 N (348 ± 15 lb_f) and 1396 ± 67 N (314 ± 15 lb_f), respectively. Each C/Ep tensile specimen had a ribbon-like cross-section, and the same tabbing as described above.

Load Schedule—The load (stress) schedule used was based on the pressure tank examination procedure described in ASTM E 1067 [**4**], similarly referred to as the manufacturer's qualification test in ASTM E 1118 [1], and is referred to in this report as the intermittent load-hold (ILH) stress schedule. The load sequence began with an initial hold period between 10 and 30 min to determine the level of spurious AE attributable to background noise as the specimen was held in an unloaded state, after which the specimen was intermittently loaded, held, unloaded, held, and re-loaded, until failure was reached (Table 1, also see Figure 3).

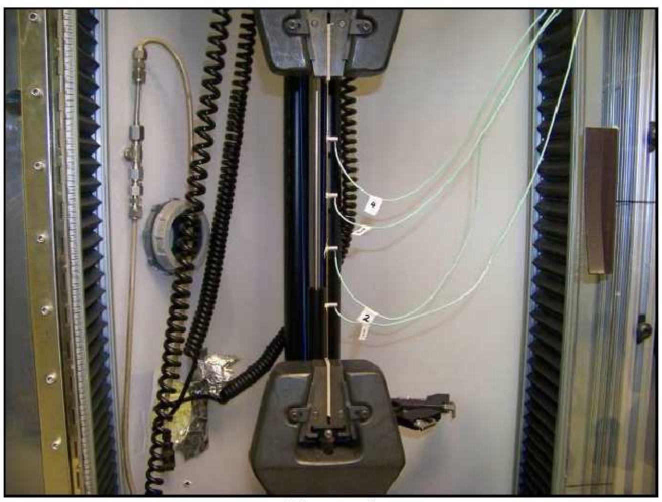

Figure 1
4560 denier K/Ep tow (25 cm gauge length) aligned in grips showing four B1080 AE sensors mounted.

Table 1
Description of Intermittent Load-Hold Stress Schedule

1	Ramp: Load to 530 N (120 lb_f)
2	10-min load hold
3	Ramp: Unload 90 N (20 lb_f)
4	10-min hold
5	Ramp 220 N (50 lb_f) to next highest load
6	Repeat Steps 2-5 until UTS is reached

Accomplishments

Discussion—One of the established tenets of AE states that if a material is loaded, unloaded, and then reloaded, new AE activity will not occur until the highest load previously experienced by the material is exceeded. This phenomenon is known as the Kaiser effect, and is observed in materials that behave elastically during reloading, (i.e., have undergone negligible plastic or permanent deformation [viscous loss processes] during previous loadings). However, once damage begins to accumulate in fiber reinforced polymer (FRP) materials, the Kaiser effect begins to be violated and new AE activity will occur in subsequent loadings (or COPV pressurizations) before the highest previous load (or pressure) is reached. The analytical parameter that describes departure from the Kaiser effect is known as Felicity ratio (FR) which is given by:

$$\text{FR} = \text{onset stress of significant AE upon re-loading} / \text{maximum previous stress} \quad (1)$$

When FR $\geq$ 1.0, the Kaiser effect is followed, while for FR $<$ 1.0, the Kaiser effect is violated. Also, the larger the departure of the FR at a value less than unity, the more pronounced the accumulated damage. Damage accumulation trends as revealed by FR data have been well documented in AE studies on concrete [**5**], and are a centerpiece of ASTM standards used for AE qualification of FRP materials. The ILH method used in this study is but one example of a stress schedule that can be used to detect violation of the Kaiser effect.

Acoustic Emission Data Reduction—Similar data filtering methods were used for K/Ep and C/Ep specimens. First, background noise checks were performed before each programmed ILH stress schedule to determine the level of spurious AE. Spurious AE was characterized by lower energy (< ca. 0.22 V^2-μs for C/Ep specimens), a flat Fast Fourier Transform (FFT) (no peaks), and an indeterminate source location (zone location = -1). Once spurious AE was removed, AE indicative of grip noise were identified via source location, and also removed. FFTs revealed that grip noise peak frequencies ranged from 150 to 190 kHz and, therefore, were clearly distinguishable from spurious AE (flat FFTs). Additional details about removal of spurious AE and grip noise is described elsewhere [**6, 7**].

The remaining source-locatable AE (Fig. 2) were found to occur at loads above 890 N (200 lb_f) in K/Ep specimens, and above 445 N (100 lb_f) in C/Ep specimens (Fig. 3). Only AE events with energies that exceeded the low energy threshold (> ca. 0.22 V^2-μs for C/Ep) were examined using source location. All events that were determined to originate outside gauge length (grip noise) were removed from the data set, while all events that were determined to originate within the gage length were retained. Source location maps were found to be useful to determining to probable locus of failure within the gage region (Fig. 4).

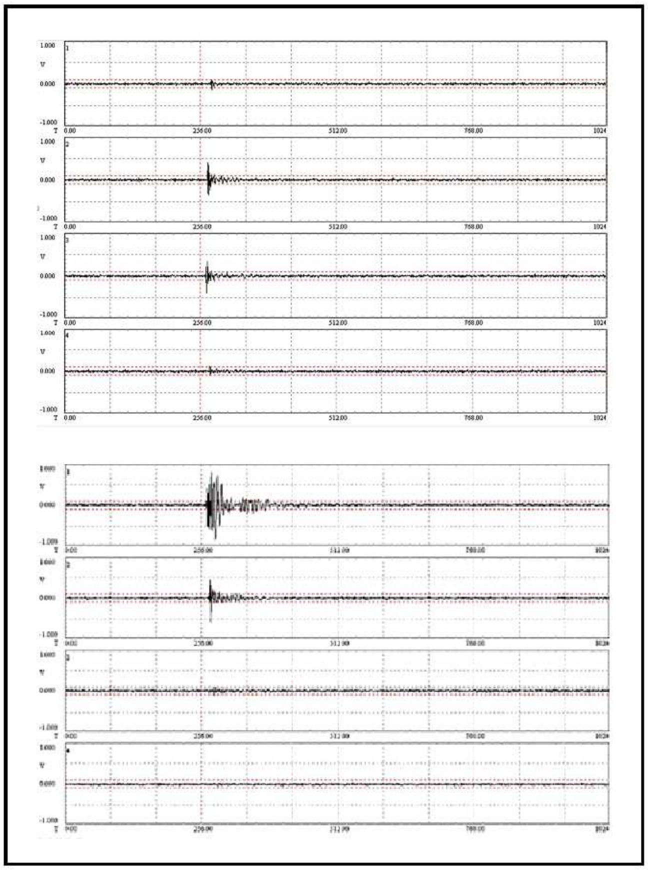

Figure 2

Significant AE occurring in the gage region for a K/Ep tensile specimen subjected to an intermittent load hold stress schedule close to 890 N (200 lb_f) where significant AE was first observed (top), and a more energetic event close to one of the grips at a later time (bottom)

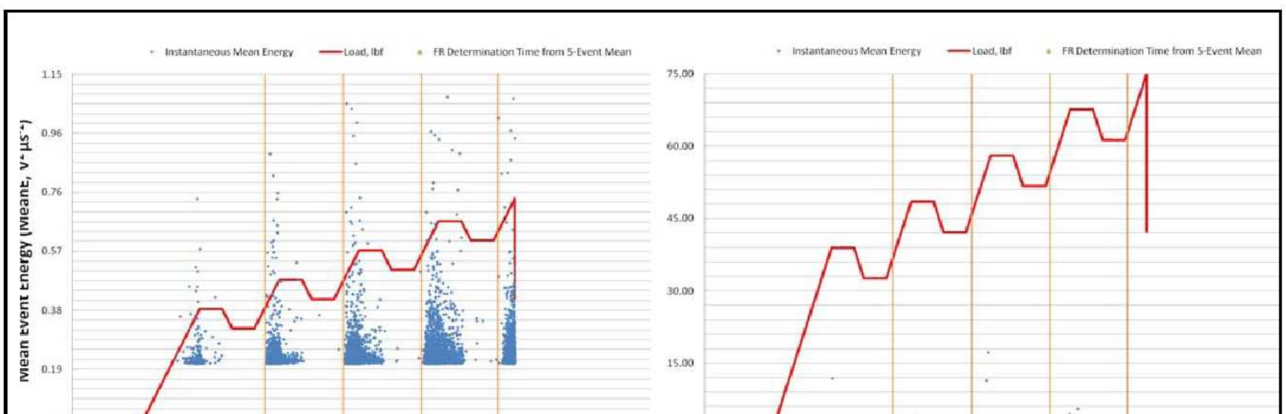

Figure 3

Representative AE data (blue data) acquired on an IM7 12k tow specimen subjected to an intermittent load hold stress schedule (red line), showing low energy events (left) and all events (right, expanded view). The vertical orange lines indicate the onset of significant AE upon loading used to calculate the Felicity ratio.

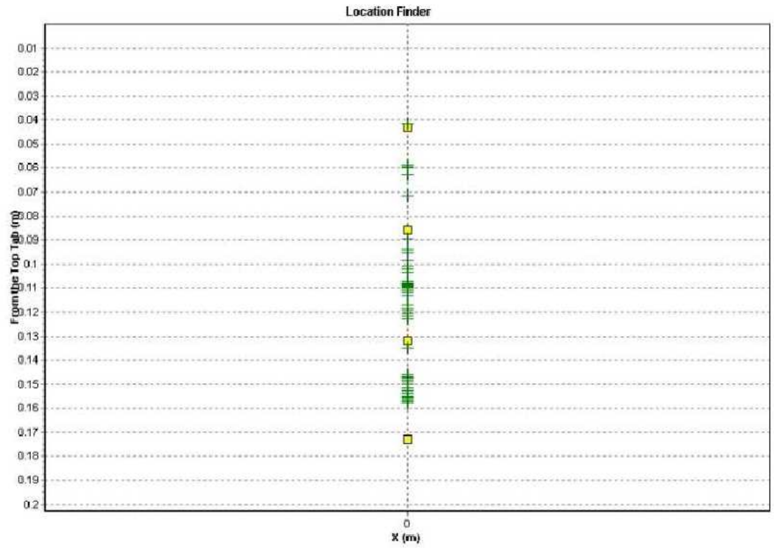

Figure 4

AE source (green crosses) location map for a T1000 12k tow specimen with a 0.203-m (8-in.) gage length, also showing the sensor locations (yellow squares)

Felicity Ratio Interpretation—The most significant finding of the present investigation was the linear decrease in the FR with increasing stress during the ILH method (Fig. 5). Taking FR = 1 as the threshold at which severe accumulated damage begin to occur, mean damage thresholds of 1059 ± 9 N (237 ± 2 lb_f) were observed for K/Ep (n = 3), 681 ± 80 N (153 ± 18 lb_f) for IM7 (n = 3), and 974 ± 89 N (219 ± 20 lb_f) for T1000 (n = 6). The standard deviations show that the C/Ep tensile and AE data were much more scattered than the K/Ep data. Also, C/Ep specimens were found to fail at comparable FRs: IM7 specimen failed at FR = 0.955 ± 0.014, T1000 specimens failed at FR = 0.967 ± 0.013. K/Ep specimens failed at a substantially lower FR = 0.872 ± 0.025. Last, K/Ep specimens were much 'quieter' than C/Ep specimens, and exhibited higher onsets of significant AE, and flatter FR versus load responses (Fig. 6).

Examination of AE energies also gave a similar indication of accumulated damage (Fig. 7). At the FR = 1.0 threshold, quite energetic gage events in excess of 10 V^2-μs began to be observed. Examination of the FFTs of these high energy events revealed the simultaneous occurrence of matrix cracking, fiber-matrix debonding, fiber pull-out, and fiber breakage (Fig. 8). Efforts are underway to determine the frequency evolution of progressive damage accumulation for all AE events. Using the formalism of de Groot [**8**], preliminary data [**7**] indicate that the amount of matrix cracking, fiber-matrix debonding, and fiber pull-out decrease during progressive damage accumulation, while the amount of fiber breakage increases during progressive damage accumulation.

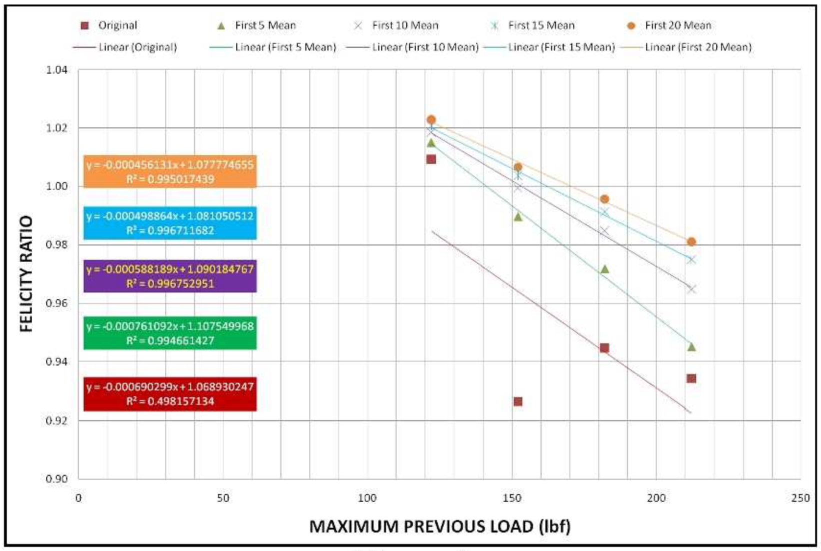

Figure 5

Linear decrease in the Felicity ratio with increasing load for an IM7 12k tow subjected to an intermittent load hold stress schedule. The different least squares fits correspond to different averaging techniques for determining the onset of significant AE upon loading.

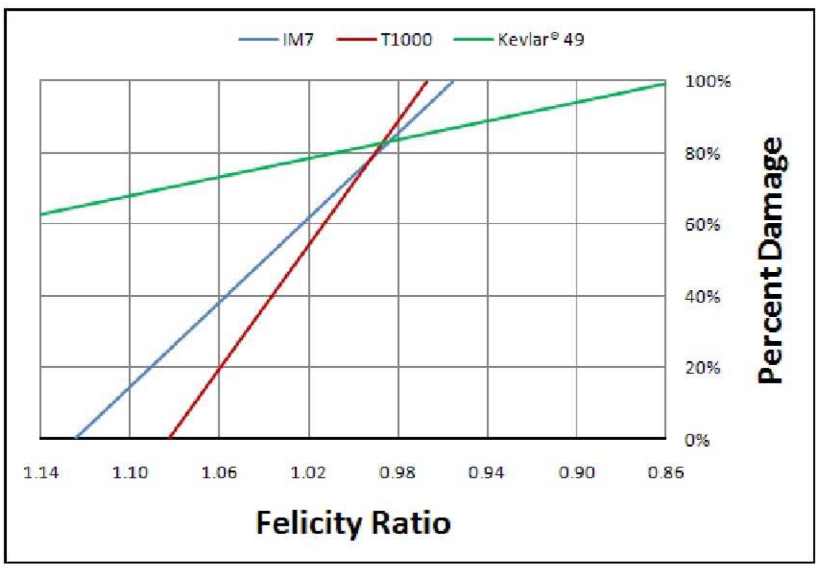

Figure 6

Averaged linear damage accumulation plots for IM7 C/Ep, T1000 C/Ep, and K/Ep composite tow the Felicity ratio (FR) at 0 % damage corresponds to the extrapolated FR for an unloaded specimen, whereas the FR at 100 % damage corresponds to the last measured FR prior to rupture

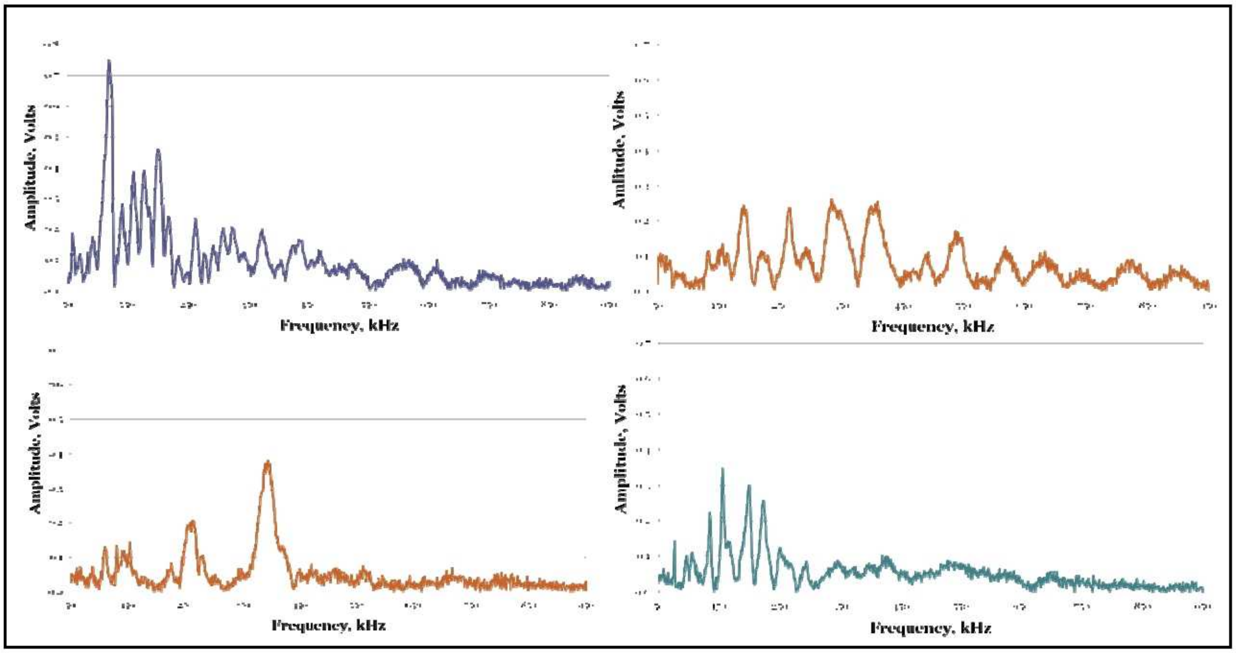

Figure 7
Fast Fourier transform frequency distributions observed for representative high energy events for a T1000 12k tow specimen subjective to an intermittent load hold stress schedule, showing presumed matrix cracking and pull-out (upper left), mixed mode failure (upper right), fiber breakage and debonding (lower left), and debonding and pull-out (lower right)

Summary

The Felicity ratio was found to give a reproducible estimate of the stress threshold at which significant accumulated damage began to occur. Further refinement of stress schedules for determining Felicity ratios could lead to robust pass-fail acceptance criteria once the type and amount of the accumulated damage is better understood. This, in turn, will entail determining what precursor events are operative and when, and how much of a given precursor event can be tolerated.

Customers

The direct customers for this task will be the Space Transportation System, International Space Station, CEV/Constellation, in-Space Prolusion/ Planetary programs, and others related to NASA's Space Exploration Initiatives. Indirect customers include those managing other space flight systems employing COPV technology. The results developed could be used across the agency, Department of Defense, and in commercial aerospace applications.

Metrics

Progress toward developing methods that show promise in understanding the micromechanical changes associated with severe damage accumulation in COPV composite overwrap materials of construction shall be reported on an annual basis and evaluated by the NNWG. The final report shall report overall project progress and make recommendations for further efforts.

Products
A formal written report documenting progress made towards the use of modal AE and acousto-optics (AO) to characterize rupture of C/Ep tow specimens shall be provided at the end of 2010. A formal publication given at the annual QNDE (or similar) conference is also anticipated. It is hoped that the work will result in quantitative accept-reject criteria for composite overwraps based on Felicity ratio, underpinned by knowledge of the underlying events associated with damage derived from FFT data. More specifically, a method will be developed that allows the cumulative amount of matrix cracking, fiber-matrix debonding, fiber pull-out, and fiber breakage to be estimated at fixed FRs preceding rupture.

Status
Summary of work accomplished through Q4 FY09: Testing of 4560 denier K/Ep tow, and 12k IM7 and T1000 C/Ep tows has been completed. The efficacy and reproducibility of FR methods for determining the onset of severe accumulated damage have been assessed. A paper detailed finding for K/Ep was given at the 2009 QNDE conference. AO sensors were attached to T1000 fibers, and the preliminary feasibility of the AO method to corroborate AE measurements was assessed.

Q1-2 FY10 Schedule

- Complete FFT analysis on all C/Ep and K/Ep data sets to determine what type of damage is occurring at and below FR = 1.
- Provide an annual status presentation and report for the NNWG.
- Apply a proof cycle or ILH pressurization schedule to a carbon fiber reinforced COPV and determine the change in FR as the ultimate burst pressure is approached.
- USRP intern mentoring.
- In collaboration with MSFC, perform follow-on proof-of-concept tests to assess the efficacy and feasibility of the AO method.

Q3-4 FY10 Schedule

- Correlate damage progression in C/Ep materials of construction with actual microscopic damage using SEM at given level of damage approaching catastrophic failure.
- Determine the feasibility of developing a consensus standard for in-service accept-reject of C/Ep materials of construction and/or C/Ep COPVs based on accumulation of severe accumulated damage and corresponding violation of the Kaiser effect.
- USRP intern mentoring.
- Issue a FY10 Final Report.

References

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3. ASTM D 2343, *Test Method for Tensile Properties of Glass Fiber Strands, Yarns, and Rovings Used in Reinforced Plastics,* American Society for Testing and Materials, West Conshohocken, PA (2008).
4. ASTM E 1067, *Standard Practice for Acoustic Emission Examination of Fiberglass Reinforced Plastic Resin (FRP) Tanks/Vessels*, American Society for Testing and Materials, West Conshohocken, PA (2007).
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6. Waller, J. M, R. L. Saulsberry, and E. Andrade, *Use of Acoustic Emission to Monitor Progressive Damage Accumulation in Kevlar® 49 Composites*, QNDE Conference, Providence, RI, July 2009.
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8. De Groot, P., P. Wijnen, and R. Janssen, "Real-time Frequency Determination of Acoustic Emission for Different Fracture Mechanisms in Carbon/Epoxy Composites," *Composites Sci. Technol.*, 55, pp. 405-421 (1995).