Use of Modal Acoustic Emission to Monitor Damage Progression in Carbon Fiber/Epoxy Tows and Implications for Composite Structures

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Background

**Issues:**

- COPVs can be at risk for catastrophic failure
  - Risk of insidious burst-before-leak (BBL) stress rupture\(^1\) (SR) failure of carbon-epoxy (C/Ep) COPVs during mid to late life
  - Risk of lower burst strength of C/Ep COPVs subjected to impact damage
- Issues with manufacturing defects and inspectability of COPVs on NASA spacecraft (ISS, deep space)
- Lack of quantitative NDE is causing problems in current and future spacecraft applications
  - Must increase safety factor or accept more risk
  - Thinner liners are driving need for better flaw detection in liner and overwrap

\(^1\) SR defined by AIAA Aerospace Pressure Vessels Standards Working Group as “the minimum time during which the composite maintains structural integrity considering the combined effects of stress level(s), time at stress level(s), and associated environment”
The problem with advanced fibers such as Kevlar® and carbon, is that no ductility is observed before rupture during tertiary creep, so the stress rupture occurs with little or no advance warning.
Effect of Fiber Choice on Stress Rupture

C/Ep COPVs are susceptible to stress rupture, although to a lesser extent than glass or Kevlar® fiber composites

Characteristic lifetimes of graphite, Kevlar® and glass-reinforced composites at different percentages of the ultimate strength. Each symbol represents the median life (50%) under sustained loads as percentage of the ultimate strength of the material §

COPVs on ISS

- Presently have 17 high pressure COPVs on ISS (most are C/Ep)
  - Up to seven additional COPVs are planned and under development
- Long term reliability risk levels are $10^{-6}$ or lower except for NTA and SpaceDRUMS COPVs, which have risk levels of $10^{-4}$ to $10^{-5}$ *
  - Reliability much lower if C/Ep overwrap sustains impact damage

### COPVs on ISS Table

<table>
<thead>
<tr>
<th>Subsystem</th>
<th>No.</th>
<th>Shape</th>
<th>Size, in.</th>
<th>Commodity</th>
<th>Materials</th>
<th>Supplier</th>
<th>FOS</th>
<th>MEOP psi</th>
</tr>
</thead>
<tbody>
<tr>
<td>ECLSS/ACS/HPGT</td>
<td>4</td>
<td>Sphere</td>
<td>8.39</td>
<td>Oxygen, Nitrogen</td>
<td>301 SS</td>
<td>GD</td>
<td>2.0</td>
<td>5000</td>
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<tr>
<td>ECLSS/MCA</td>
<td>1</td>
<td>Cylinder</td>
<td>7.22 L x 3.55 D</td>
<td>Calibrated air</td>
<td>Al</td>
<td>SCI</td>
<td>3.4</td>
<td>3000</td>
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<tr>
<td>TCS/NTA</td>
<td>2</td>
<td>Cylinder</td>
<td>45 L x 19.7 D</td>
<td>Nitrogen</td>
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<td>GD</td>
<td>2.52</td>
<td>3000</td>
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<tr>
<td>EVA/SAFER</td>
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<td>Cylinder</td>
<td>9 L x 6 D</td>
<td>Nitrogen</td>
<td>SS</td>
<td>ARDÉ</td>
<td>3.0</td>
<td>10,000</td>
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<tr>
<td>Environment/PCU</td>
<td>2</td>
<td>Sphere</td>
<td>15.37</td>
<td>Xenon</td>
<td>301 SS</td>
<td>T-1000</td>
<td>4.17</td>
<td>3000</td>
</tr>
<tr>
<td>Payloads/SpaceDRUMS</td>
<td>5</td>
<td>Cylinder</td>
<td>17.1 L x 8.5 D</td>
<td>Argon</td>
<td>Al</td>
<td>T-1000</td>
<td>2.28</td>
<td>2350</td>
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<tr>
<td>Payloads/VCAM*</td>
<td>1</td>
<td>Cylinder</td>
<td>8.1 L x 3.68 D</td>
<td>Helium</td>
<td>Al</td>
<td>Gr/ep-2150</td>
<td>3.4</td>
<td>1985</td>
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<tr>
<td>AMS-01*</td>
<td>2</td>
<td>Sphere</td>
<td>12.4; 15.8</td>
<td>Carbon dioxide, Xenon</td>
<td>301 SS</td>
<td>T-1000</td>
<td>3.05.4.4</td>
<td>1440-2000</td>
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<td>ECLSS/TCS/NORS**</td>
<td>0</td>
<td>TBD</td>
<td>TBD</td>
<td>TBD</td>
<td>TBD</td>
<td>TBD</td>
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<td>TBD</td>
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<tr>
<td>CIPAA***</td>
<td>4</td>
<td>Cylinder</td>
<td>4.04 D x 9.6 L</td>
<td>Carbon dioxide</td>
<td>Al</td>
<td>Gr/E-Glass</td>
<td>4.67</td>
<td>4500</td>
</tr>
</tbody>
</table>

*The VCAM and AMS systems have not been manifested.

**The NORS system is still under development.

***The CIPAA system is transported to and from the ISS with each Shuttle mission. The very high FOS indicates a very low risk of rupture.

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Goals

- Develop quantitative AE procedures specific to C/Ep overwraps, but which also have utility for general monitoring of damage accumulation in composites
- Lay groundwork for establishing critical thresholds for accumulated damage in composite components, such as COPVs, so that precautionary or preemptive engineering steps can be implemented to minimize or obviate the risk of catastrophic failure
  - Felicity ratio ($FR$), coupled with fast Fourier transform (FFT) frequency analysis shows promise as an analytical pass/fail criterion
    - Would identify COPVs at a critical $FR$, or $FR^*$, below 1.0, indicative of severe accumulated damage
    - Could also identify COPVs at a hazardous level of cumulative fiber breakage or matrix cracking
Load control and AE data acquisition system (DACS) consisted of:

- Instron® 5569 Series Electromechanical Test Instrument (left)
- DigitalWave Corp. FM-1 8-channel DACS (lower right)
- AE and tensile test CPU controllers (upper right)
**Experimental**

**AE Sensors**: Each channel (4 used) was connected to a DWC PA-0, 0 dB Gain preamplifier, and then to a broadband high fidelity B1080 piezoelectric sensor with a frequency range 1 kHz to 1.5 MHz. Sensors were mounted on cardboard-tabbed C/Ep tow specimens (8-in. gage length) using Lord Corp. AE-10 acrylic adhesive.
Experimental

Tabbing: shear strength of epoxy and bonded grip length important variables

\[ L_{\text{min}} = \frac{F_{\text{su}} h}{2 F_{\text{st}}} \]

where:
- \( L_{\text{min}} \) = minimum required bonded tab length, mm [in.];
- \( F_{\text{su}} \) = ultimate tensile strength of coupon material, MPa [psi];
- \( h \) = coupon thickness, mm [in.]; and
- \( F_{\text{st}} \) = ultimate shear strength of adhesive, coupon material, or tab material (whichever is lowest), MPa [psi].

Felicity Ratio Analysis
Experimental

- For purposes of quick turn-around, an intermittent load hold (ILH) stress schedule was used (red data)

- ILH profile is based on the pressure tank examination procedure described in ASTM E 1067 §

Results & Discussion

- Linear decrease in FR with load noted for T1000 ($R^2 > 0.9$) and IM-7 ($R^2 > 0.99$) C/Ep, similar to the behavior noted for Kevlar 49-epoxy K/Ep

- For same material and averaging method, the slope of least squares fit is indicative of damage tolerance
  - Flatter slopes correspond to good damage tolerance (in-character behavior)
  - Steep slopes correspond to low damage tolerance (out-of-character behavior)

- Kaiser effect violated at FR<1 ⇒ onset of severe accumulated damage

- C/Ep produced more AE than K/Ep (but AE less energetic on average)
Results & Discussion

• Formation of characteristic damage state very evident at Load Ratio (LR) < 0.6 for IM-7

• In quasi-isotropic composite lay-ups, for example, characteristic damage state formation thought to involve predominant matrix cracking

• For uniaxial tow, FFTs revealed the characteristic damage state formation involves a mixed mode failure mechanism (cooperative matrix cracking, fiber/matrix debonding, fiber pull-out, fiber breakage)
### Summary of FR Results for Carbon/Epoxy

<table>
<thead>
<tr>
<th>Date</th>
<th>Material &amp; Spool #</th>
<th>Filter¹</th>
<th>$F_{@FR=1}$ (lbf)</th>
<th>$F_{\text{max}}$ (lbf)</th>
<th>$\sigma_{@FR=1}$ (ksi)</th>
<th>$\sigma_{\text{max}}$ (ksi)</th>
<th>$FR^*$</th>
<th>Failure Type²</th>
</tr>
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<tr>
<td>83109</td>
<td>IM7 #95</td>
<td>32%</td>
<td>135</td>
<td>210</td>
<td>342</td>
<td>532</td>
<td>0.95</td>
<td>XGB</td>
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<td>90109</td>
<td>IM7 #95</td>
<td>27%</td>
<td>151</td>
<td>234</td>
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<td>591</td>
<td>0.945</td>
<td>XGM</td>
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<tr>
<td>90809</td>
<td>IM7 #95</td>
<td>58%</td>
<td>171</td>
<td>210</td>
<td>433</td>
<td>530</td>
<td>0.971</td>
<td>XGM</td>
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<tr>
<td>111009</td>
<td>IM7 #117</td>
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<td>252</td>
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<tr>
<td>32610</td>
<td>IM7 #61</td>
<td>19%</td>
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<td>228</td>
<td>464</td>
<td>578</td>
<td>0.97</td>
<td>XGM</td>
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<tr>
<td>82509</td>
<td>T1000 #74</td>
<td>32%</td>
<td>240</td>
<td>355</td>
<td>658</td>
<td>972</td>
<td>0.972</td>
<td>XGT</td>
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<td>T1000 #74</td>
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<td>231</td>
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<td>633</td>
<td>1010</td>
<td>0.953</td>
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<td>992</td>
<td>977</td>
<td>0.977</td>
<td>XGT</td>
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<tr>
<td>112509</td>
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<td>325</td>
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<td>T1000 #155</td>
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<td>181</td>
<td>374</td>
<td>493</td>
<td>1024</td>
<td>0.95</td>
<td>XGM</td>
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<tr>
<td>Mean</td>
<td>IM7</td>
<td>29%</td>
<td>167</td>
<td>227</td>
<td>422</td>
<td>575</td>
<td>0.959</td>
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<td>Std. Dev.</td>
<td>T1000</td>
<td>18%</td>
<td>24</td>
<td>18</td>
<td>60</td>
<td>45</td>
<td>0.012</td>
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</tr>
<tr>
<td>Mean</td>
<td>T1000</td>
<td>22%</td>
<td>211</td>
<td>361</td>
<td>577</td>
<td>988</td>
<td>0.961</td>
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<tr>
<td>Std. Dev.</td>
<td>T1000</td>
<td>18%</td>
<td>26</td>
<td>19</td>
<td>71</td>
<td>53</td>
<td>0.013</td>
<td></td>
</tr>
</tbody>
</table>

1. Data filter reflects percentage of events removed from the raw AE data
3. Improved tabbing method

- Let $FR^*$ = extrapolated $FR$ at rupture predicted by the least squares fit
- $FR^*$ behaves like a universal parameter that varies less than the UTS
Results & Discussion

- Consistent $FR^*$ values noted for T1000 and IM-7
- Suggests that the FR can be used as an analytical PASS/FAIL criterion for C/Ep composite materials
- Precedent: ASTM suggests using $FR < 0.95$ as failure criteria in fiberglass reinforced pressure vessels
  - Experimental C/Ep failure criteria from strand tests
    » IM7: $FR < 0.959$
    » T1000: $FR < 0.961$
  - Also can use counts and number of hits above high energy threshold
- Opens up possibility that C/Ep composite materials can be subjected to ILH profiles to assess in- or out-of-family response
  - Need to verify that test specimens or articles with low initial $FR$, or steep ‘$FR$ vs. load’ slopes in fact fail prematurely, or in the case in COPVs, fail at lower burst pressure

Waveform and FFT Analysis
Results & Discussion

• AE frequency ranges have been correlated with micromechanical damage mechanisms in C/Ep§


Results & Discussion

FFT (unfiltered) showing concerted failure using De Groot’s frequency ranges

FFT FREQUENCY DISTRIBUTION

T1000 Spool 74 tested 9/9/09, Y=14.8 cm (2/5 from S3 to S4)
N=2597, E=3.39 V²-µs, FAC-4

- fiber breakage
- matrix cracking
- pull-out
- debonding
Results & Discussion

• In general, three different waveforms were observed for C/Ep

1. Matrix Cracking
2. Fiber Breakage

waveform  FFT  waveform  FFT
160 kHz  575 kHz
Results & Discussion

• Three different waveforms were observed for C/Ep (cont.)

3. Concerted, mixed mode failure
Results & Discussion

• IM-7 early vs. late life events

- Early Life
  - LR > 0.8
  - last ILH ramp up

- Late Life
  - LR < 0.5
  - first ILH ramp up

• Notice change from ordered (early) to unordered peaks (late life)
Results & Discussion

- IM-7 low vs. high energy events

Low Energy

High Energy

E < 2 V^2-\mu sec

E > 2 V^2-\mu sec

- Low energy events have similar damage ‘footprint’ (top), while high energy events have a more variable damage ‘footprint’ (bottom)
- Similar observation of a fiber breakage dominated ‘footprint’ for FR events
Results & Discussion

- High frequency peaks shifted downward with increasing load ratio:
  \[731 \text{ kHz} \Rightarrow 728 \text{ kHz} \Rightarrow 685 \text{ kHz} \Rightarrow 640 \text{ kHz}\]
- Attributed to increasing accumulated damage, hence lower modulus, causing slower stress wave propagation
Results & Discussion

• The FFTs of IM-7 and T1000 Felicity ratio events (first ten events) were then compared to see if they had a characteristic damage mode, or if the damage mode changed with load.

• Fiber breakage dominates FR events
  – otherwise FR events involve concerted failure for both types of C/Ep

• Some differences, but same overall trend noted for T1000 & IM-7:
  300-1000 kHz > 90-190 kHz > 190-300 kHz
  *(fiber breakage > matrix cracking > debonding/pull-out)*
Results & Discussion

Source location of FR events show they occur at or near locus of failure

- IM7_032610 specimen had intact tow between and 0 (lower tab) and 0.115 m 0.17 and 0.20 m (upper tab)
- Tow region between 0.115 and 0.17 m obliterated (explosive failure)
- Most FR events were source located in the missing region that failed explosively
Application to
Composite Overwrapped Pressure Vessels (COPVs)
Results & Discussion

A 6.3-in. diameter IM-7 COPV was subjected to an ILH pressure schedule at $LR \approx 0.3$ to $0.9$.
Results & Discussion

IM-7 COPV data (♦ symbols) overlap IM-7 tow data (open green symbols)

Least squares fits (solid lines) and 99 % confidence intervals (dash-dot-dot lines) also shown for T1000 and Kevlar® 49
COPV Felicity Ratio Results

First 8 Data Points:
- \( FR^* = 1.000 \pm 0.010 \)
- \( P^*_{projected} = 7,540 \pm 75 \) psi
- \( P^*_{observed} = 7,529 \) psi based on hydroburst data on 2 identical COPVs

All Data:
- \( FR^* = 0.961 \pm 0.018 \)
- \( P^*_{projected} = 7,870 \pm 144 \) psi
- \( P^*_{observed} = 7,869 \) psi higher than expected based on hydroburst data

- First 8 data points predict a burst in agreement w/ burst tests result on 2 other identical COPVs (\( P^* = 7529 \) psi expected)
  - However, high initial \( FR^* = 1.00 (>0.96) \) suggests COPV may burst higher @ >7529 psi
  - Actual burst was 7870 psi
COPV AE Waveforms

Raw Waveform

Direct Wave

Background Noise

Raw Waveform w/ reflections

Effect of filtering reflections on frequency distributions

Potential (V) vs. Frequency (kHz)
Results & Discussion

Frequency distribution of IM-7 COPV FR events

- ILH load profile
- based on direct wave only, reflections removed
- In-house regression method used (later load ramp based on more AE events)
- 6-sensor IM-7 COPV (SN070908-02 from 1/6/2010)
- Frequencies below 90 kHz ignored
- Based on de Groot’s frequency ranges
  - Fiber breakage and matrix cracking are predominant failure modes
Conclusions

- ASTM-based ILH methods were found to give a reproducible, quantitative estimate of the stress threshold at which significant accumulated damage began to occur.
  - FR events are low energy (<2 V²μs)
  - FR events occur close to the observed failure locus
  - FR events consist of more than 30% fiber breakage (>300 kHz)
  - FR events show a consistent hierarchy of cooperative damage for composite tow, and for the COPV tested, regardless of applied load
- Application of ILH or related stress profiles could lead to robust pass/fail acceptance criteria based on the FR
- Initial application of FR and FFT analysis of AE data acquired on COPVs is promising
Acknowledgments

Shawn Arnette (TRI, Austin, TX)
Supplying K/Ep test specimens & tabbing suggestions

S. Leigh Phoenix (Cornell University, Ithaca, NY)
Supplying C/Ep test specimens & tabbing suggestions

Paul Spencer, Brooks Wolle and Ben Gonzalez (WSTF-JSC)
Universal tensile tester set-up & tabbing

Ralph Lucero and Anthony Carden (WSTF-JSC)
COPV-level tests and AE data acquisition

Office of Safety and Mission Assurance (NASA, Washington, DC)
Support to develop AE methods specific to K/Ep and C/Ep
(NDE of composite micromechanics)
Back-up Slides
Background

Actions Needed:

• Develop and demonstrate critical NDE which can be implemented during:
  a) process design & optimization
  b) on-line process control
  c) after manufacture inspection
  d) in-service inspection
  e) health monitoring

• Need expressed by JPL, WSTF, Orion, NESC Composite Pressure Vessel Working Group (CPVWG), and others
Acoustic Emission Testing

Acoustic Emission refers to the generation of transient elastic waves produced by a sudden redistribution of stress in a material. When a structure is subjected to an external stimulus (change in pressure, load, or temperature), localized sources trigger the release of energy, in the form of stress waves, which propagate to the surface and are recorded by sensors.

(http://www.ndt-ed.org/)

- 100 mV
Felicity ratio (FR)

\[
FR = \frac{\text{stress at onset of significant acoustic emission during loading}}{\text{maximum previous stress plateau}}
\]

\[
FR = \frac{121.2}{120} = 1.01
\]
WaveExplorer™ Interface
Using Digital Wave Corp. (DWC) equipment and B-1025 microdot AE sensors:

<table>
<thead>
<tr>
<th>WaveExplorer Configuration</th>
<th>FM-1 Test Configuration</th>
</tr>
</thead>
<tbody>
<tr>
<td>Setup File: COPV860</td>
<td>Line Driver Switch: +V/75Ω</td>
</tr>
<tr>
<td># of Channels: 7&lt;sup&gt;1&lt;/sup&gt;</td>
<td>Preamp Gain: 18 dB</td>
</tr>
<tr>
<td>Sampling Rate: 2 MHz</td>
<td>Signal Gain: 12 dB</td>
</tr>
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<td>Num of Points: 4096</td>
<td>Signal H.P. Filter: 20 kHz</td>
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<tr>
<td>Pretrigger Points: 512</td>
<td>Trigger Gain: 21 dB</td>
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<tr>
<td>Voltage Range: +/-1 V</td>
<td>Trigger H.P. Filter: 50 K</td>
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<tr>
<td></td>
<td>Trigger L.P. Filter: 1.5 MHz</td>
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### Parametric Setup Parameters

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<tr>
<th>Parametric Setup Parameters</th>
<th>FM-1 Lead Break Configuration&lt;sup&gt;2&lt;/sup&gt;</th>
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<td>Parametric Sampling Rate:</td>
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<td>1Hz</td>
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<td>Parametric Voltage Range:</td>
<td>Signal Gain: 12 dB</td>
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<td>+/-10 V</td>
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<tr>
<td>Total Parametric Channels:</td>
<td>Trigger Gain: 18 dB</td>
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<tr>
<td>1</td>
<td></td>
</tr>
</tbody>
</table>

<sup>1</sup> The first channel was defective, so channels 2-7 were used.

<sup>2</sup> A pre-installation sensor check was performed on a ¼” Al plate with 0.2mm lead break 1/8 in. from the sensor edge (Preamp=35, Signal=24, trigger=18).
## COPV Data Reduction Parameters

### Steps involved in filtering DWC WaveExplorer data by order of application:

<table>
<thead>
<tr>
<th>Filter</th>
<th>Data Reduction Rationale and Associated Restrictions</th>
</tr>
</thead>
</table>
| Mitigate Reflections        | Define the time centered on threshold tolerance low enough to remove most of the reflections by observing the typical flexural wave duration:  
                                  *Threshold* ≥ 100 mV (88 dB)  
                                  *TCOT* ≤ 125 µs                                                                                                               |
| Enforce Frequency Restrictions | Optional – Define the frequency range over which the data will be analyzed.  
                                  For example, de Groot (1995) has defined fiber breakage events occur within the 300-600 kHz regime, so the waveform data would be filtered to exclude frequencies outside of this range:  
                                  *Frequency* ≥ 300 kHz  
                                  *Frequency* ≤ 600 kHz                                                                                                           |
| Eliminate Background Noise  | Define the waveform threshold value to exceed the typical background noise amplitude on at least one channel (takes awhile to process):  
                                  *Must pass on at least 1 channel*  
                                  *Threshold* ≥ 100 mV (88 dB)                                                                                                     |
| Eliminate System Noise      | Remove all events from exported text files and spreadsheets that first arrive on the internal channel -1.                                                                       |
AE Data Filtering

• Significant AE determined using source location and energy
  – Source Location
    • Source location based on arrival time (picked up by at least 3 sensors)
    • Events originating outside the gage region were eliminated
      – however, events located within 0.3 mm of grip were retained
    • Default wave velocity for graphite used in all tests (4600 m/s)
      – verified using PLBs: 4356 m/s value obtained
    • Non-locatable events (picked up by 1-2 sensors) included only if they exceeded the minimum energy threshold below
  – Energy
    • Energy levels across all 4 channels were averaged for each event
      – The average energy of background events (usually < 0.22 V^2-µs) was recorded for 30 min for C/Ep specimens held under a small preload (≤ 5 lbf)
      – Events with an average energy above this value were considered significant
Results & Discussion

• Characteristics of significant AE:
  – For Kevlar-epoxy, and T1000 and IM-7 carbon-epoxy, nonlinear increases in AE event rate were observed immediately before rupture, indicative of ‘critically intense’ AE activity per ASTM E 1067 and E 1118:
  – Areas of critically intense AE activity also showed greatest violation of Kaiser effect, hence, the lowest FR values
Results & Discussion

- For Kevlar-epoxy 4650 denier tow, correlation coefficients for ILH1 & 2 methods indicated good ($R^2 = 0.866$) to excellent ($R^2 = 0.985$) agreement:
Results & Discussion

AE source location method improved

Old

New

- For 2D or 3D specimens, arrival times from at least 3 sensors are needed for accurate source location
  - for a 1D tow specimen, by splitting 4-channel *.wave file into 2-channel *.wave files, it was possible to reduce this number to 2 sensors
- Arrival times not always accurate
  - Manual correction was done
- Erroneous events were eliminated or located more accurately
- > 300 % more events were located