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

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# Background

## Problems:

- Composite overwrapped pressure vessels (COPVs) 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 (International Space Station (ISS), deep space)
- Lack of quantitative nondestructive evaluation (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 Vessel 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"*

# Background

## Actions Needed

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

COPV Manufacturers

Aerospace Primes

NASA (on ground and in-flight)

- Need expressed by Jet Propulsion Laboratory (JPL), White Sands Test Facility (WSTF), Orion, NASA Engineering and Safety Center (NESC) Composite Pressure Vessel Working Group (CPVWG), and others

# 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 nitrogen tank assembly (NTA) and SpaceDRUMS COPVs, which have risk levels of $10^{-4}$ to $10^{-5}$ §
  - Reliability <u>much</u> lower if C/Ep overwrap sustains impact damage

| Subsystem | No. | Shape | Size, in. | Commodity | Materials | | Supplier | FOS | MEOP psi |
|---|---|---|---|---|---|---|---|---|---|
| | | | | | Liner | Wrap | | | |
| ECLSS/ACS HPGT | 4 | Sphere | 37.89 | Oxygen, Nitrogen | 301 SS | IM-7W | GD | 2.0 | 5000 |
| ECLSS/MCA | 1 | Cylinder | 7.22 L x 3.55 D | Calibrated air | Al | S-Glass | SCI | 3.4 | 3000 |
| TCS/NTA | 2 | Cylinder | 45 L x 19.7 D | Nitrogen | Al | T-1000 | GD | 2.52 | 3000 |
| EVA/SAFER | 3 | Cylinder | 9 L x 6 D | Nitrogen | SS | T-1000 | ARDÉ | 3.0 | 10,000 |
| Environments/PCU | 2 | Sphere | 15.37 | Xenon | 301 SS | T-1000 | ARDÉ | 4.17 | 3000 |
| Payloads/ SpaceDRUMS | 5 | Cylinder | 17.1 L x 8.5 D | Argon | Al | T-1000 | GD | 2.28 - | 2350 |
| Payloads/ VCAM* | 1 | Cylinder | 8.1 L x 3.68 D | Helium | Al | Gr/ep-2150 | Carleton | 3.4 | 1985 |
| AMS-02* | 2 | Sphere | 12.4; 15.8 | Carbon dioxide, Xenon | 301 SS | T-1000 | ARDÉ | 3.05-4.4 | 1440-2900 |
| ECLSS&TCS/NORS** | 0 | TBD | TBD | TBD | TBD | TBD | TBD | TBD | TBD |
| CIPAA*** | 4 | Cylinder | 4.04 D x 9.6 L | Carbon dioxide | Al | Gr/E-Glass | Carleton | 4.67 | 4500 |

*The VCAM and AMS systems have not been manifested.

**The NORS system is still under development.

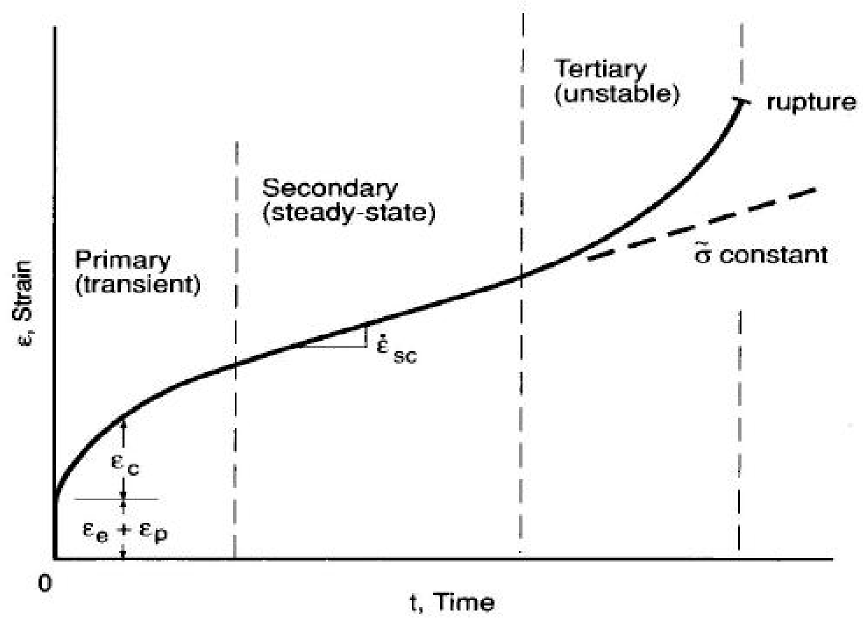
***The CIPAA system is transported to and from the ISS with each Shuttle mission. The very high FOS indicates a very low risk of rupture.



§ E. Y. Robinson, R. Kohli, "*Preliminary Stress Rupture Risk Assessment for Graphite/Epoxy Composite Overwrapped Pressure Vessels on the International Space Station,*" Aerospace Report No. ATR-2009(5298)-6, Sept. 30, 2009.

# Strain vs. Time Behavior During Creep

## Classical Case



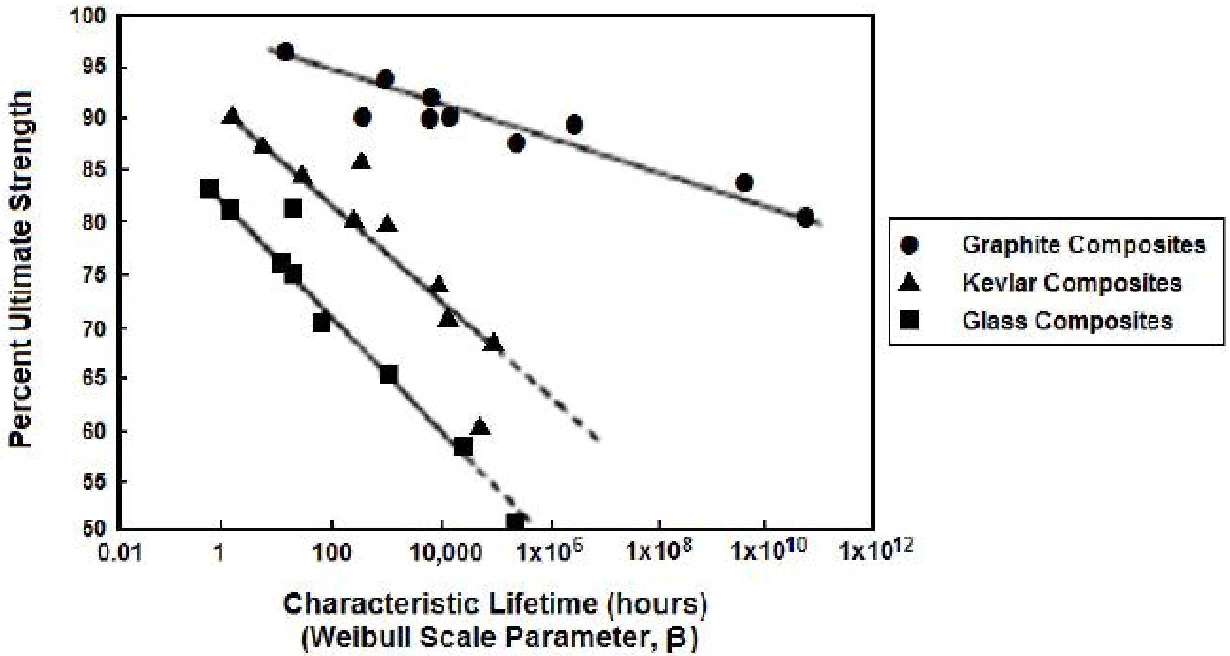

**distinct tertiary creep phase**

(ductility observed before rupture)

**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.§



§ J. T. Shaffer, "*Stress Rupture of Carbon Fiber Composite Materials,*" in Proc. 18th Intl. SAMPE Technical Conference, p. 613 (1986).

# Goals

- Develop quantitative acoustic emission (AE) procedures specific to C/Ep overwraps, but which also have utility for monitoring damage accumulation in composite structures in general
- Lay groundwork for establishing critical thresholds for accumulated damage in composite structures, 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 fail COPVs at a critical FR below 1.0, indicative of severe accumulated damage and a known level of fiber breakage

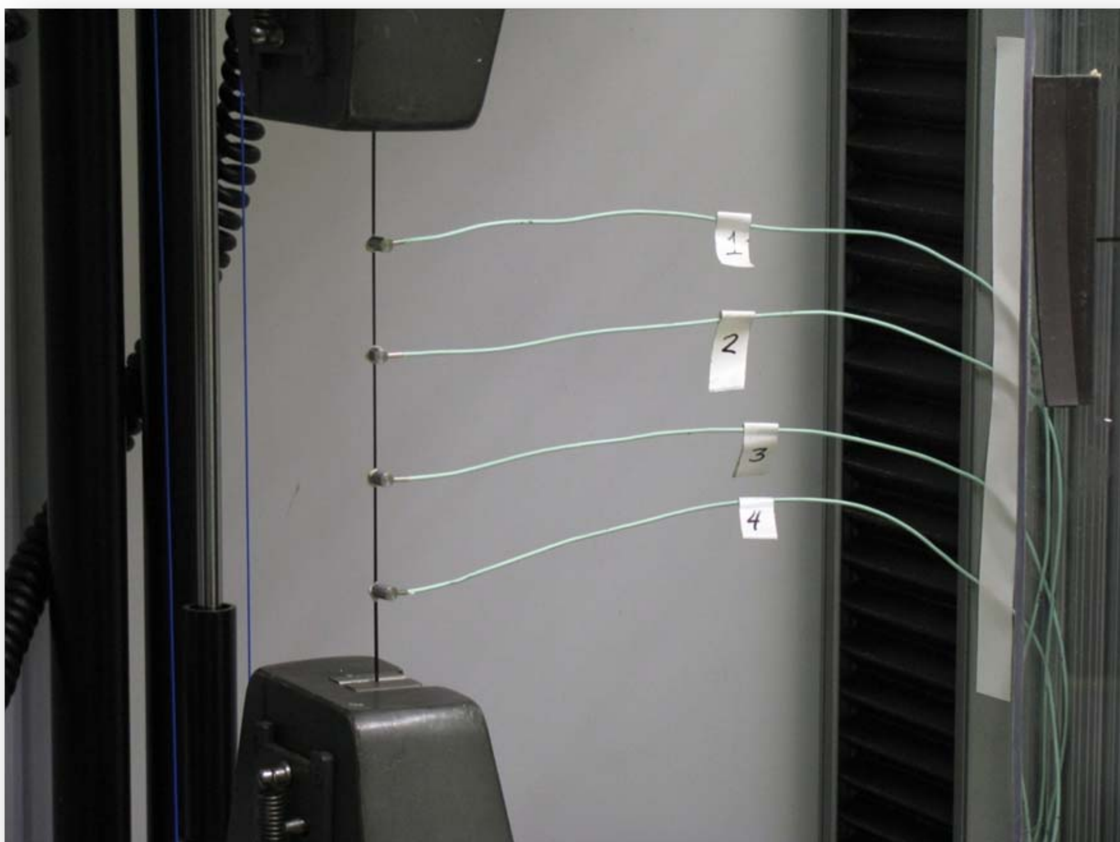
# *Experimental*

Load control and AE data acquisition system consisting 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)

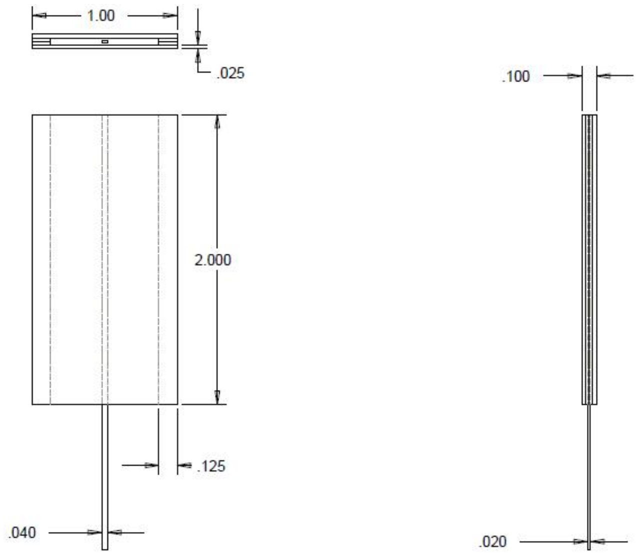
# *Experimental*

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

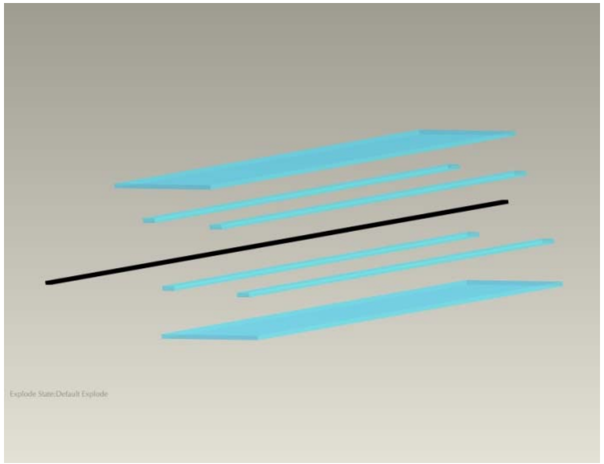
Tabbing: shear strength of epoxy and bonded grip length important variables§

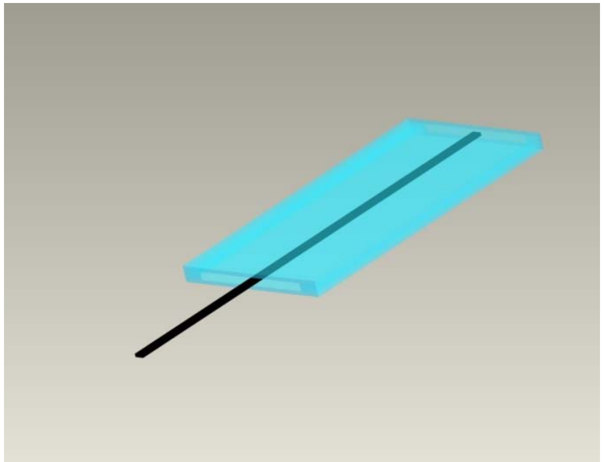



$$L_{min} = F^{tu}h/2F^{su}$$

where:

$L_{min}$ = minimum required bonded tab length, mm [in.];
$F^{tu}$ = ultimate tensile strength of coupon material, MPa [psi];
$h$ = coupon thickness, mm [in.]; and
$F^{su}$ = ultimate shear strength of adhesive, coupon material, or tab material (whichever is lowest), MPa [psi].





§ 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)
ASTM D 3039, *Test Method for Determining Tensile Properties of Polymer Matrix Composite Materials*, American Society for Testing and Materials, West Conshohocken, PA (2007)

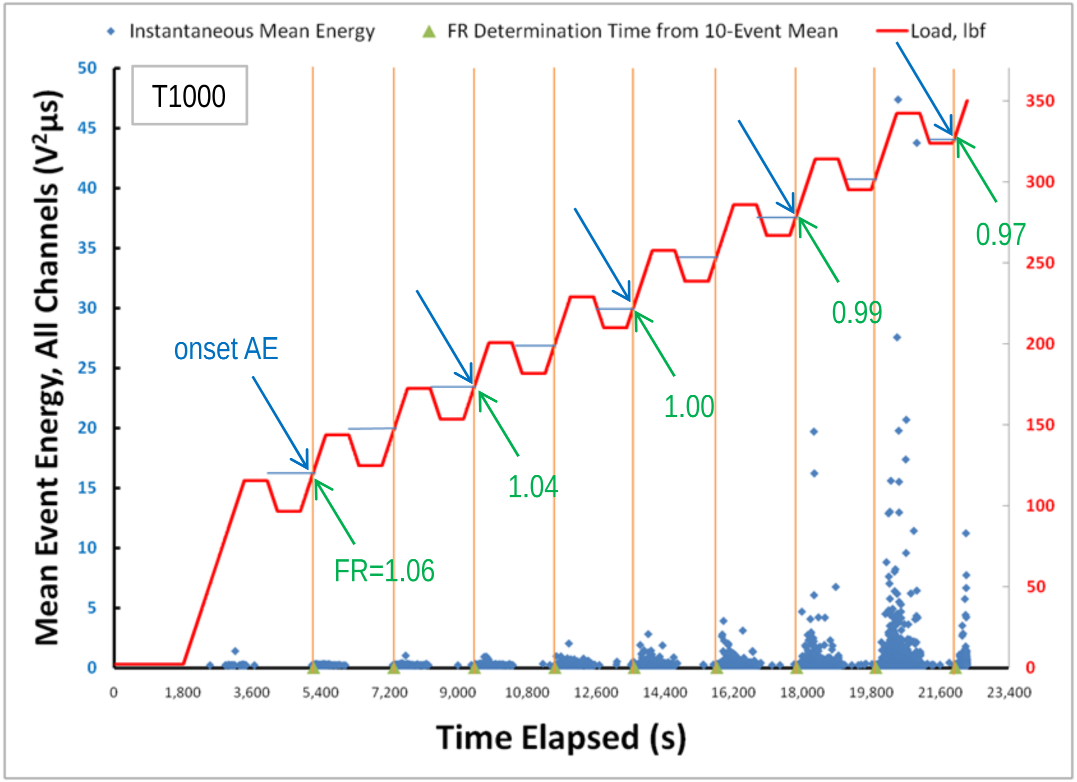
# AE Data Filtering

- 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 $lb_f$)
      - Events with an average energy above this value were considered significant

# Felicity Ratio Analysis

# Experimental

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

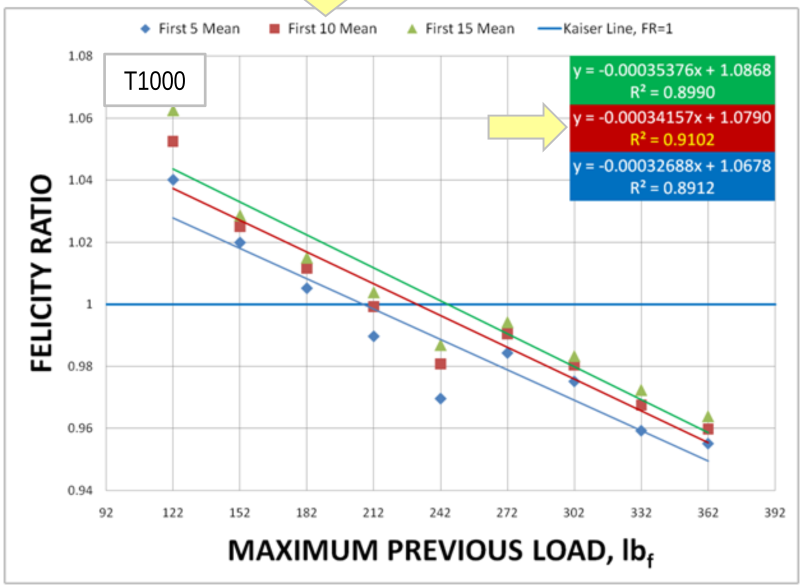


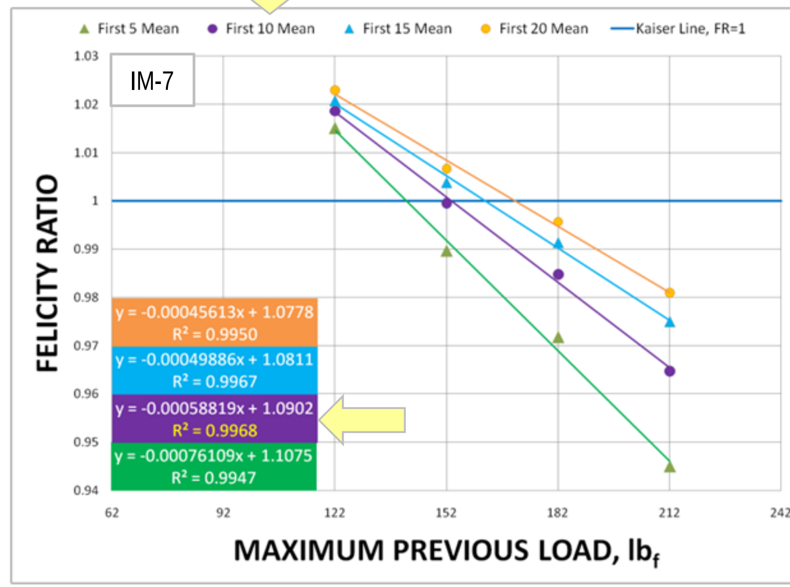

- ILH profile is based on the pressure tank examination procedure described in ASTM E 1067 §

§ ASTM, Practice for Acoustic Emission Examination of Fiberglass Reinforced Plastic Resin (FRP) Tanks/Vessels, E 1067, *American Society for Testing and Materials*, West Conshohocken, PA, 19428-2959, 2001.

# Results & Discussion

- Linear decrease in FR with load noted for T1000 and IM-7 C/Ep, similar to the behavior noted for Kevlar 49-epoxy K/Ep

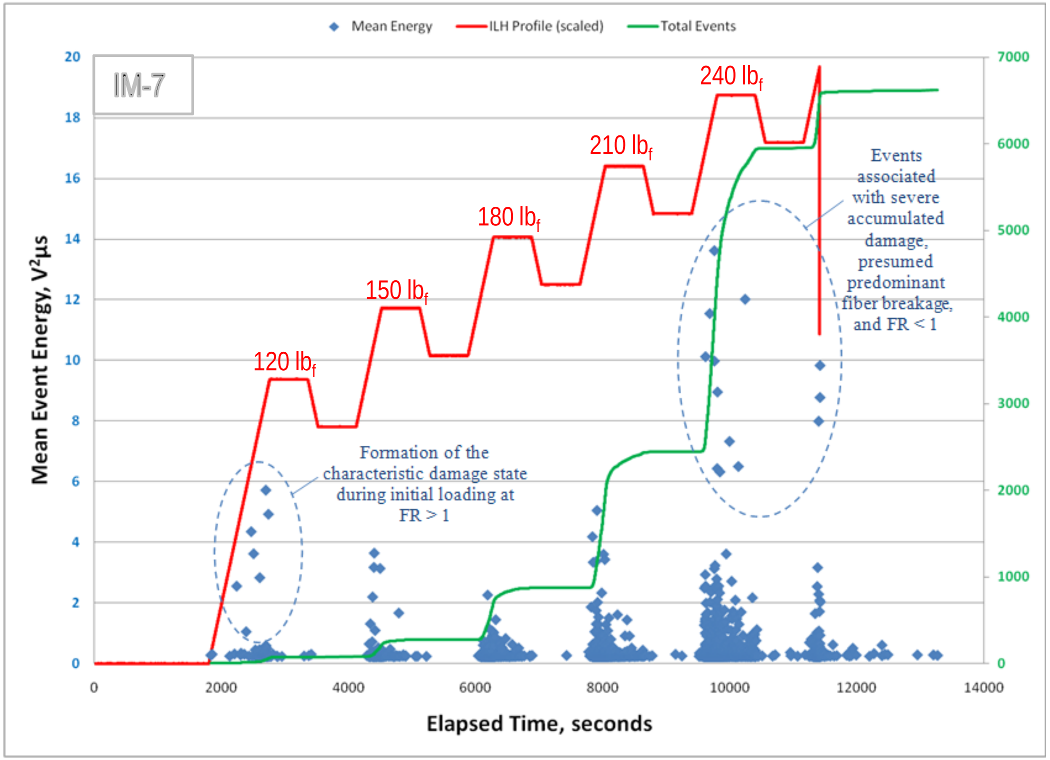



- For a given material and averaging method, the slope of least squares fit is indicative of damage tolerance
  - Flatter slopes correspond to good damage tolerance (in-character behavior)
  - Steep slopes correspond to low damage tolerance (out-of-character behavior)
- Kaiser effect violated at FR<1 ⇒ onset of severe accumulated damage
- C/Ep produced more AE than K/Ep

# Results & Discussion

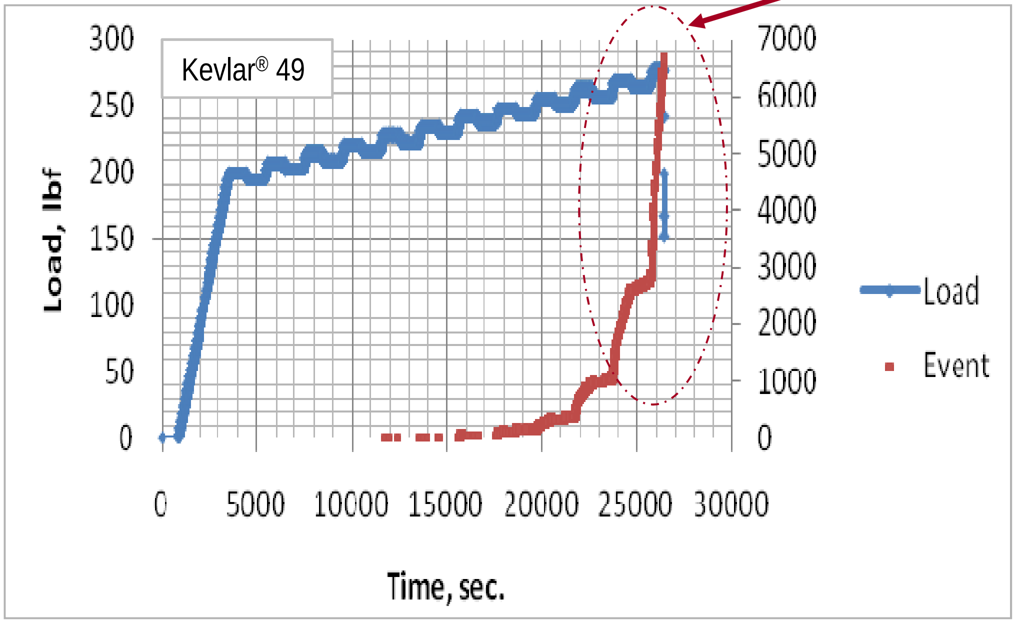
- Formation of characteristic damage state very evident at Load Ratios (LR) < 0.6 for IM-7




- 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 mixed mode failure mechanisms (cooperative matrix cracking, fiber/matrix debonding, fiber pull-out, fiber breakage)

# 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

# Summary of FR Results for Carbon/Epoxy

| Date | Test # | Material & Spool # | Filter[1] | F @ FR=1 ($lb_f$) | $F_{max}$ ($lb_f$) | σ @ FR=1 (ksi) | $\sigma_{max}$ (ksi) | FR* | Failure[2] |
|---|---|---|---|---|---|---|---|---|---|
| 83109 | 5 | IM7 #95 | 32% | 135 | 210 | 342 | 532 | 0.95 | XGB |
| 90109 | 6 | IM7 #95 | 27% | 151 | 234 | 383 | 591 | 0.945 | XGM |
| 90809 | 8 | IM7 #95 | 58% | 171 | 210 | 433 | 530 | 0.971 | XGM |
| 111009 | 10 | IM7 #117 | 9% | 193 | 252 | 488 | 637 | 0.961 | XGM |
| 32610 | D1 | IM7 #61 | 19% | 183 | 228 | 464 | 578 | 0.97 | XGM |
| 82509 | 1 | T1000 #74 | 32% | 240 | 355 | 658 | 972 | 0.972 | XGT |
| 82609 | 2 | T1000 #74 | 46% | 231 | 369 | 633 | 1010 | 0.953 | XGT |
| 82809 | 4 | T1000 #74 | 37% | 226 | 362 | 618 | 992 | 0.977 | XGT |
| 90909 | 9 | T1000 #74 | 41% | 194 | 301 | 532 | 823[3] | 0.949 | Pull Out |
| 111809 | 11 | T1000 #74 | 6% | 152 | 181 | | 497[3] | 0.961 | Pull Out |
| 112309 | 12 | T1000 #74 | 5% | 212 | 238 | | 651[4] | 0.969 | XGB |
| 112409 | 13 | T1000 #155 | 4% | 181 | 379 | | 1037 | 0.945 | SGM |
| 112509 | 14 | T1000 #74 | 6% | 206 | 325 | | 890 | 0.966 | LGM |
| 40910 | D3 | T1000 #155 | 6% | 181 | 374 | 493 | 1024 | 0.95 | XGM |
| **Mean** | | **IM7** | **29%** | **167** | **227** | **422** | **575** | **0.959** | |
| | | **Std. Dev.** | **18%** | **24** | **18** | **60** | **45** | **0.012** | |
| **Mean** | | **T1000** | **22%** | **211** | **361** | **577** | **988** | **0.961** | |
| | | **Std. Dev.** | **18%** | **26** | **19** | **71** | **53** | **0.013** | |

UTS
5.3-7.9% scatter

FR*
1.2-1.4% scatter

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

[1] Data filter reflects percentage of events removed from the raw AE data

[2] Failure abbreviations per ASTM D 3039, *Test Method for Determining Tensile Properties of Polymer Matrix Composite Materials*, American Society for Testing and Materials, West Conshohocken, PA (2007)

# 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 a few ILH profiles to assess in-family or out-of-family response
  - Need to verify that specimens or test articles with low initial FR or steep FR vs. load slopes in fact fail prematurely, or in the case of COPVs, fail at lower burst pressure

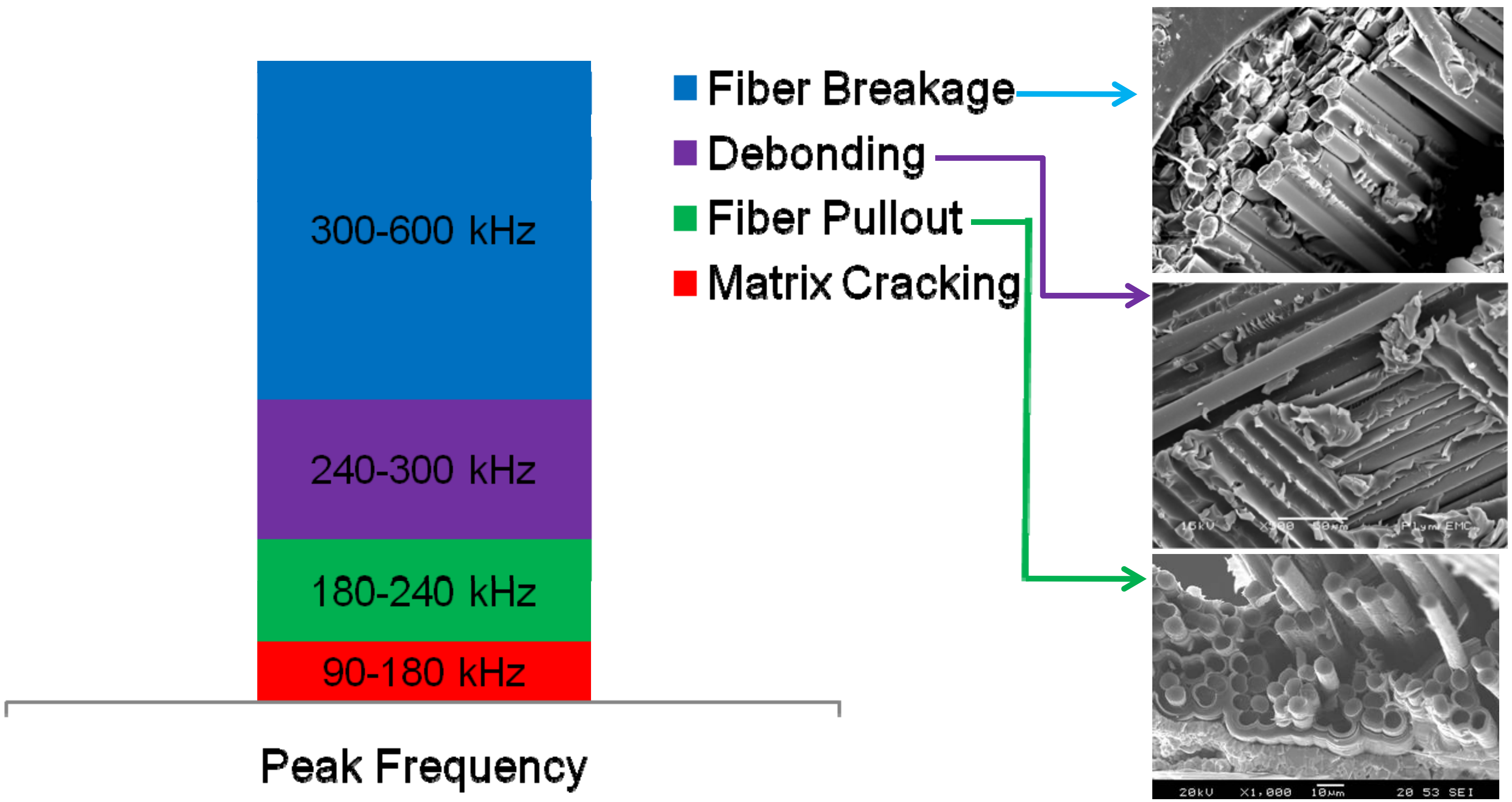


§ ASTM, Practice for Acoustic Emission Examination of Fiberglass Reinforced Plastic Resin (FRP) Tanks/Vessels, E 1067, *American Society for Testing and Materials*, West Conshohocken, PA, 19428-2959, 2001

# Waveform and FFT Analysis

# Results & Discussion

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



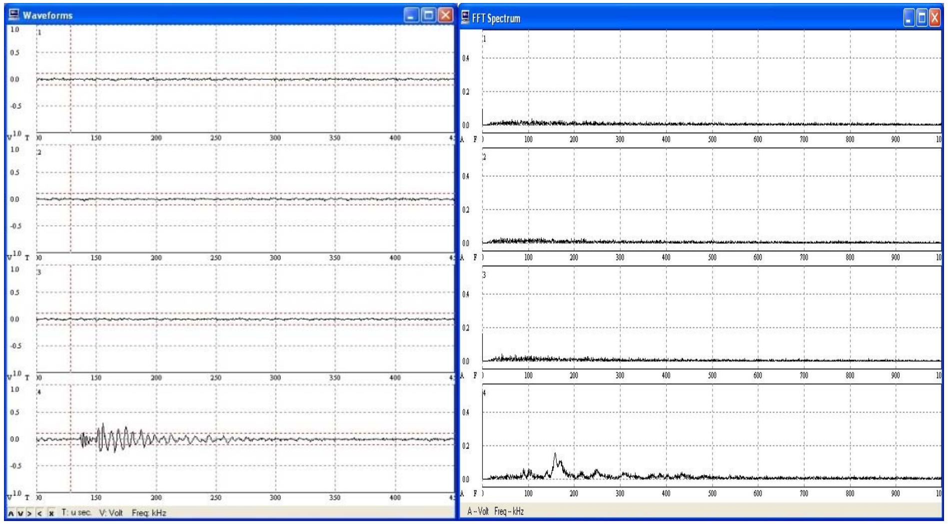

§ De Groot, P., P. Wijnen, and R. Janssen, "Real-time Frequency Determination of Acoustic Emission for Different Fracture Mechanisms in Carbon/Epoxy Composites," *Composites Sci. Technol.*, **55**, pp. 405-421 (1995).

Dzenzis, Y. A., and J. Qian, "Analysis of Microdamage Evolution Histories in Composites," *Int. J. Solids and Structures*, **38**, pp.1831-1854 (2001).

# Results & Discussion

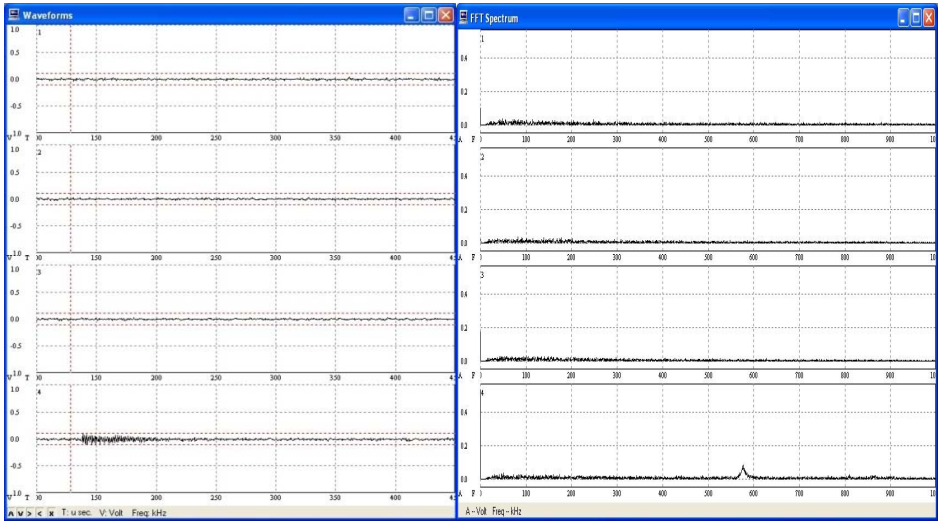
- In general, three different waveforms were observed for C/Ep

**1. Matrix Cracking**




waveform FFT

**2. Fiber Breakage**

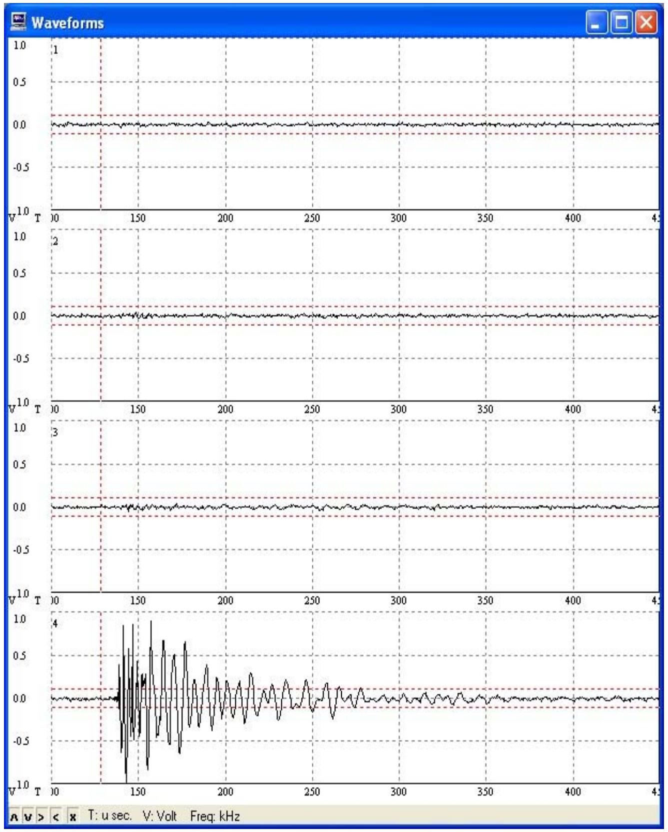



waveform FFT

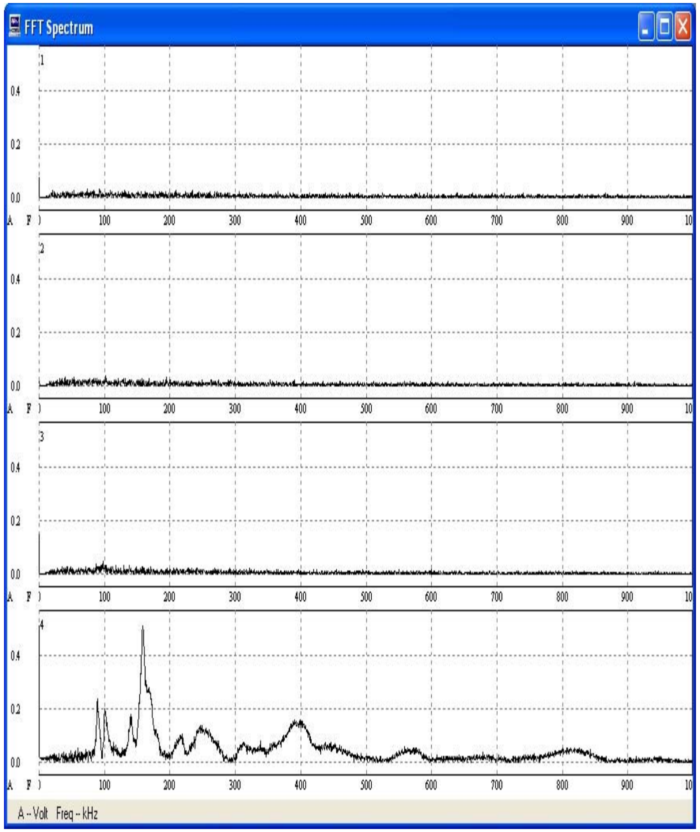
# Results & Discussion

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

### 3. Concerted, mixed mode failure



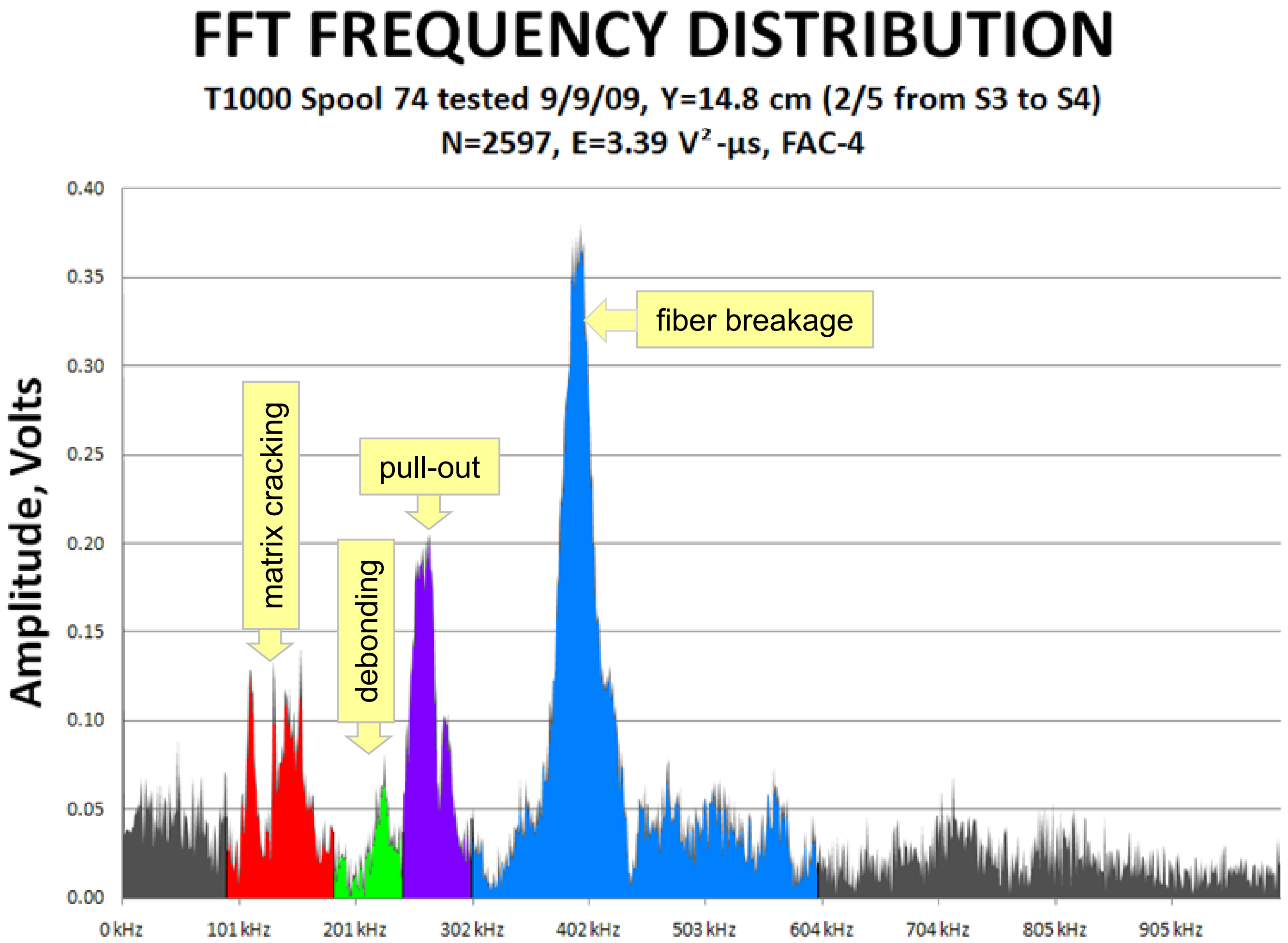

waveform




FFT

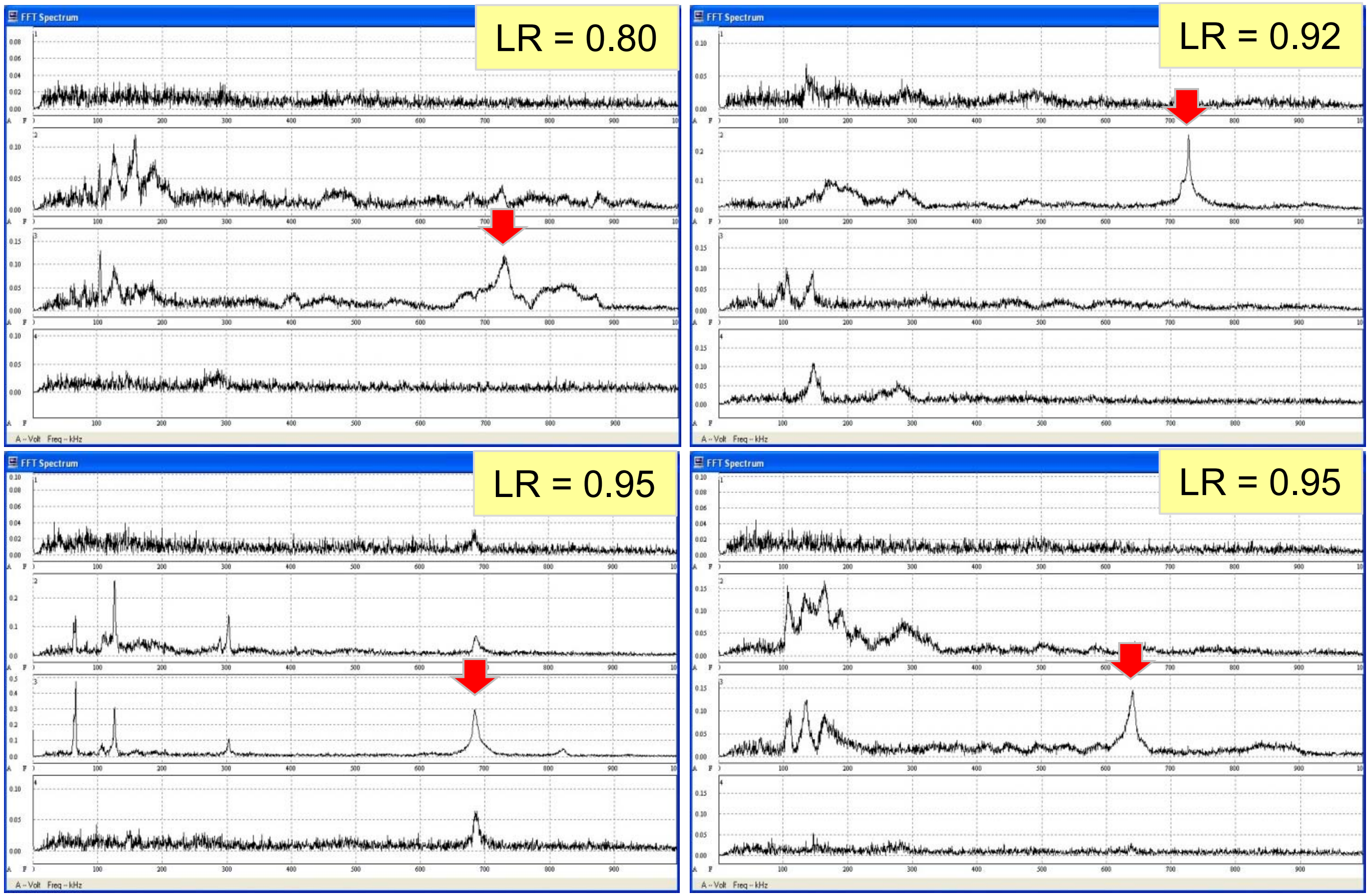
# Results & Discussion

## FFT showing concerted failure using De Groot's frequency ranges



# Results & Discussion

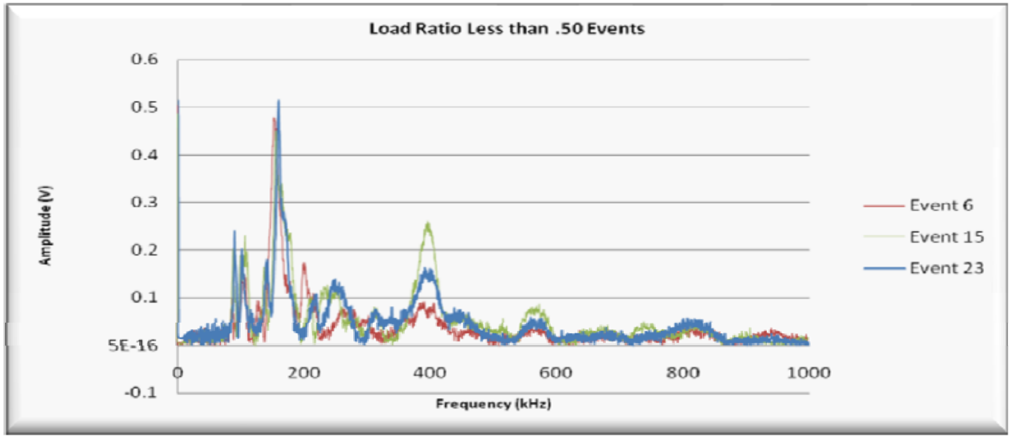
- High frequency peaks shifted downward with increasing load ratio: 731 kHz ⇒ 728 kHz ⇒ 685 kHz ⇒ 640 kHz
- Attributed to increasing accumulated damage, hence lower modulus, causing slower stress wave propagation



# Results & Discussion

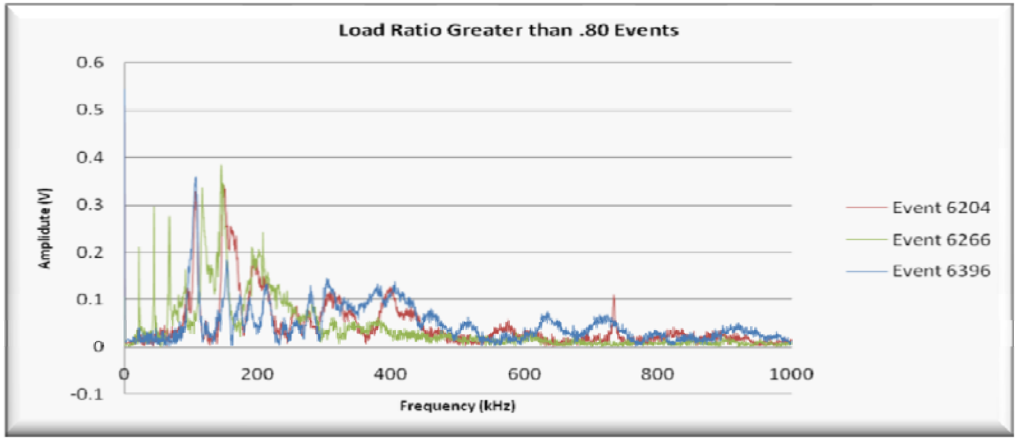
- IM-7 early *vs*. late life events

**Early Life**




LR < 0.5
first ILH ramp up

**Late Life**



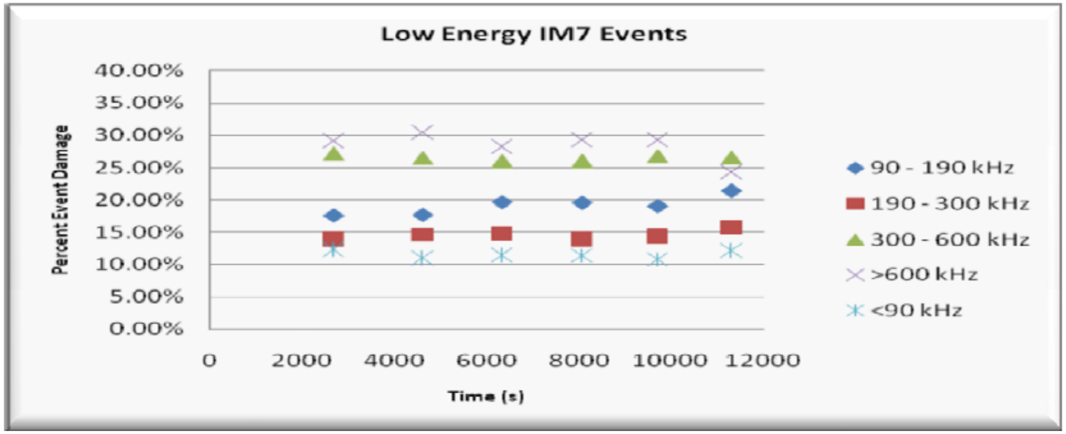

LR > 0.8
last ILH ramp up

- Notice the change from ordered to unordered peaks

# Results & Discussion

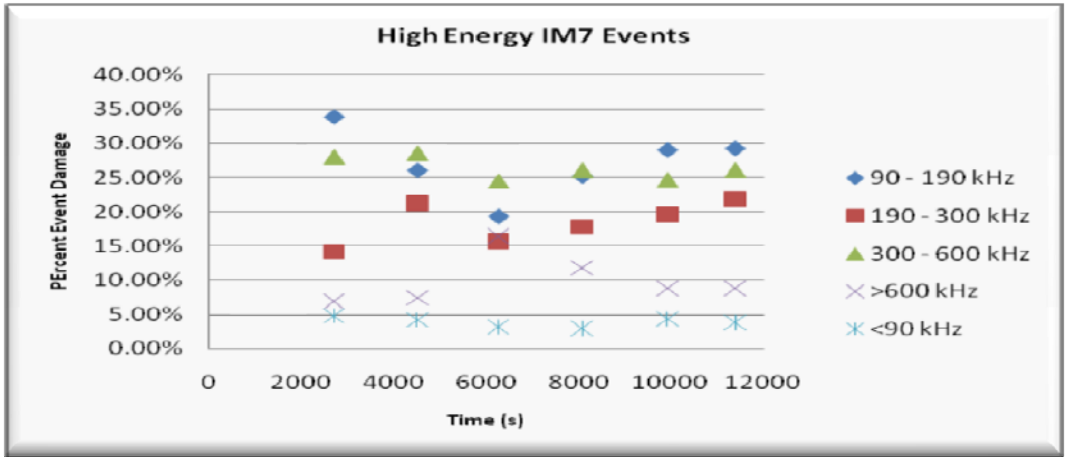
- IM-7 low *vs.* high energy events

**Low Energy**




$E < 2\ V^2$-μsec

**High Energy**

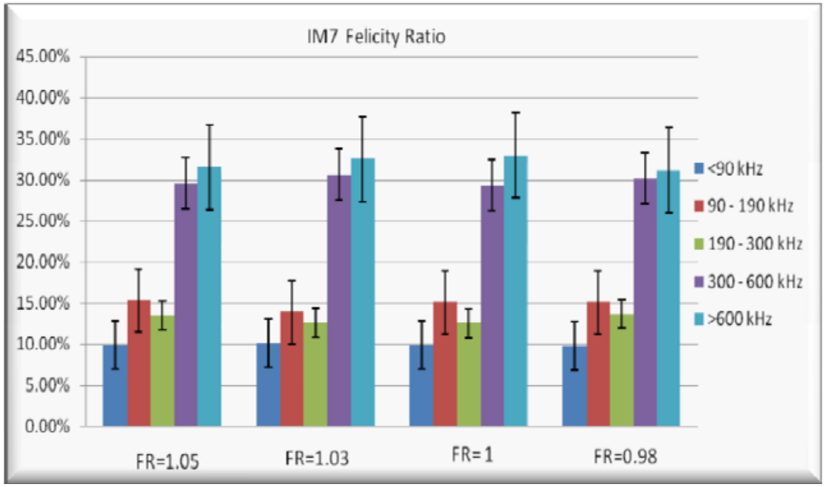


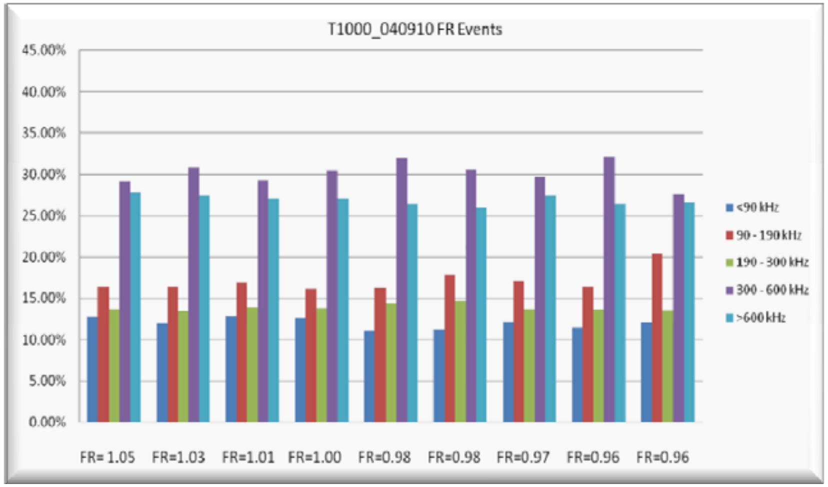

$E > 2\ V^2$-μsec

- Low energy events behave in an ordered fashion, while high energy events appear more random

# Results & Discussion

- IM-7 (2 specimens) and T1000 (1 specimen) Felicity ratio events (first ten events, $FR_{10}$ method) were then compared to see if they had a characteristic frequency distribution or energy




- Fiber breakage dominates the FR, otherwise FR events reflect a concerted failure mode for both types of C/Ep
- Other minor differences between IM-7 and T1000 noted, but same overall trend:

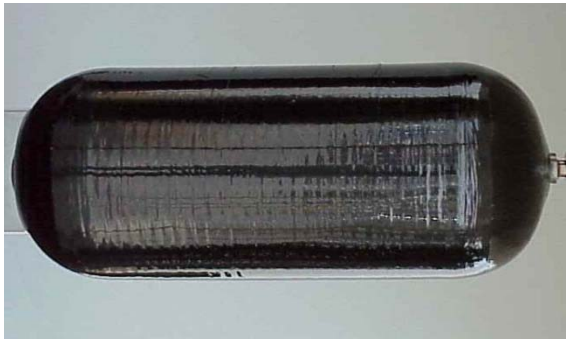
300-1000 kHz > 90-190 kHz > 190-300 kHz

(*fiber breakage > matrix cracking > debonding/pull-out*)

# Application to Composite Overwrapped Pressure Vessels (COPVs)

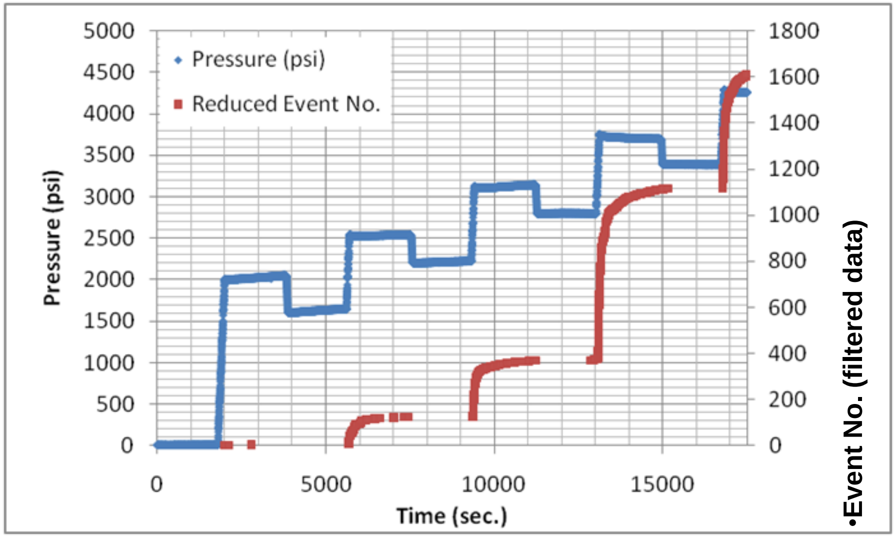
# Results & Discussion

A 6.3-in. diameter IM-7 COPV was subjected to an ILH pressure schedule at LR ≈ 0.3 to 0.9

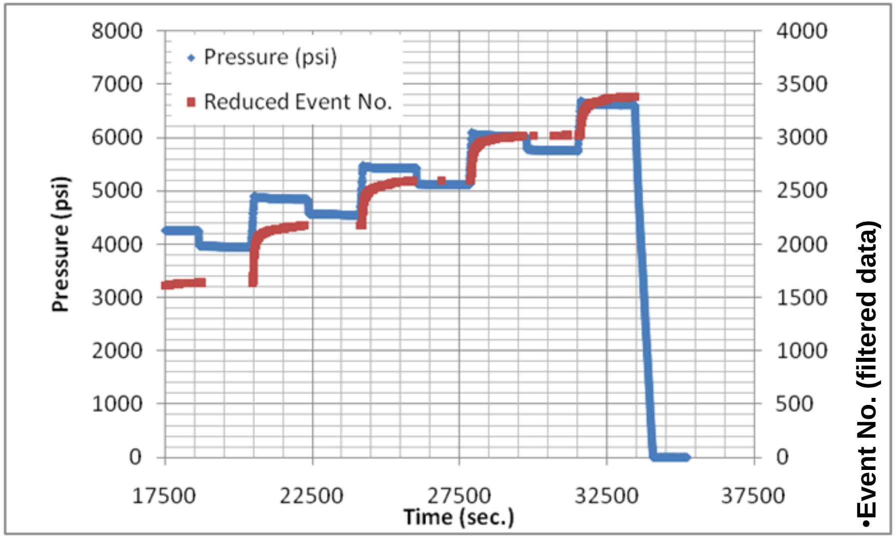


## Pressure & Events vs. Time

**0 to 17500 s**

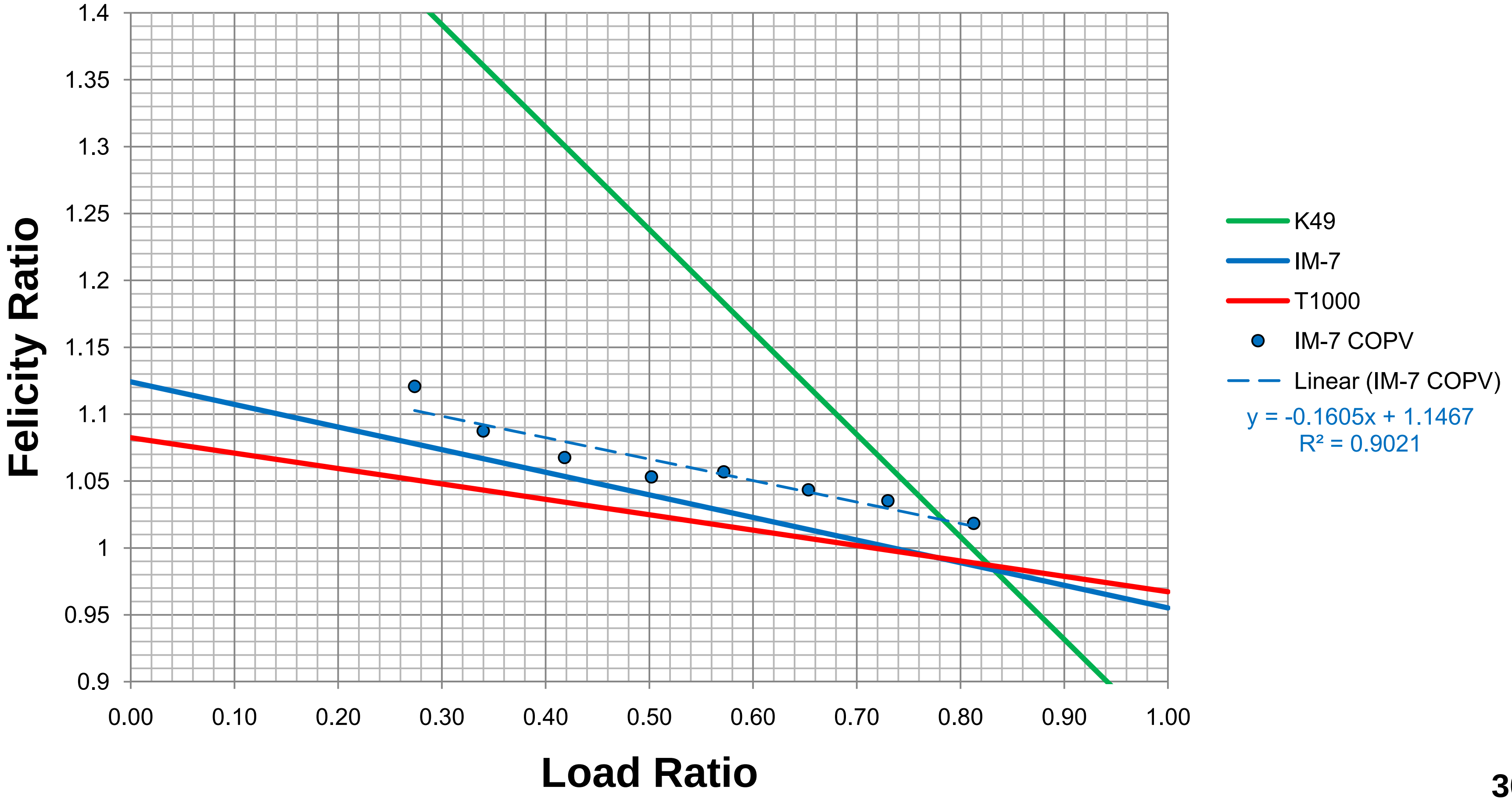



**17500 to 37500 s**



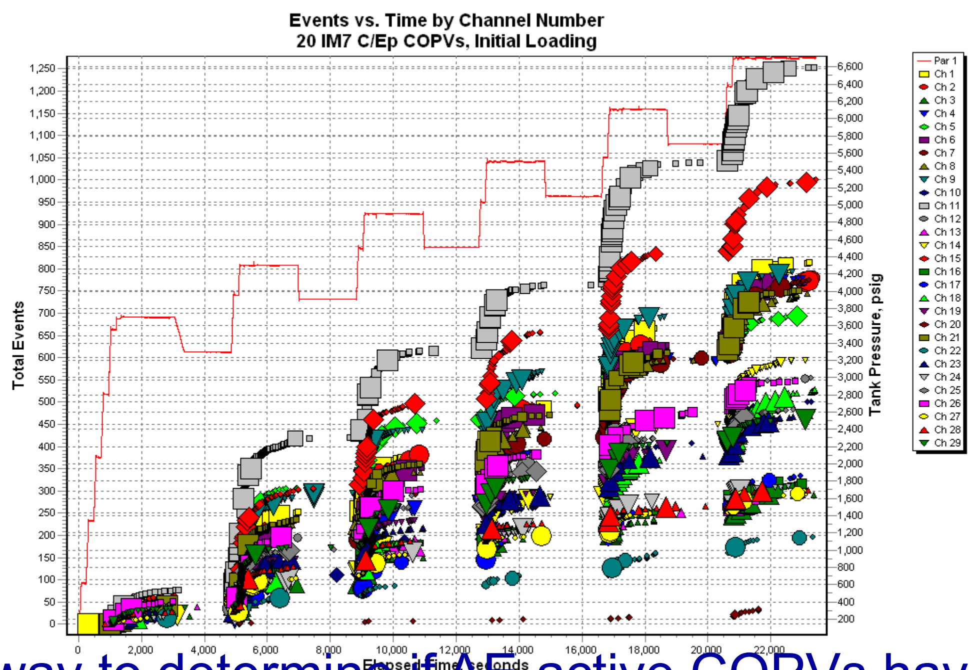
# Results & Discussion

## IM-7 tow data (solid blue line) consistent with IM-7 COPV data (blue symbols)



# Results & Discussion

- Follow-on application of ILH methodology to a 20-bottle COPV set (IM-7 overwraps) revealed variable response within same batch and lot




- Efforts underway to determine if AE-active COPVs have lower FR, steeper FR *vs*. pressure curve, or are more prone to burst at lower pressures
- Only 1-3 sensors used, so source location is not possible/problematic. As well as detection of lower energy, high frequency events now contribute to a linear FR response.

# Conclusions

- FR* behaves like a universal parameter that varies less than the UTS
  - Offers the possibility of using the FR as a robust pass/fail acceptance criterion for C/Ep composite materials
  - By analogy, FR* would be expected to vary less than the burst pressure for a family of COPVs of equivalent design, for example
- ASTM-based ILH methods were found to give a reproducible, quantitative estimate of the stress threshold at which significant accumulated damage occurs
  - FR events are low energy (<2 $V^2\mu s$)
  - FR events occur close to failure locus
  - FR events composed of >50% fiber breakage (>300 kHz)
    - true for multiple IM-7 and T1000 uniaxial tow specimens
    - different trend may exist in quasi-isotropic lay-ups
  - FR events showed a consistent hierarchy of concerted, mixed mode damage mechanisms regardless of applied ILH load ratio
- Initial application of FR and FFT analysis to COPVs shows great promise

# Acknowledgments


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

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

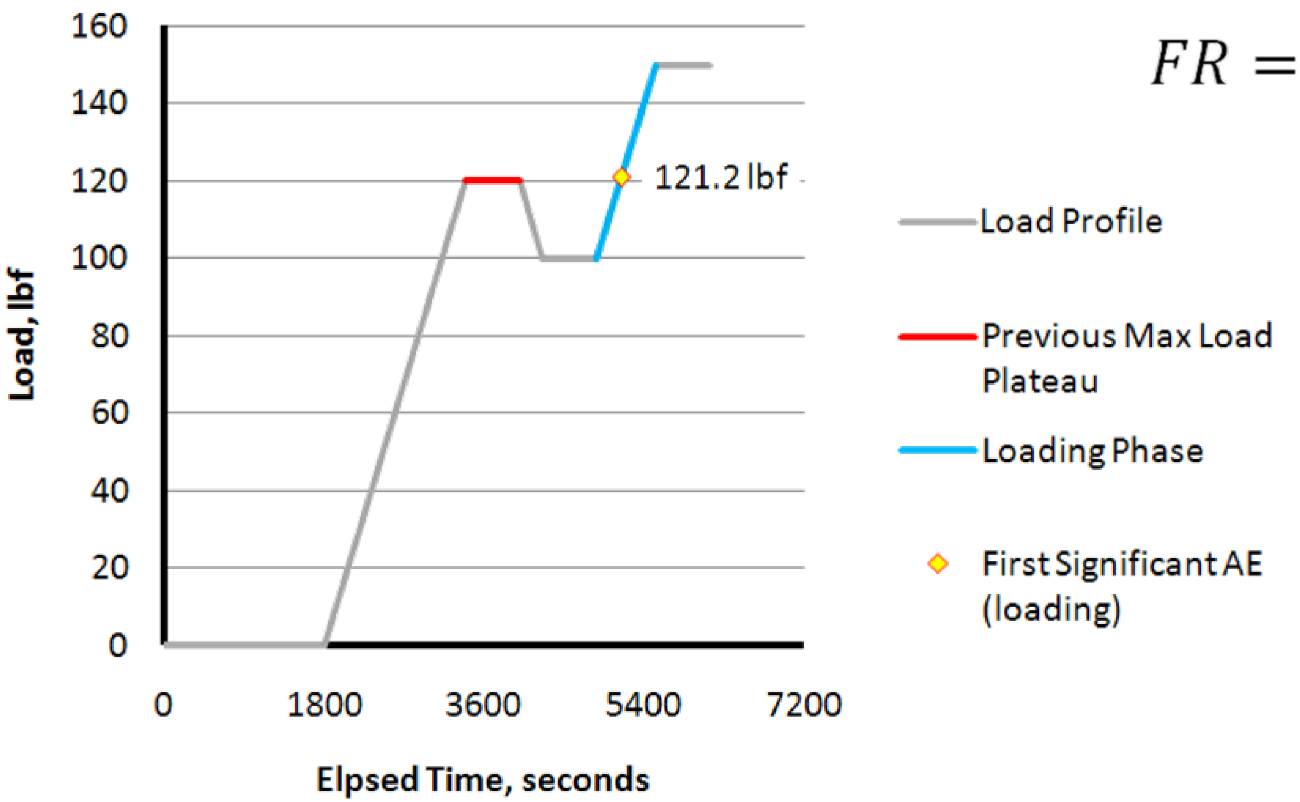
Paul Spencer, Brooks Wolle, and Ben Gonzalez (NASA JSC WSTF)
Universal tensile tester set-up & tabbing

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

# Felicity ratio (FR)

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

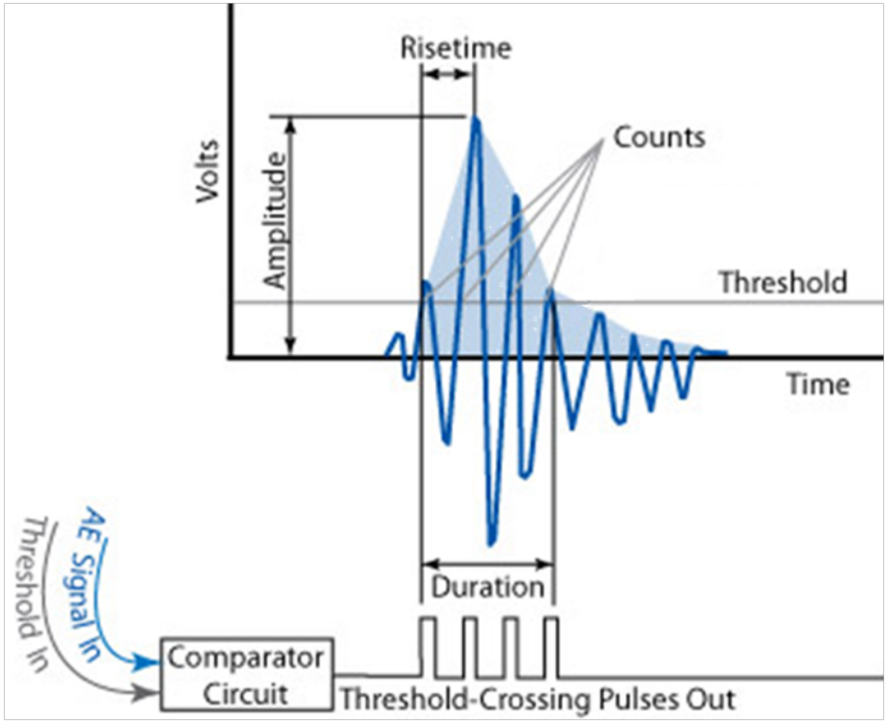



$$FR = \frac{121.2}{120} = 1.01$$

# *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/)

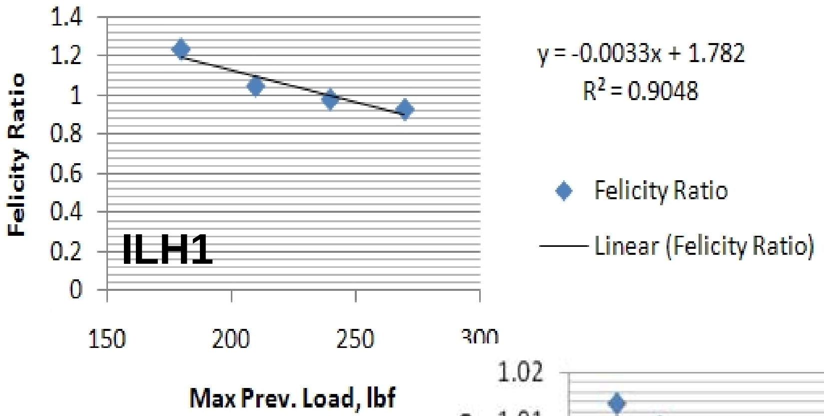


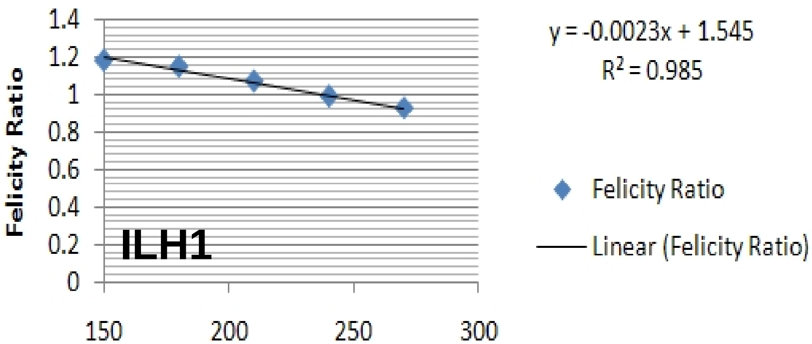

•100 mV

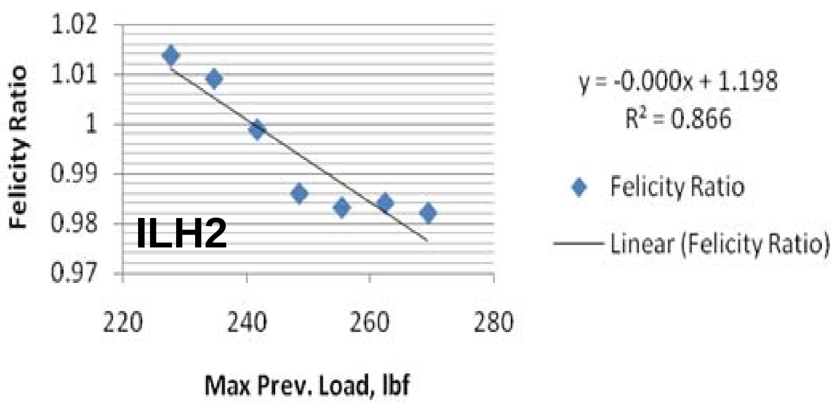
# Results & Discussion

- For Kevlar-epoxy 4650 denier tow, correlation coefficients for ILH 1 & 2 methods indicated good ($R^2$ = 0.866) to excellent ($R^2$ = 0.985) agreement:

Kevlar® 49-epoxy

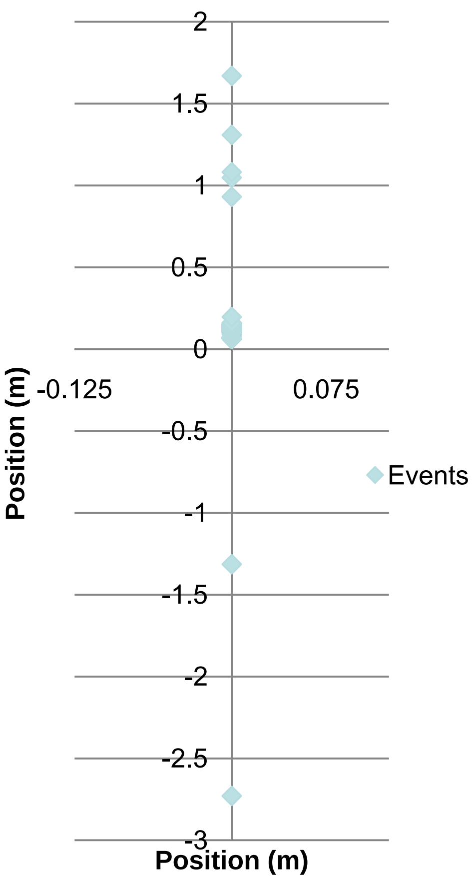


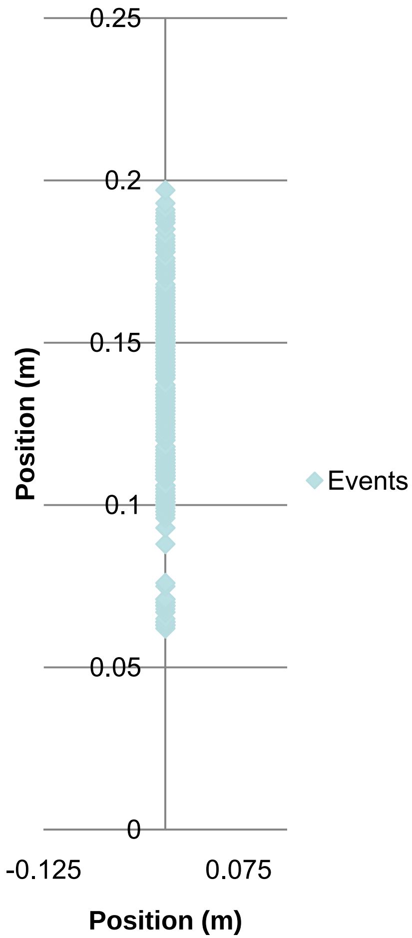



# AE source location method improved

Old



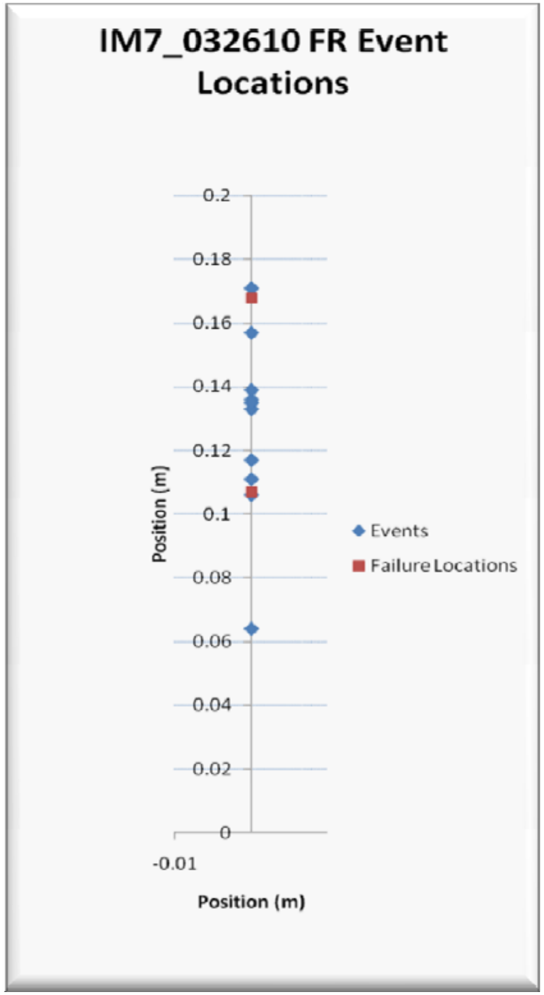

New




- 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 files 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

# Results & Discussion

## Source location of FR events showed that they occur at or near the locus of failure




- IM7_032610 specimen had intact tow between 0.17 and 0.20 m (upper tab) and 0 (lower tab) and 0.115 m
- Tow region between 0.001 and 0.17 m obliterated (explosive failure)
- Most FR events were source located in the missing region that failed explosively