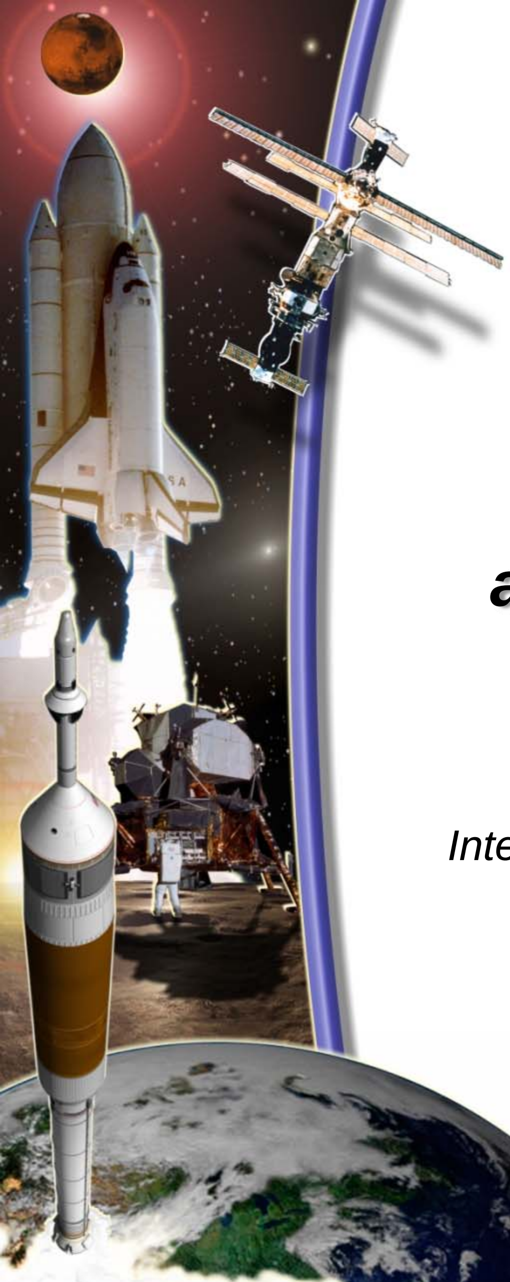


Use of Modal Acoustic Emission to Monitor Damage Progression in Carbon Fiber/Epoxy Tows and Implications for Composite Structures

Jess M. Waller, Regor L. Saulsberry, Ralph Lucero
NASA-JSC White Sands Test Facility
Charles T. Nichols
Intern, New Mexico State Univ., Dept. Mech. and Aerospace Engineering
Daniel J. Wentzel
Intern, Miami University, Department of Physics

ASNT Fall Conference & Quality Testing Show

NASA NDE II

Houston, TX

Thursday, 18 November 2010

Background

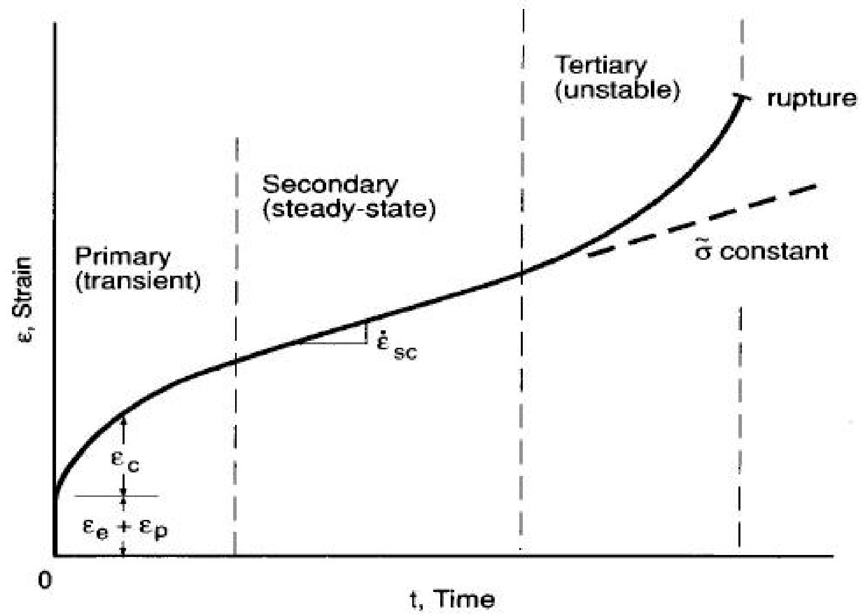
Issues:

- 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 inspectabilty 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"*

Strain vs. Time Behavior During Creep

Classical Case



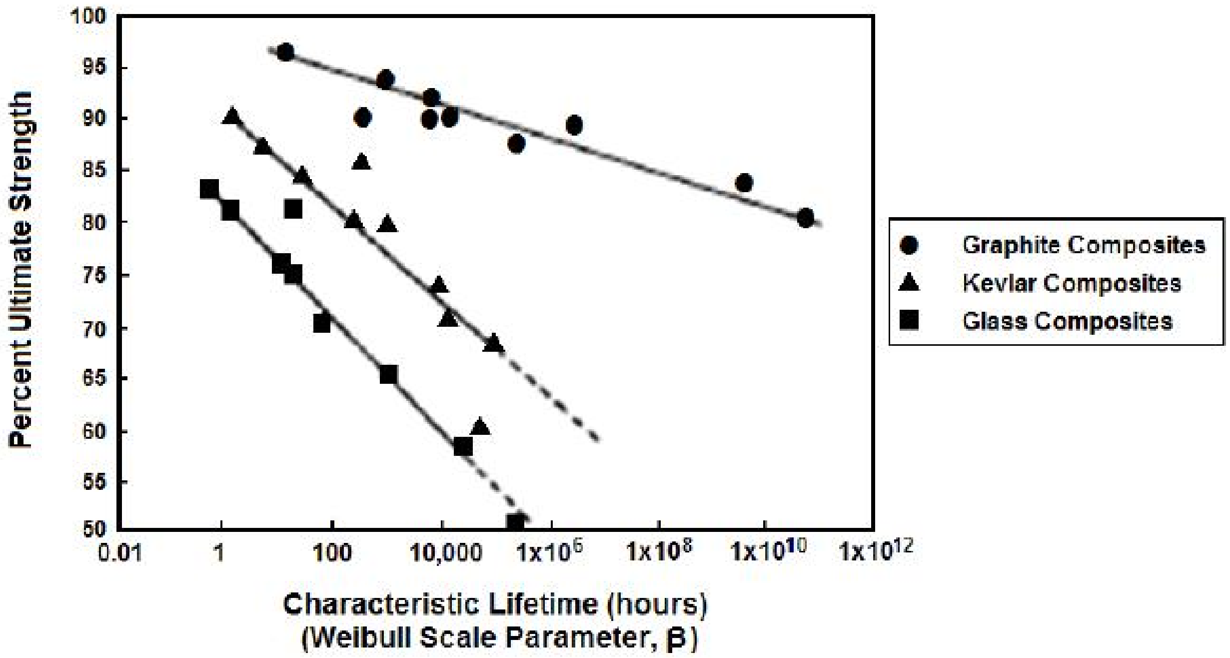
distinct tertiary creep phase

(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).

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 <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.

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

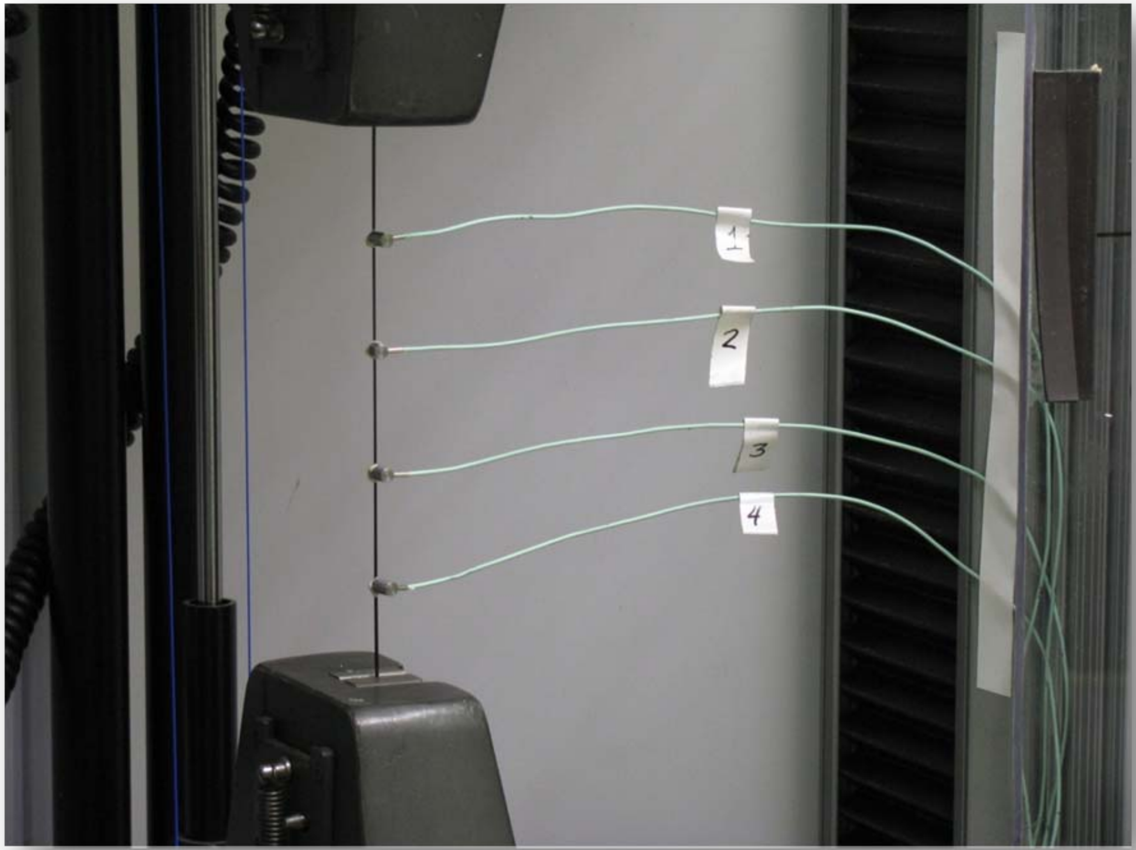
Experimental

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)

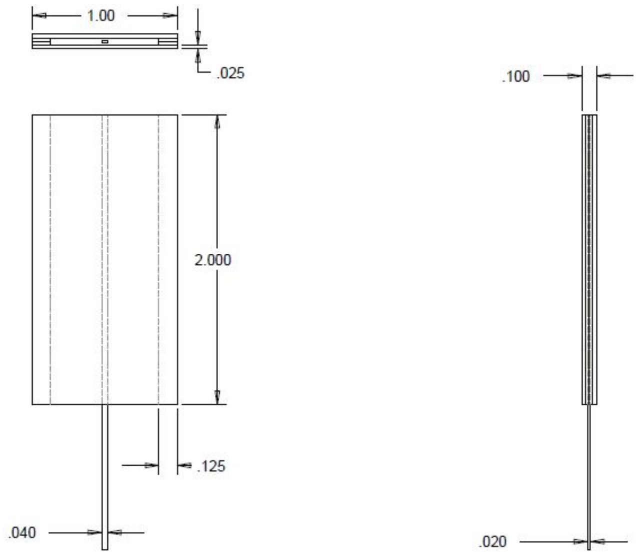
Experimental

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

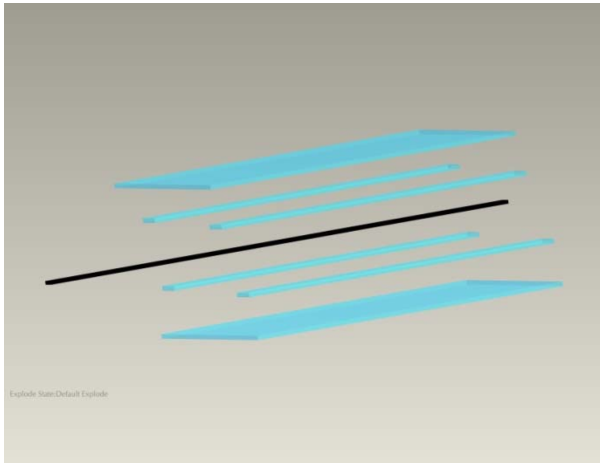
Tabbing: shear strength of epoxy and bonded grip length important variables§

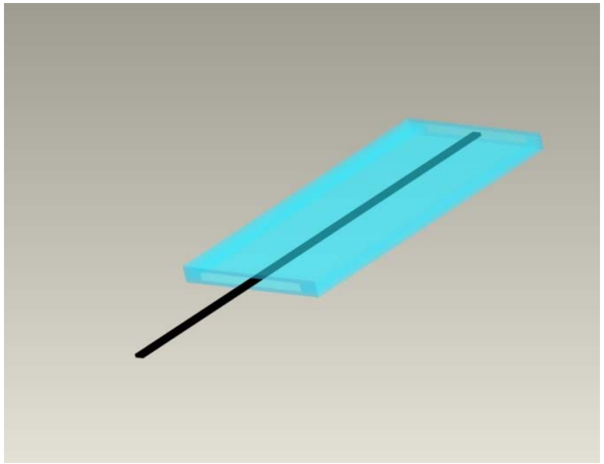


$$L_{min} = F^{tu}h/2F^{su}$$

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].



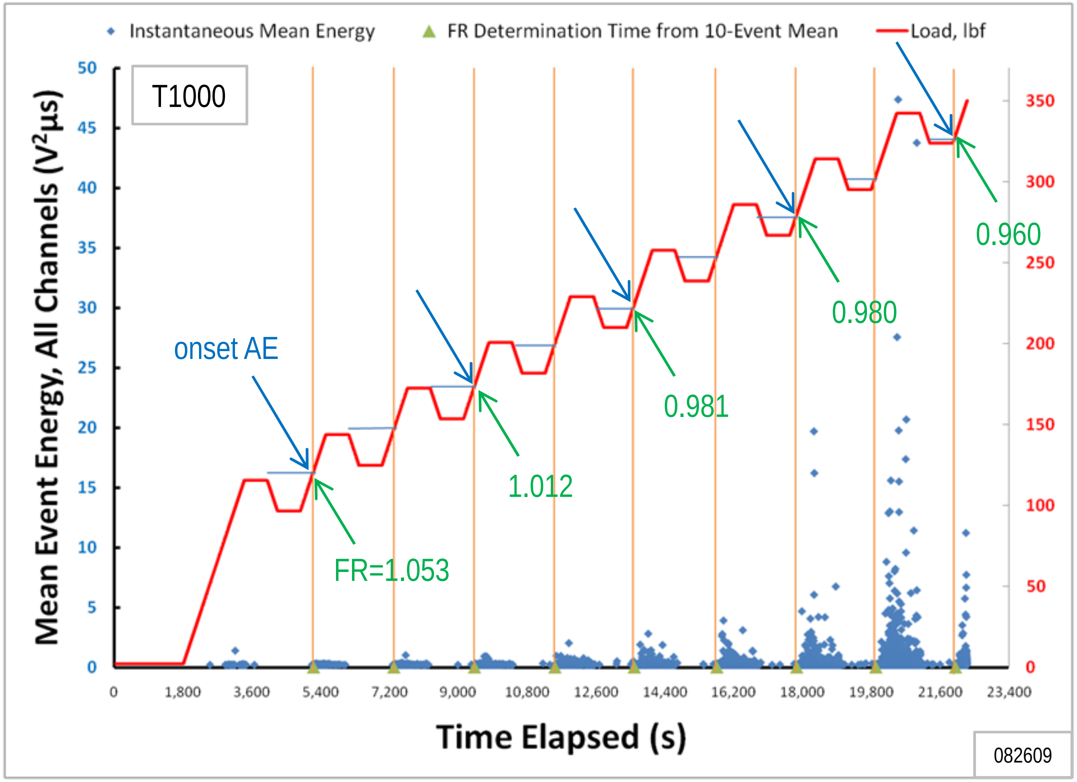


§ ASTM D 2343, *Test Method for Tensile Properties of Glass Fiber Strands, Yarns, and Rovings Used in Reinforced Plastics,* American Society for Testing and Materials, West Conshohocken, PA (2008)
ASTM D 3039, *Test Method for Determining Tensile Properties of Polymer Matrix Composite Materials*, American Society for Testing and Materials, West Conshohocken, PA (2007)

Felicity Ratio Analysis

Experimental

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

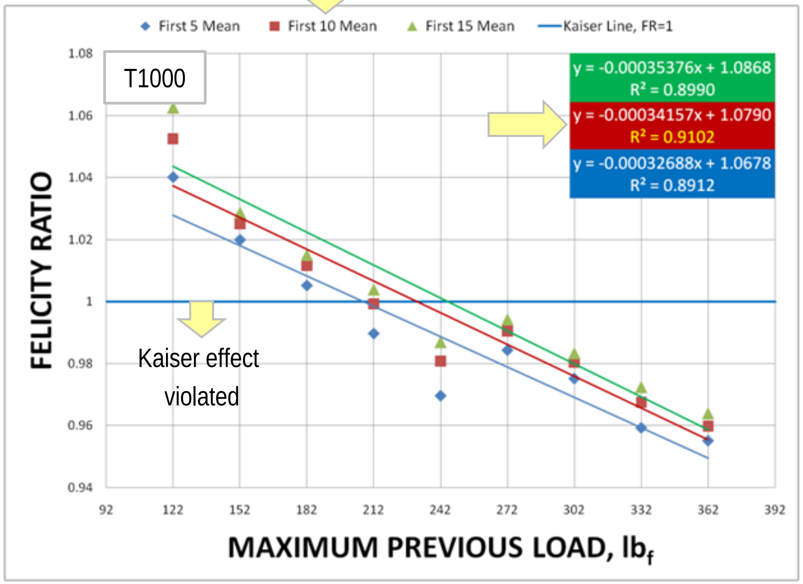


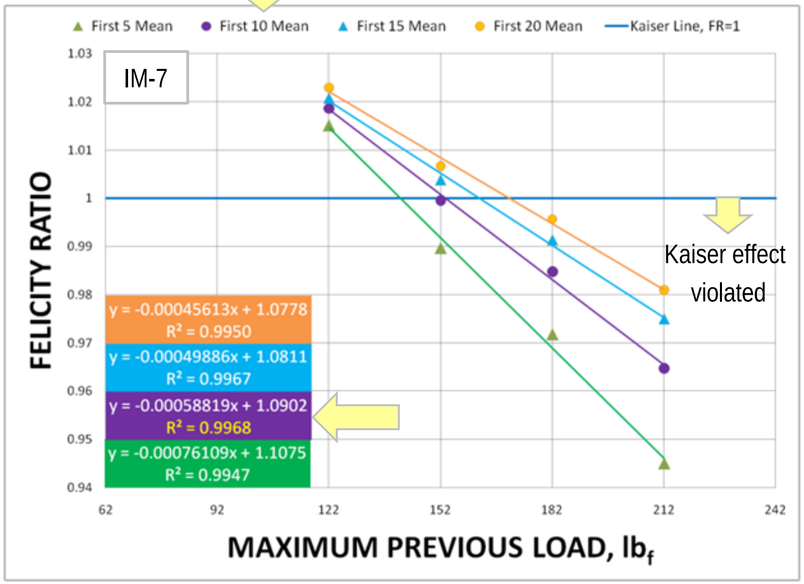
- ILH profile is based on the pressure tank examination procedure described in ASTM E 1067 §

§ 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 (R^2 > 0.9) and IM-7 (R^2 > 0.99) C/Ep, similar to the behavior noted for Kevlar 49-epoxy K/Ep

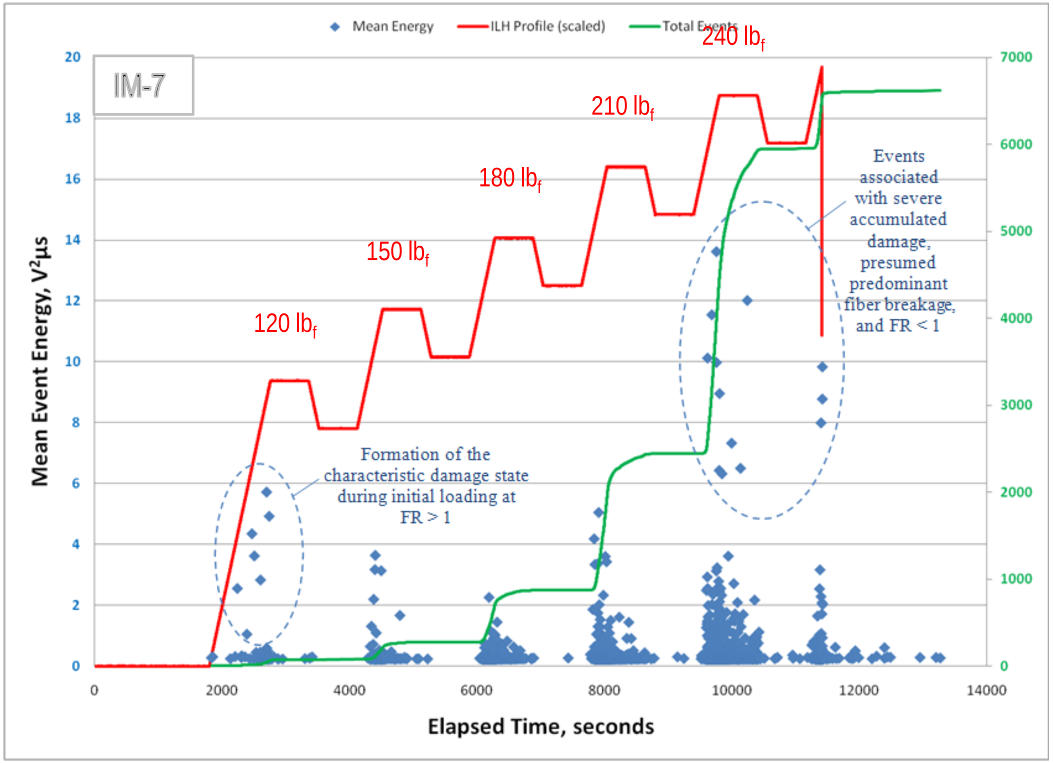




- For same material and averaging method, the slope of least squares fit is indicative of damage tolerance
  - Flatter slopes correspond to good damage tolerance (in-character behavior)
  - Steep slopes correspond to low damage tolerance (out-of-character behavior)
- Kaiser effect violated at FR<1 ⇒ 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

Date	Material & Spool #	Filter[1]	F @ FR=1 (lb_f)	F_{max} (lb_f)	σ @ FR=1 (ksi)	σ_{max} (ksi)	FR^*	Failure Type[2]
83109	IM7 #95	32%	135	210	342	532	0.95	XGB
90109	IM7 #95	27%	151	234	383	591	0.945	XGM
90809	IM7 #95	58%	171	210	433	530	0.971	XGM
111009	IM7 #117	9%[3]	193	252	488	637	0.961	XGM
32610	IM7 #61	19%	183	228	464	578	0.97	XGM
82509	T1000 #74	32%	240	355	658	972	0.972	XGT
82609	T1000 #74	46%	231	369	633	1010	0.953	XGT
82809	T1000 #74	37%	226	362	[illegible]	992	0.977	XGT
112409	T1000 #155	4%[3]	181	379	[illegible]	1037	0.945	SGM
112509	T1000 #74	6%[3]	206	325	[illegible]	890	0.966	LGM
40910	T1000 #155	6%[3]	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)

3 Improved tabbing method

Results & Discussion

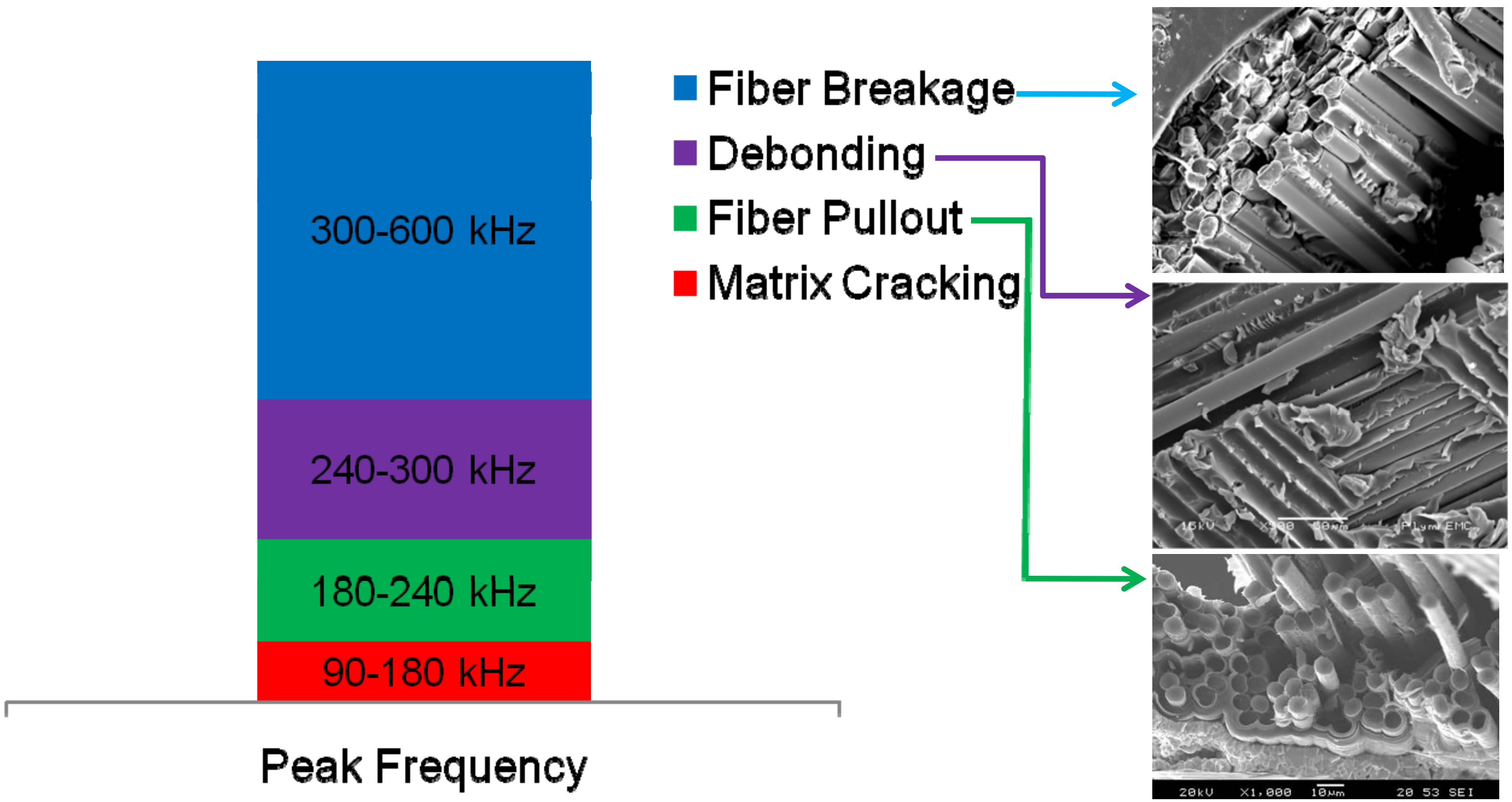
- Consistent *FR** values noted for T1000 and IM-7
- Suggests that the FR can be used as an analytical PASS/FAIL criterion for C/Ep composite materials
- Precedent: ASTM suggests using *FR* < 0.95 as failure criteria in fiberglass reinforced pressure vessels §
  - Experimental C/Ep failure criteria from 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

§ 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§

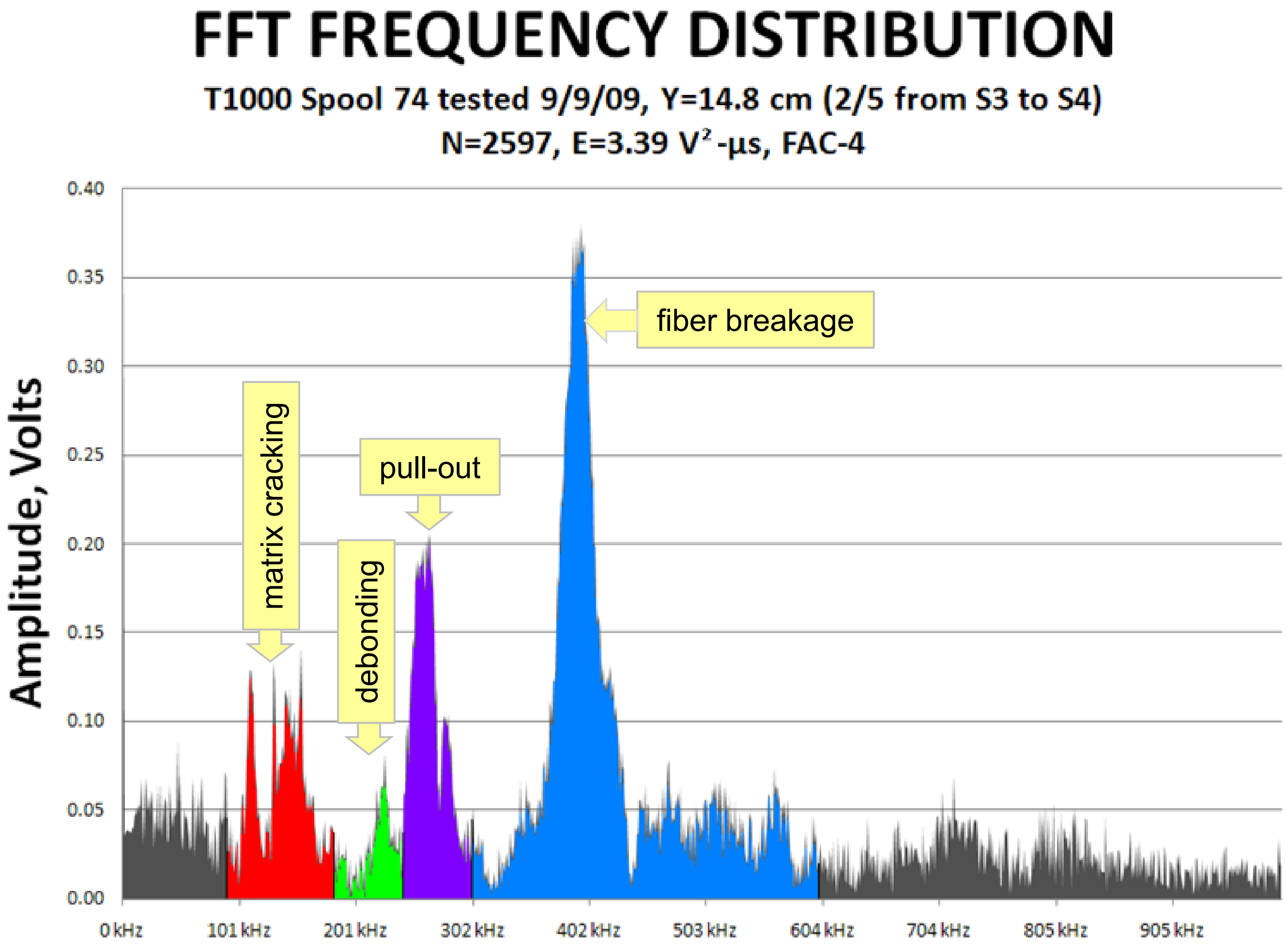


§ De Groot, P., P. Wijnen, and R. Janssen, "Real-time Frequency Determination of Acoustic Emission for Different Fracture Mechanisms in Carbon/Epoxy Composites," *Composites Sci. Technol*., **55**, pp. 405-421 (1995).

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

Results & Discussion

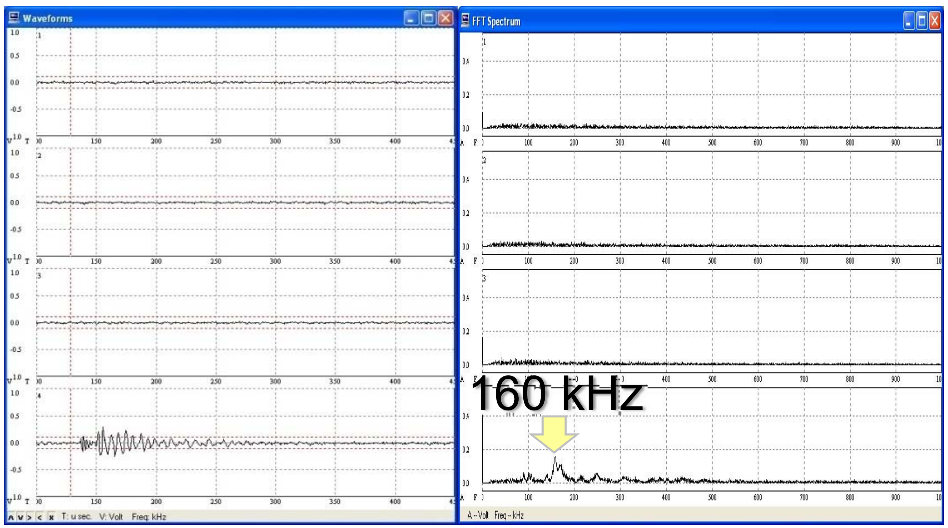
FFT (unfiltered) showing concerted failure using De Groot's frequency ranges



Results & Discussion

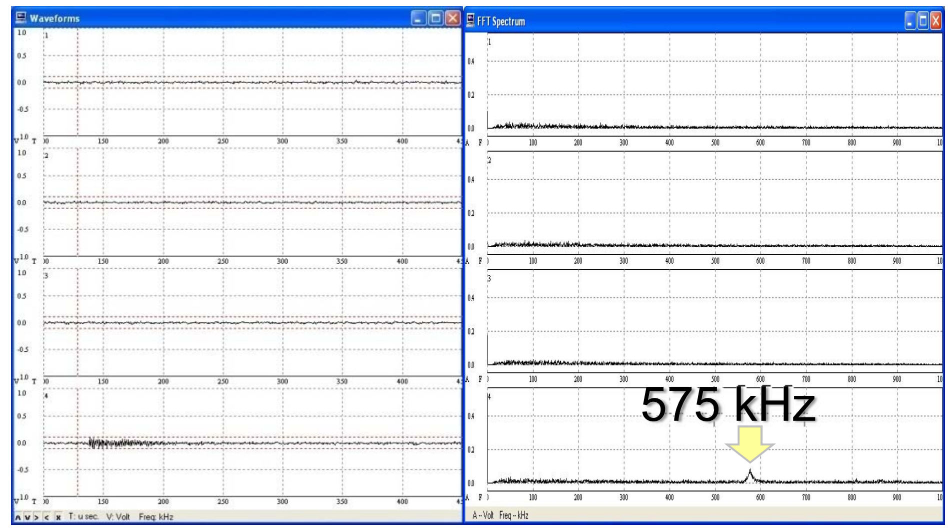
- In general, three different waveforms were observed for C/Ep

1. Matrix Cracking



waveform FFT

2. Fiber Breakage

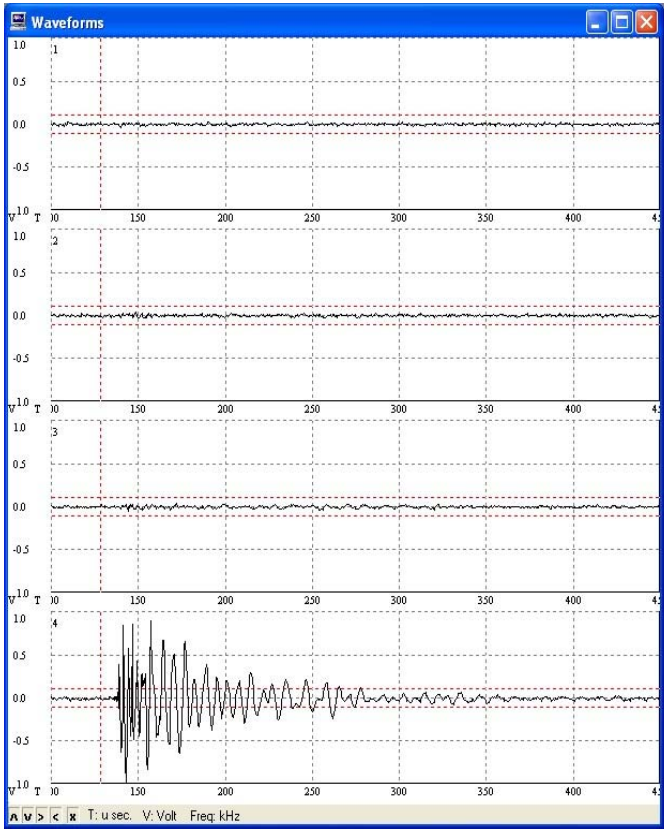


waveform FFT

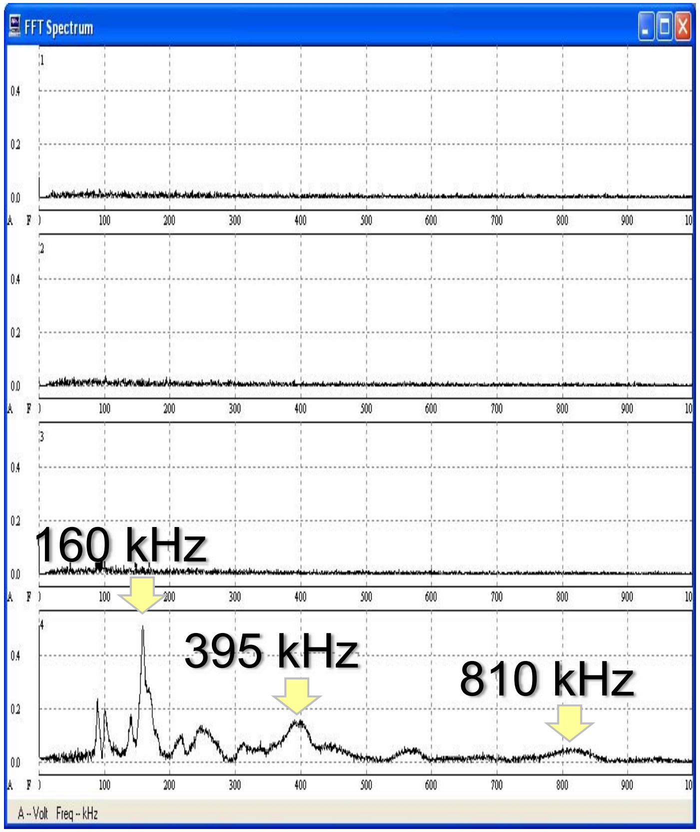
Results & Discussion

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

3. Concerted, mixed mode failure



waveform

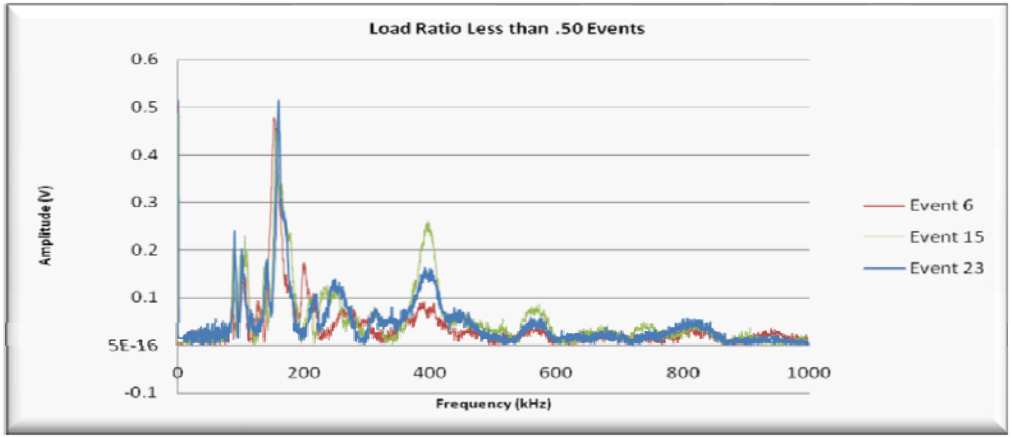


FFT

Results & Discussion

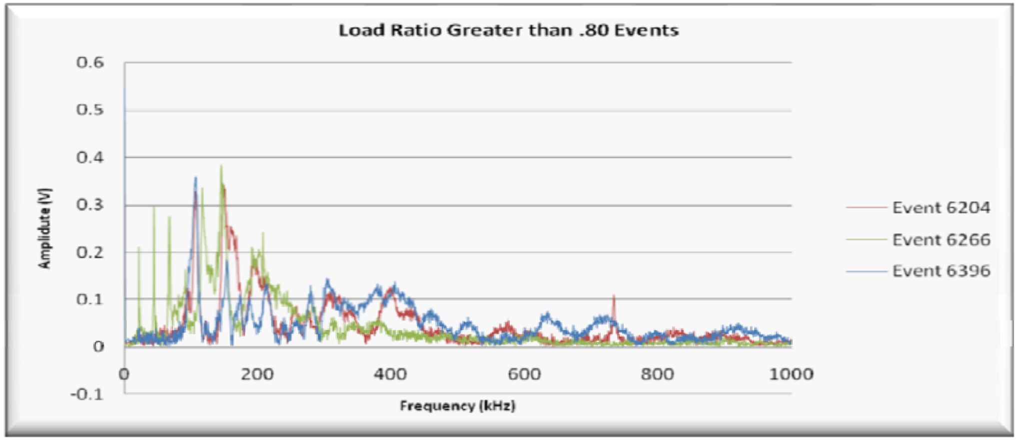
- IM-7 early *vs*. late life events

Early Life



LR < 0.5
first ILH ramp up

Late Life



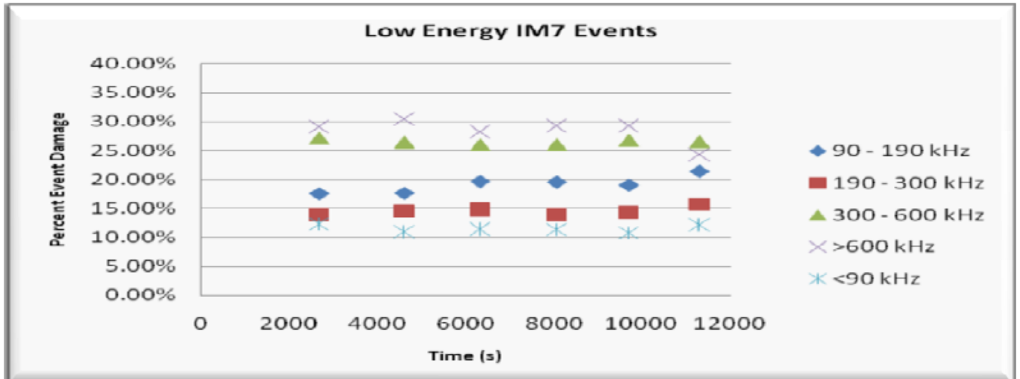
LR > 0.8
last ILH ramp up

- Notice change from ordered (early) to unordered peaks (late life)

Results & Discussion

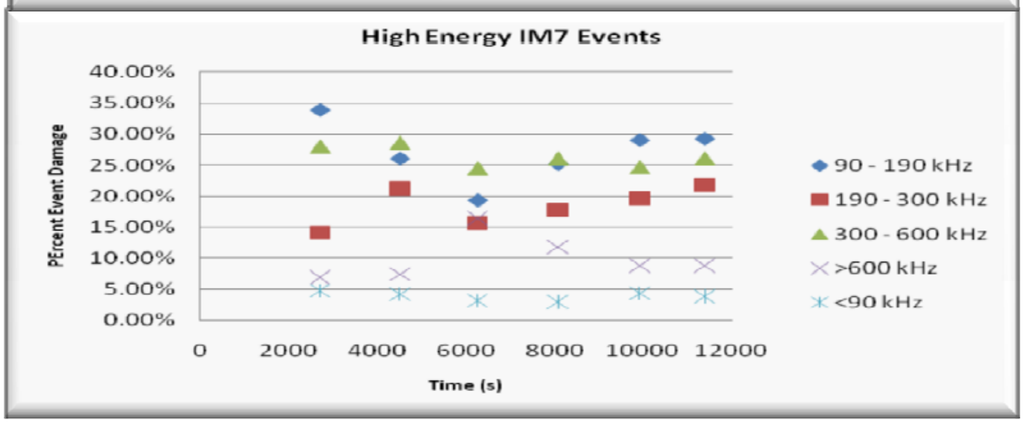
- IM-7 low *vs*. high energy events

Low Energy



E < 2 V^2-μsec

High Energy

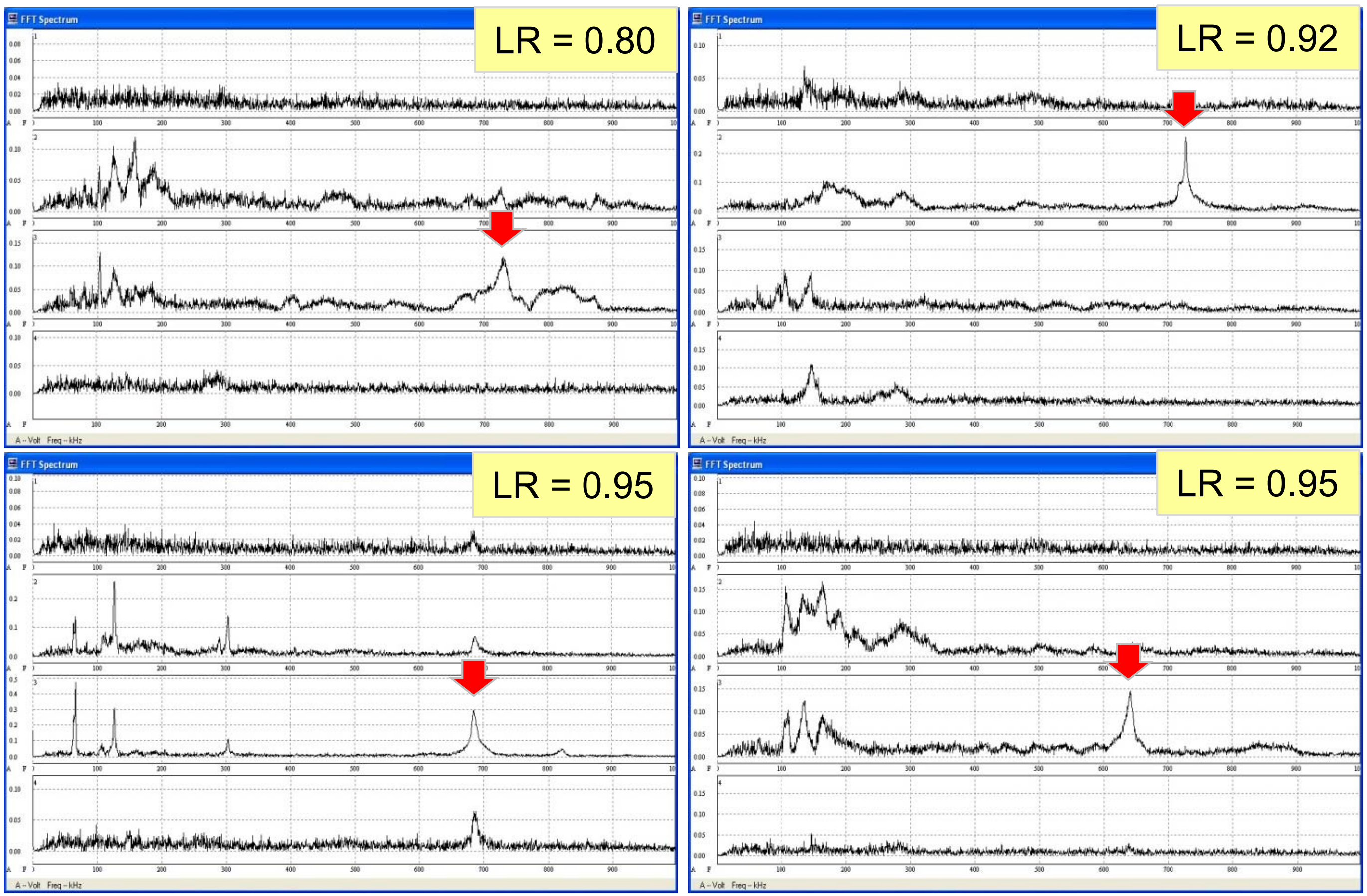


E > 2 V^2-μsec

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

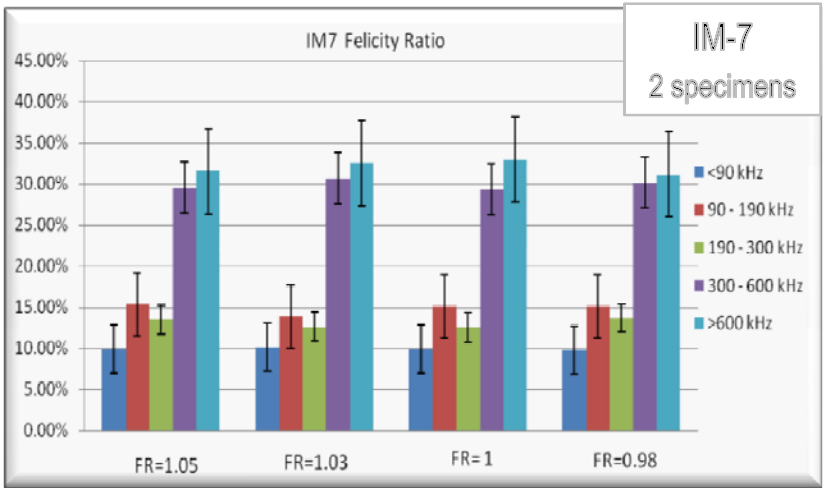
Results & Discussion

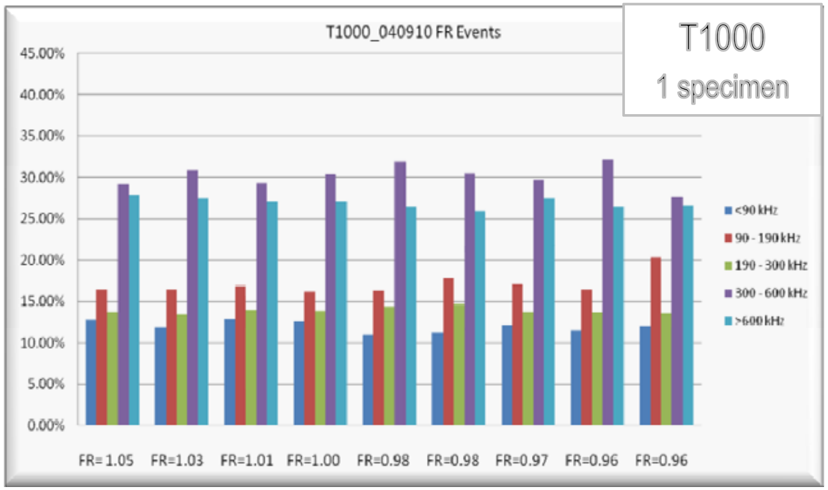
- High frequency peaks shifted downward with increasing load ratio:
  731 kHz ⇒ 728 kHz ⇒ 685 kHz ⇒ 640 kHz
- Attributed to increasing accumulated damage, hence lower modulus, causing slower stress wave propagation



Results & Discussion

- 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





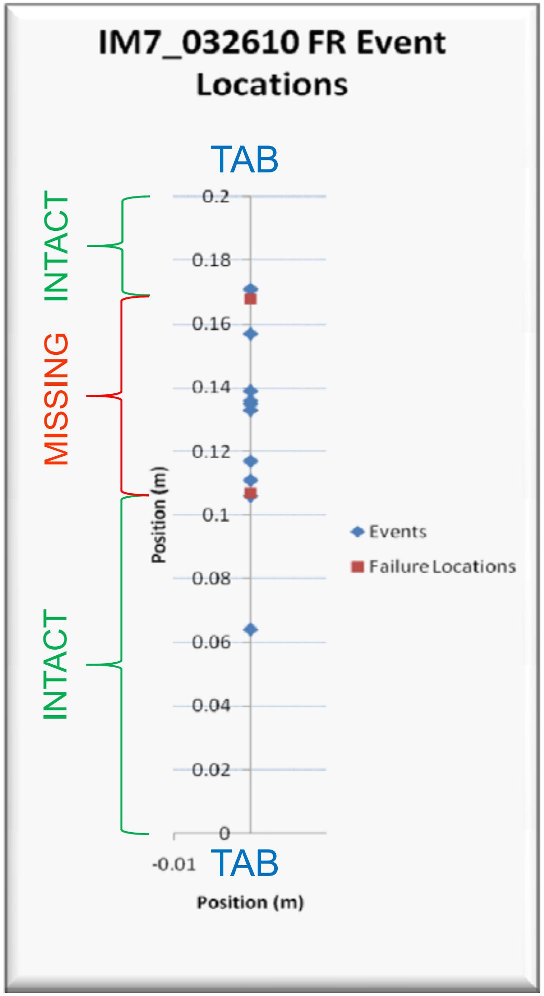
- Fiber breakage dominates FR events
  - otherwise FR events involve concerted failure for both types of C/Ep
- Some differences, but same overall trend noted for T1000 & IM-7:

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

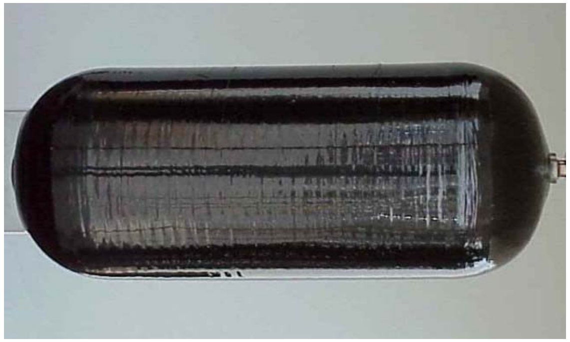


- IM7_032610 specimen had intact tow between and 0 (lower tab) and 0.115 m 0.17 and 0.20 m (upper tab)
- Tow region between 0.115 and 0.17 m obliterated (explosive failure)
- Most FR events were source located in the missing region that failed explosively

Application to Composite Overwrapped Pressure Vessels (COPVs)

Results & Discussion

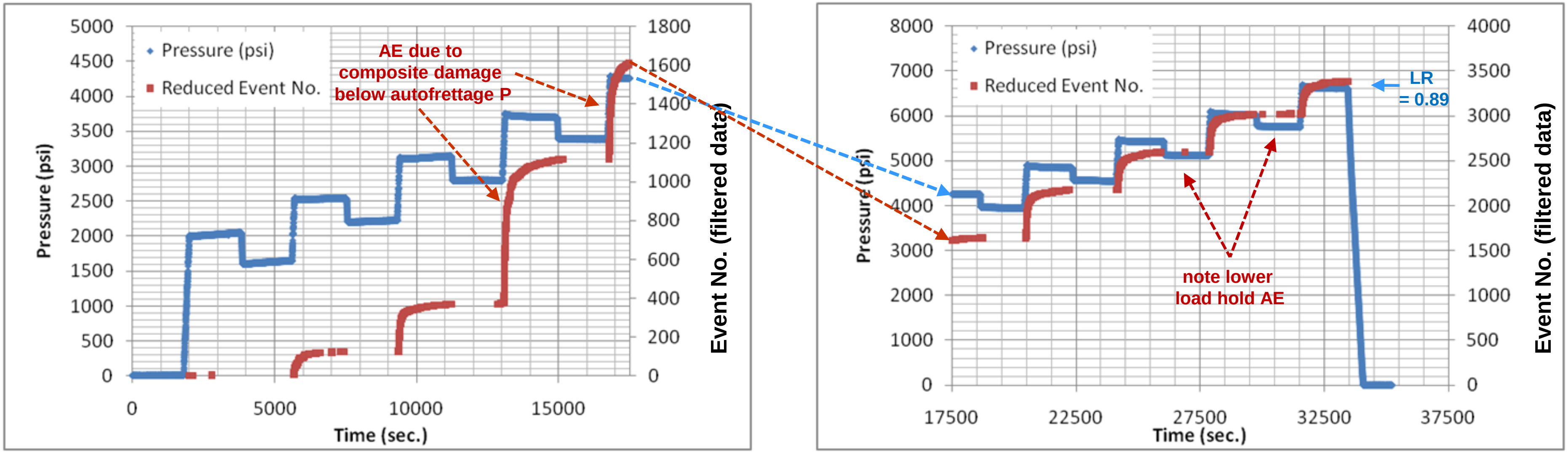
A 6.3-in. diameter IM-7 COPV was subjected to an ILH pressure schedule at LR ≈ 0.3 to 0.9



Pressure & Events vs. Time

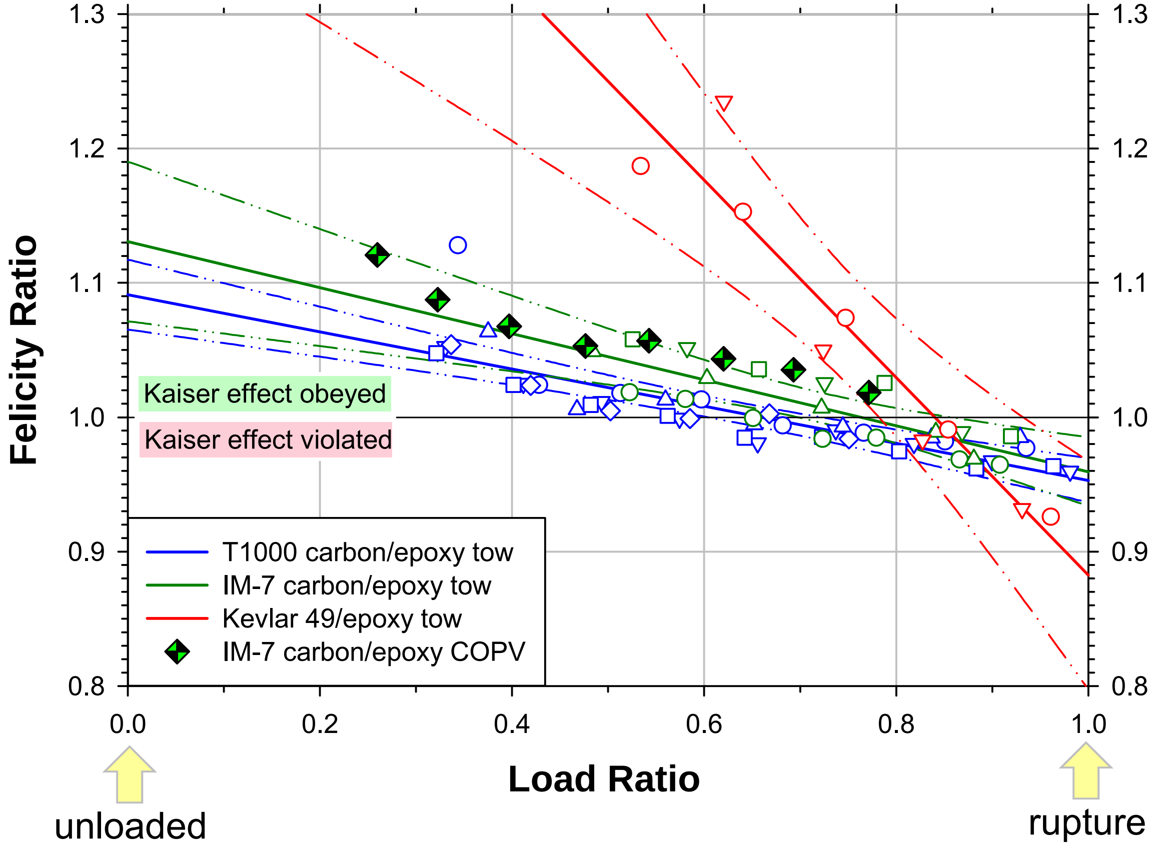
0 to 17500 s

17500 to 37500 s (cont.)



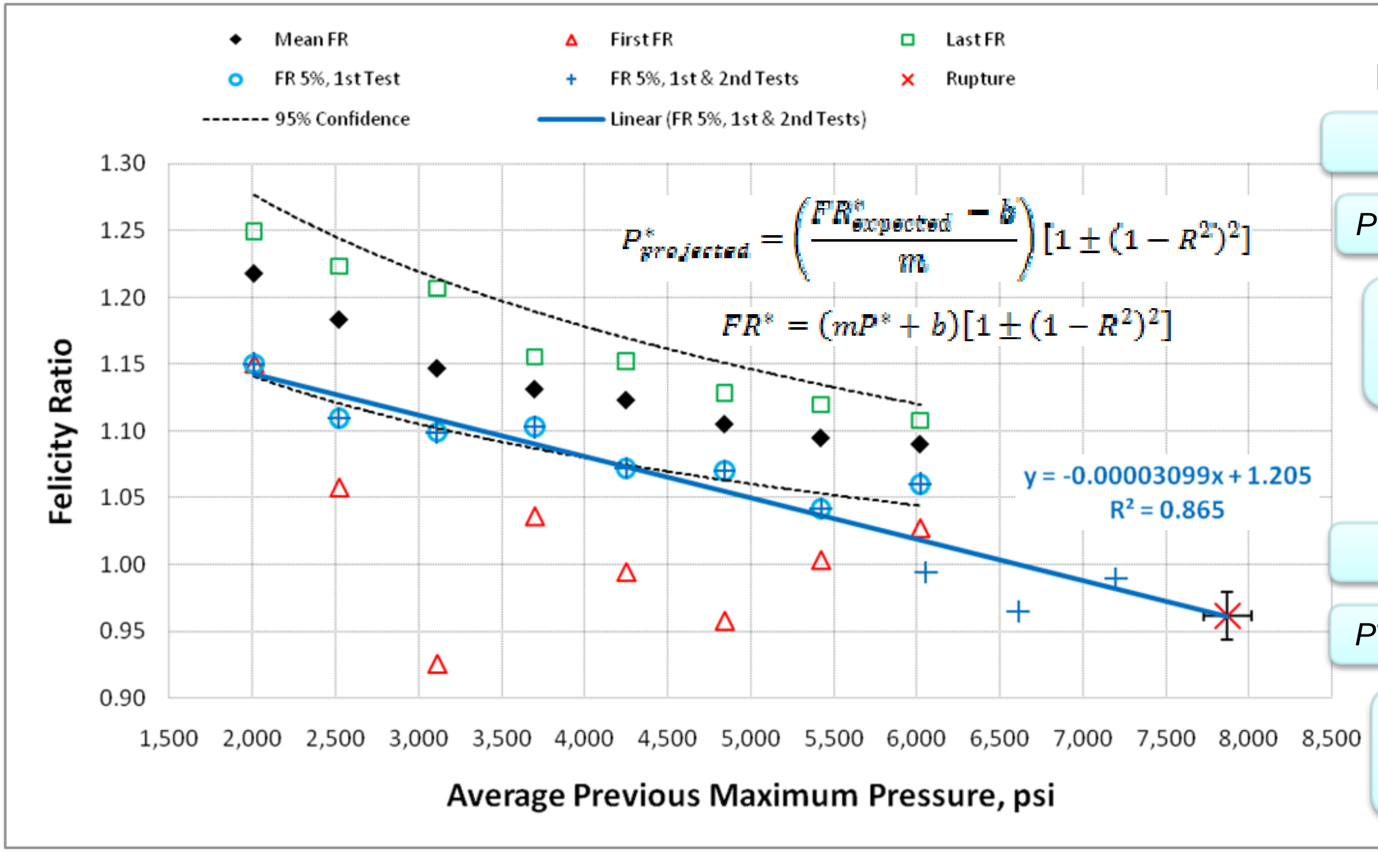
Results & Discussion

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** ± 0.010

$P^*_{projected}$ = **7,540** ± 75 psi

$P^*_{observed}$ = **7,529** psi based on hydroburst data on 2 identical COPVs

All Data:

*FR** = **0.961** ± 0.018

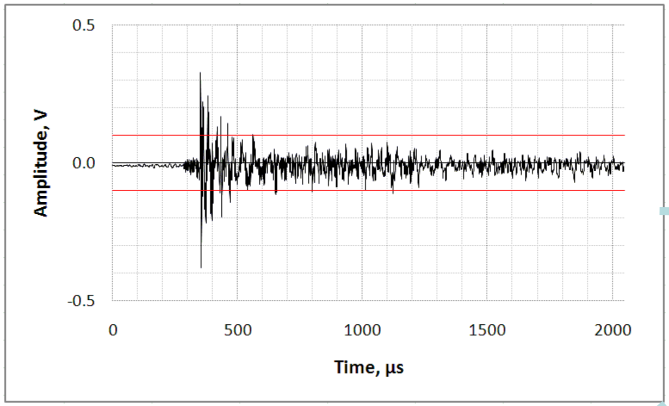
$P^*_{projected}$ = **7,870** ± 144 psi

$P^*_{observed}$ = **7,869** psi higher than expected based on hydroburst data

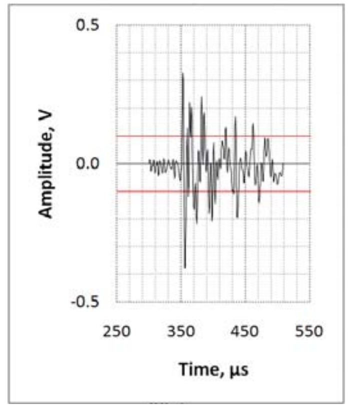
- First 8 data points predict a burst in agreement w/ burst tests result on 2 other identical COPVs (*P** = 7529 psi expected)
  - However, high initial *FR** = 1.00 (>0.96) suggests COPV may burst higher @ >7529 psi
  - Actual burst was 7870 psi

COPV AE Waveforms

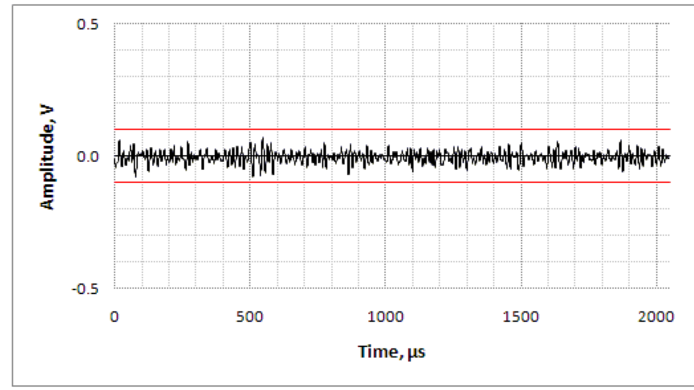
Raw Waveform



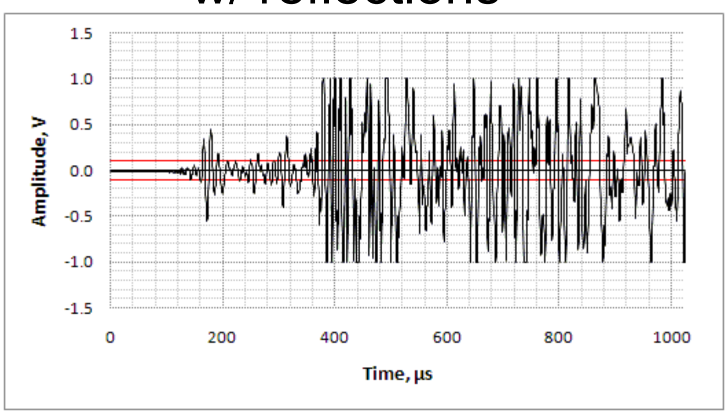
Direct Wave



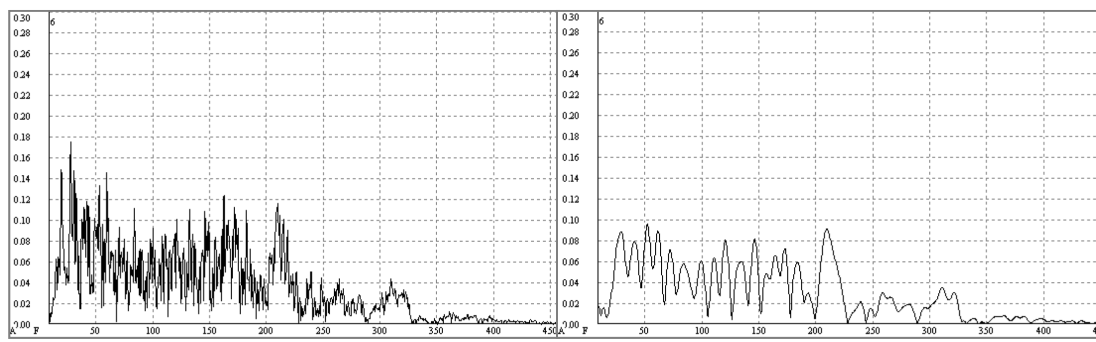
Background Noise



Raw Waveform
w/ reflections



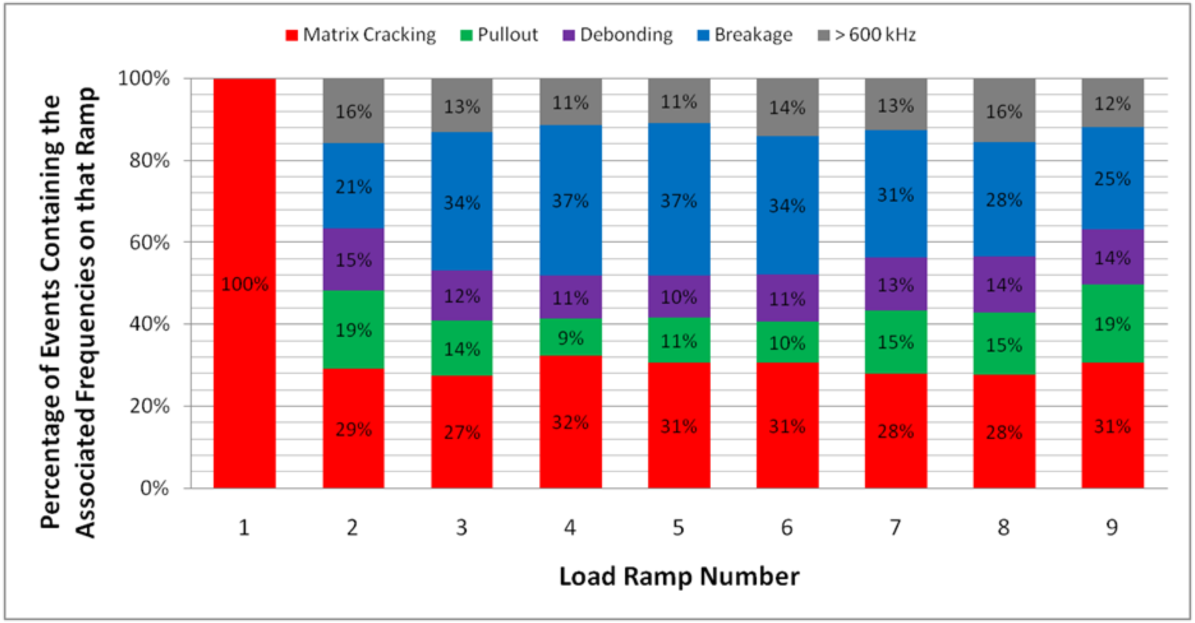
Effect of filtering *reflections*
on frequency distributions



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 $V^2\mu s$)
  - 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

COPV Manufacturers

Aerospace Primes

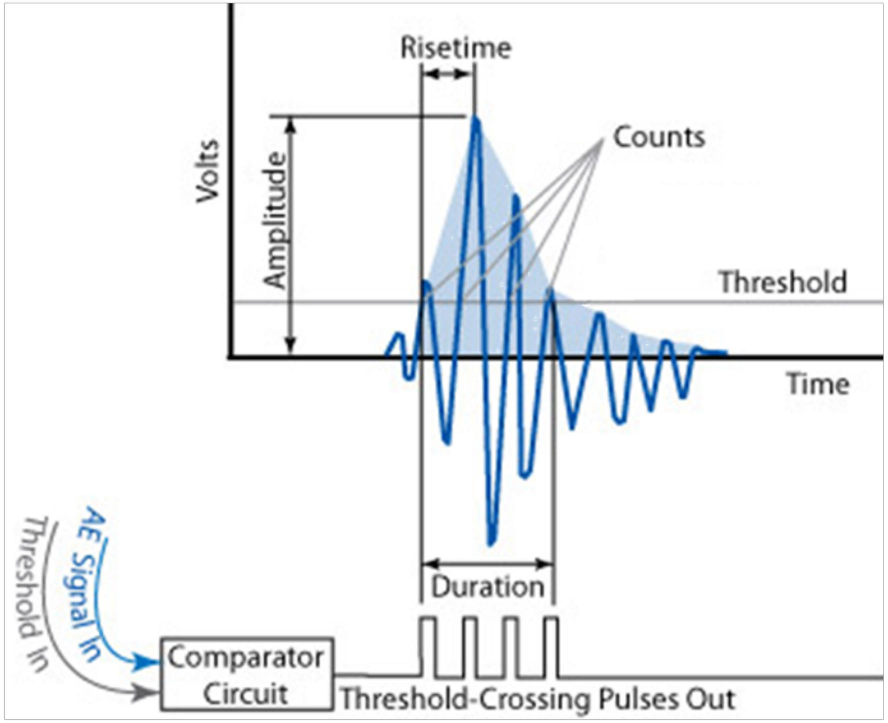
NASA (on ground and in-flight)

- 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.

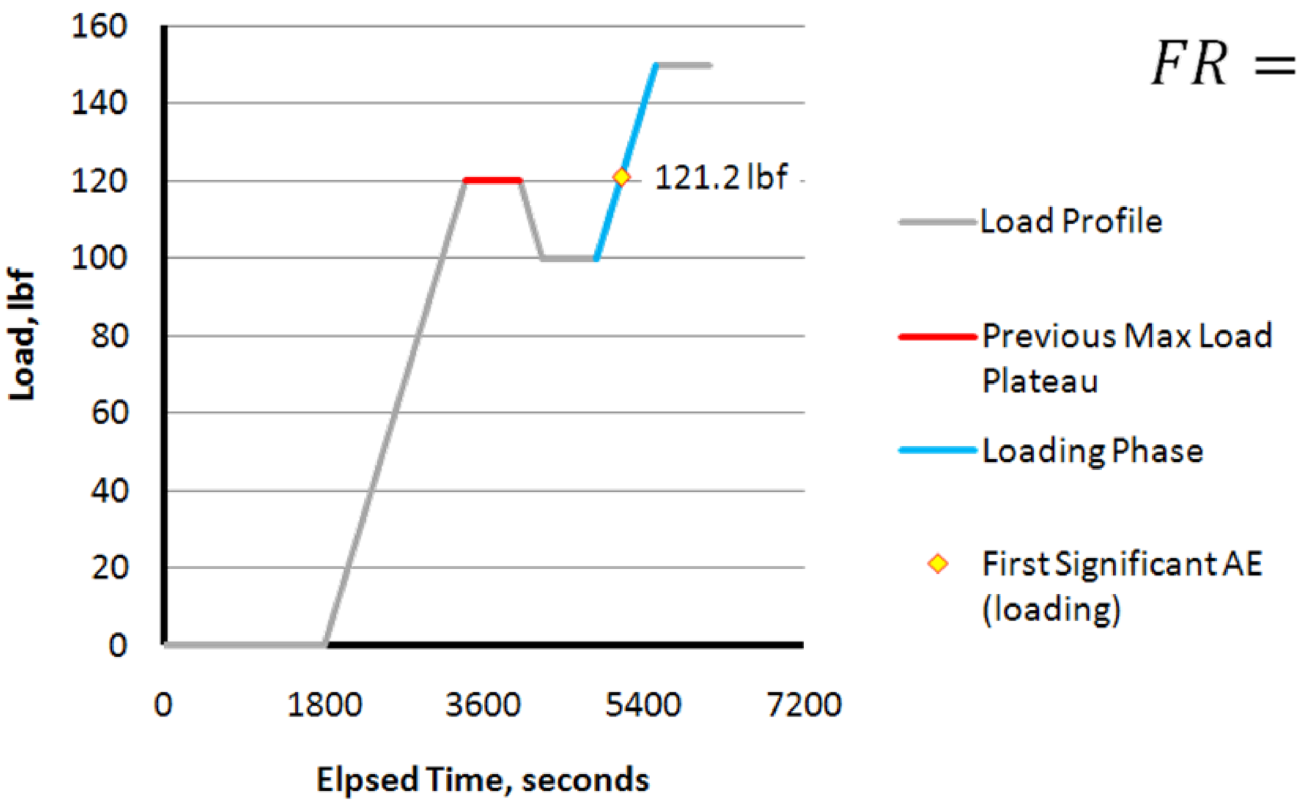
(http://www.ndt-ed.org/)



•100 mV

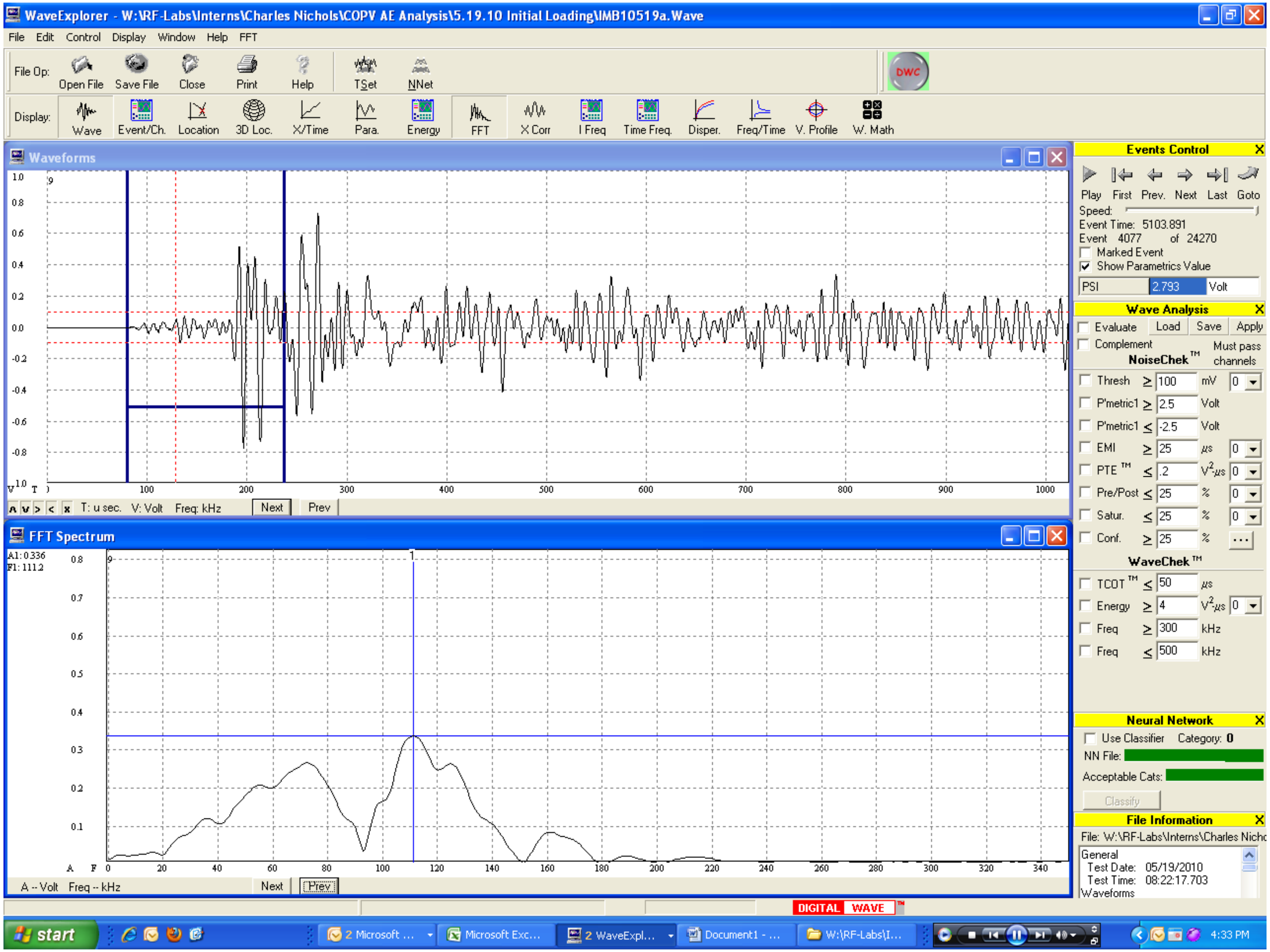
Felicity ratio (FR)

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



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

WaveExplorer™ Interface



COPV Data Acquisition Parameters

Using Digital Wave Corp. (DWC) equipment and B-1025 microdot AE sensors:

WaveExplorer Configuration		FM-1 Test Configuration	
Setup File:	COPV860	Line Driver Switch:	+V/75Ω
# of Channels:	7[1]	Preamp Gain:	18 dB
Sampling Rate:	2 MHz	Signal Gain:	12 dB
Num of Points:	4096	Signal H.P. Filter:	20 kHz
Pretrigger Points:	512	Trigger Gain:	21 dB
Voltage Range:	+/-1 V	Trigger H.P. Filter:	50 K
		Trigger L.P. Filter:	1.5 MHz
Parametric Setup Parameters		**FM-1 Lead Break Configuration[2]**	
Parametric Sampling Rate:	1Hz	Preamp Gain:	24 dB
Parametric Voltage Range:	+/-10 V	Signal Gain:	12 dB
Total Parametric Channels:	1	Trigger Gain:	18 dB

[1] The first channel was defective, so channels 2-7 were used.

[2] 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:

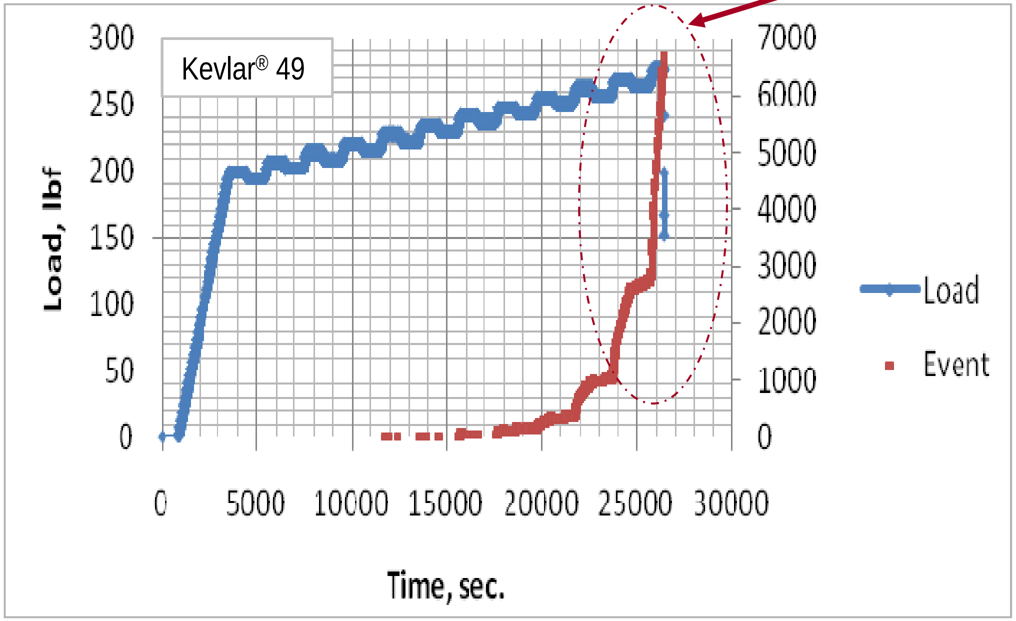
Filter	Data Reduction Rationale and Associated Restrictions
Mitigate Reflections	Define the time centered on threshold tolerance low enough to remove most of the reflections by observing the typical flexural wave duration: *Threshold ≥ 100 mV (88 dB)* *TCOT™ ≤ 125 μs*
Enforce Frequency Restrictions	Optional – 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 ($\leq 5\ lb_f$)
      - Events with an average energy above this value were considered significant

Results & Discussion

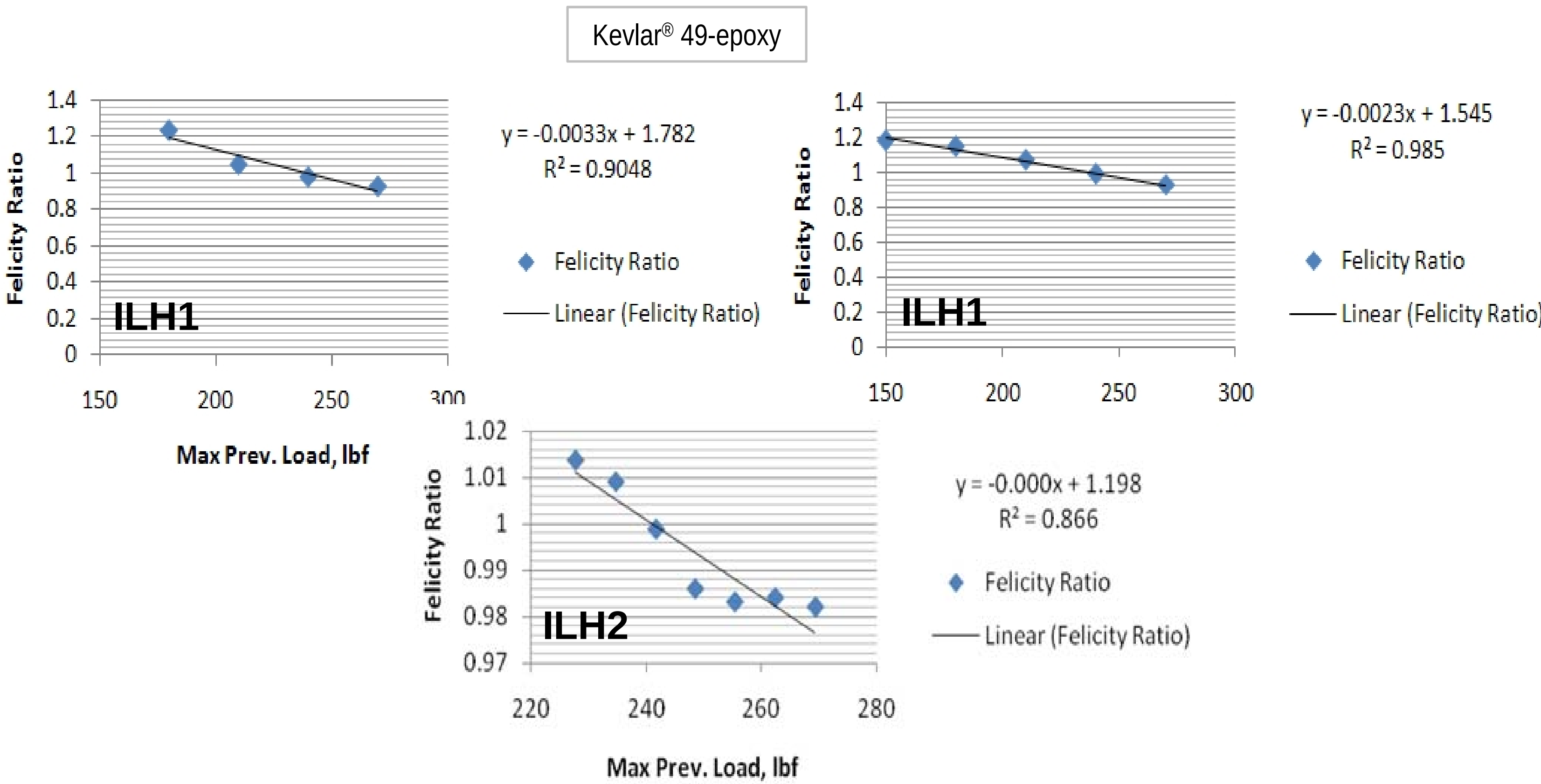
- Characteristics of significant AE:
  - For Kevlar-epoxy, and T1000 and IM-7 carbon-epoxy, nonlinear increases in AE event rate were observed immediately before rupture, indicative of '*critically intense*' AE activity per ASTM E 1067 and E 1118:



  - Areas of critically intense AE activity also showed greatest violation of Kaiser effect, hence, the lowest FR values

Results & Discussion

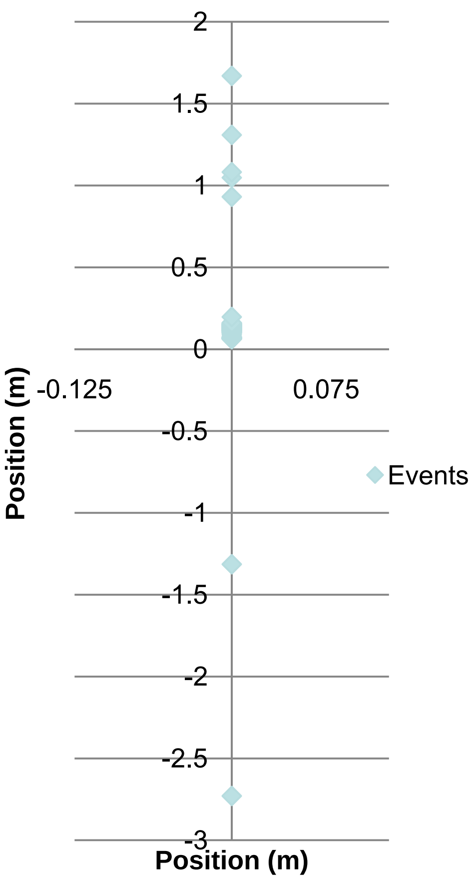
- For Kevlar-epoxy 4650 denier tow, correlation coefficients for ILH1 & 2 methods indicated good (R^2 = 0.866) to excellent (R^2 = 0.985) agreement:



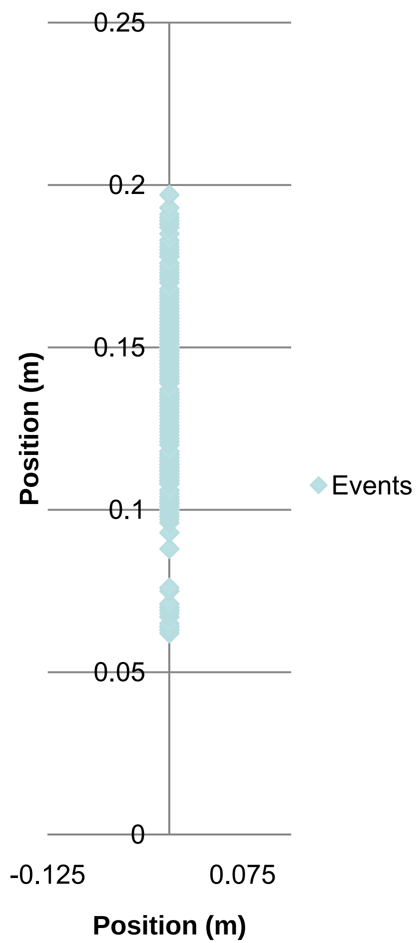
Results & Discussion

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