Developing Novel Acoustic Emission Procedures for Failure Prediction of Carbon-Epoxy COPVs and Related Composite Materials

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Session 5:

Non-Destructive Evaluation (Health Monitoring)

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Background

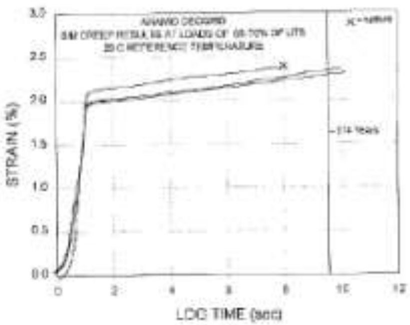
Issues:

- 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 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

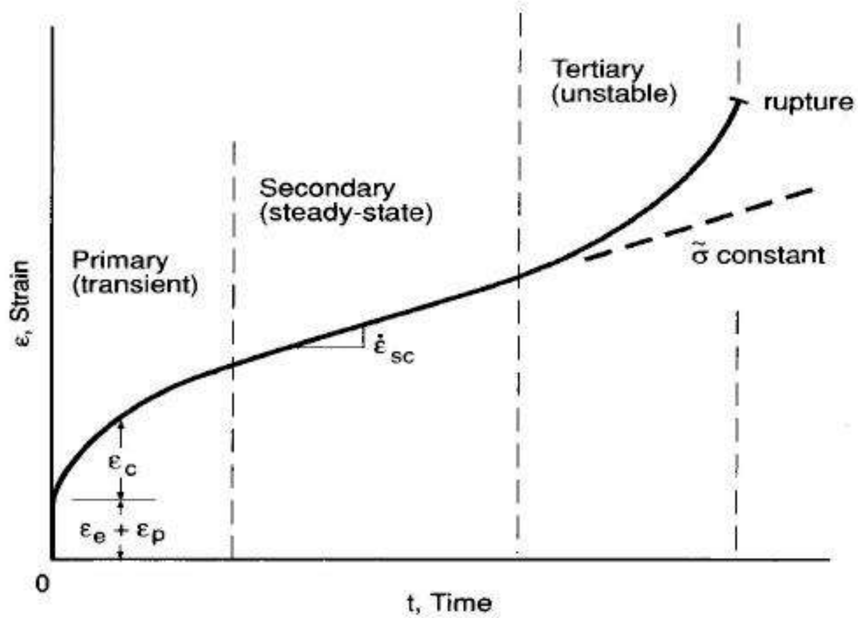
[1] *SR defined by AIAA Aerospace Pressure Vessels Standards Working Group as "the minimum time during which the composite maintains structural integrity considering the combined effects of stress level(s), time at stress level(s), and associated environment."*

Strain vs. Time Behavior During Creep

Classical Case



carbon or PPTA (Kevlar®)



BBL rupture of a COPV

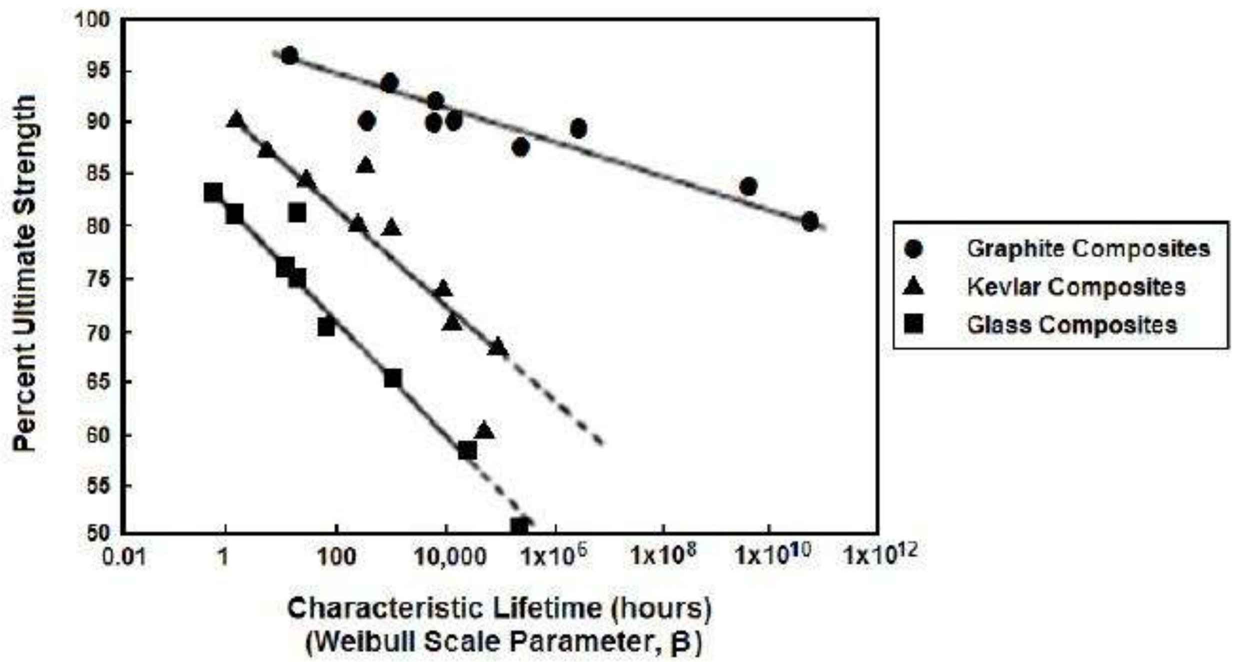
distinct tertiary creep phase

(ductility observed before rupture)

The problem with advanced fibers such as carbon or Kevlar® 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 monitoring damage accumulation in composites in general
- 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 fail COPVs at a critical *FR (FR**) below 1.0, indicative of severe accumulated damage
    - Could also fail COPVs at a known levels cumulative of fiber breakage or matrix cracking

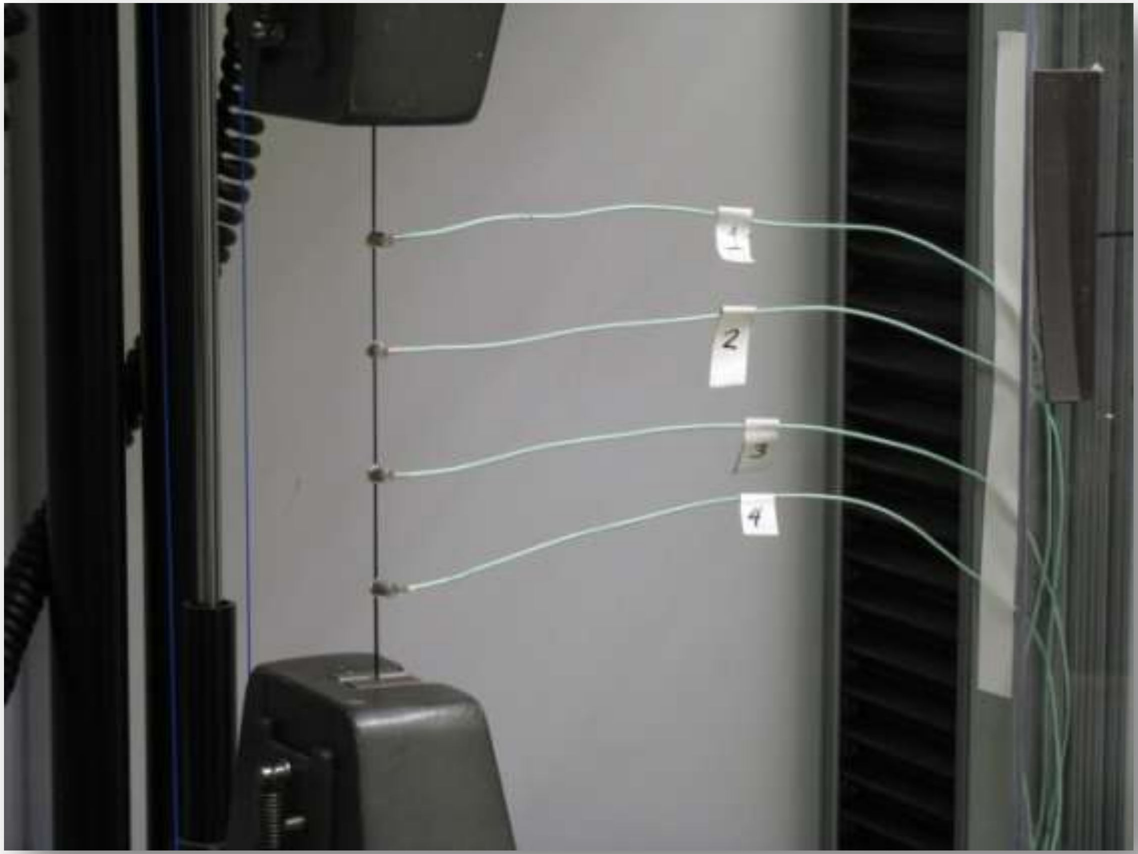
Experimental

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)

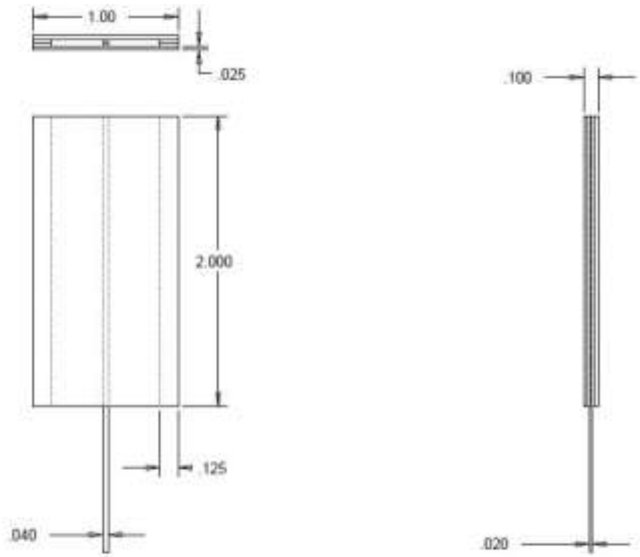
Experimental

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

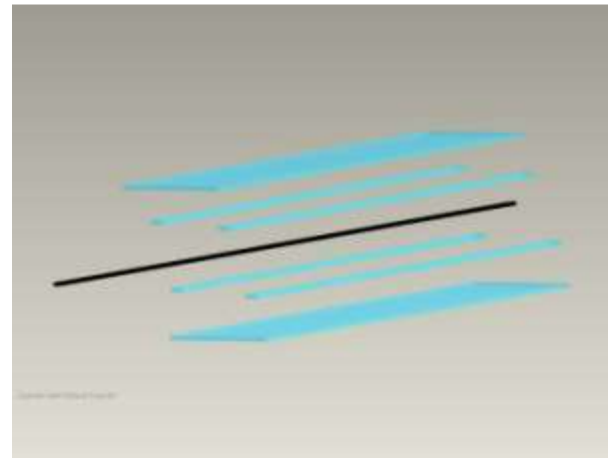
Tabbing: shear strength of epoxy and bonded grip length important variables[§]

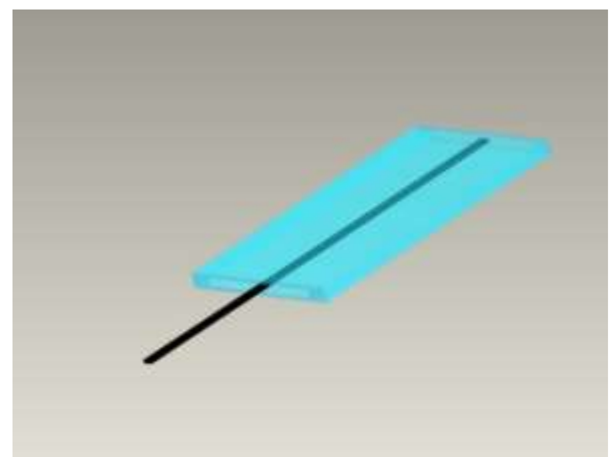


$$L_{min} = F^{tu}h/2F^{su}$$

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].





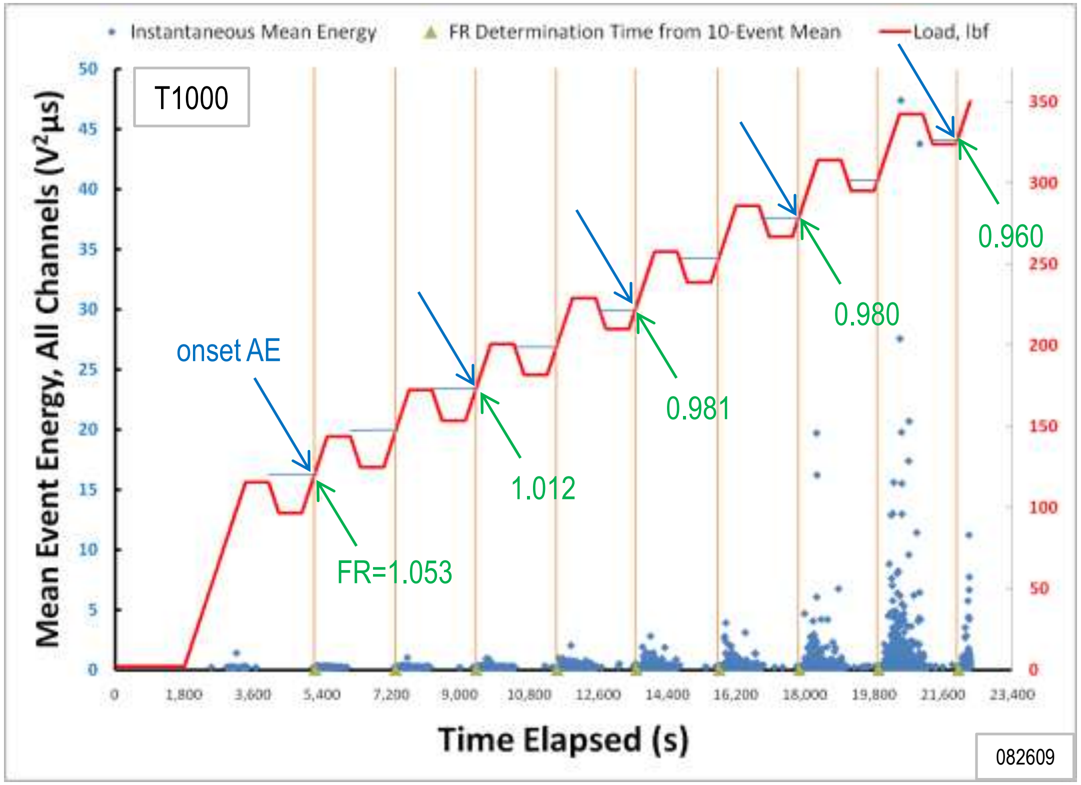
§ ASTM D 2343, *Test Method for Tensile Properties of Glass Fiber Strands, Yarns, and Rovings Used in Reinforced Plastics,* American Society for Testing and Materials, West Conshohocken, PA (2008).
ASTM D 3039, *Test Method for Determining Tensile Properties of Polymer Matrix Composite Materials*, American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

Felicity Ratio Analysis

(IM7 & T1000 composite tow)

Experimental

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

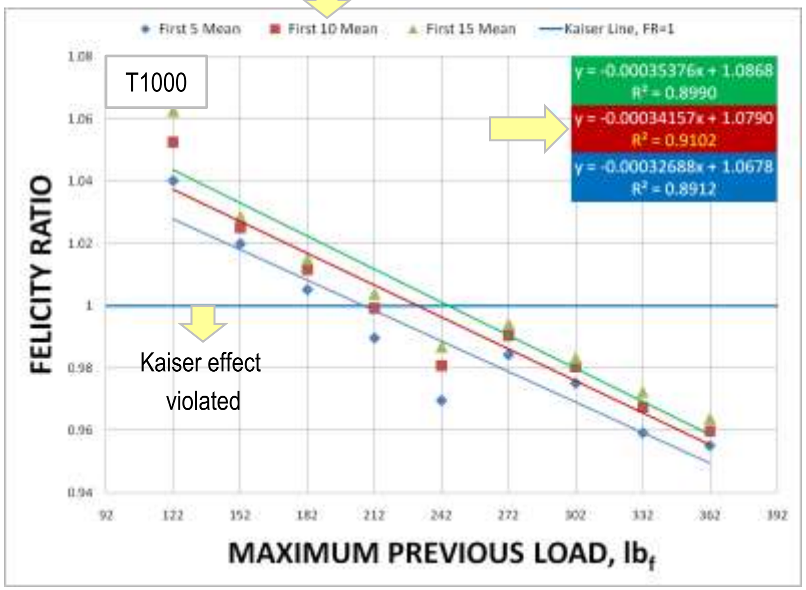


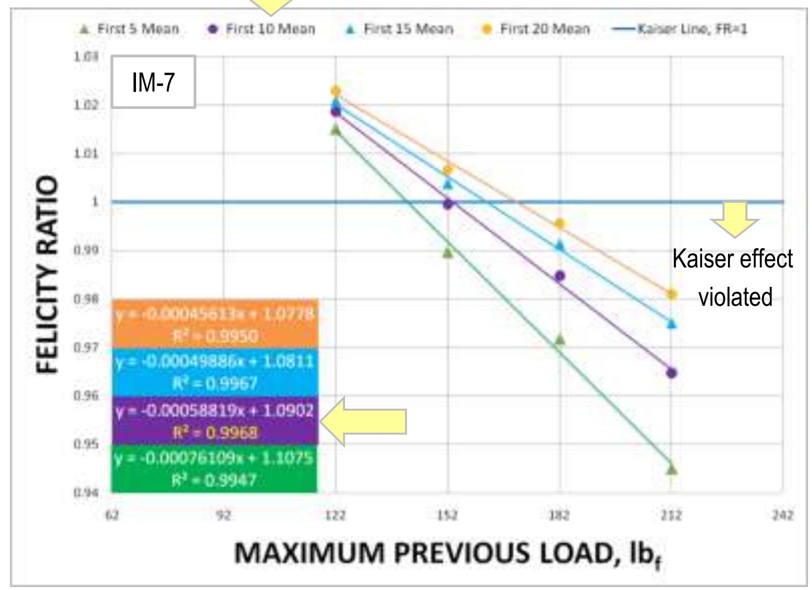
- ILH profile is based on the pressure tank examination procedure described in ASTM E 1067 §

§ ASTM E1067, *Practice for Acoustic Emission Examination of Fiberglass Reinforced Plastic Resin (FRP) Tanks/Vessels*, American Society for Testing and Materials, West Conshohocken, PA, 19428- (2011).

Results & Discussion
composite tow

- 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

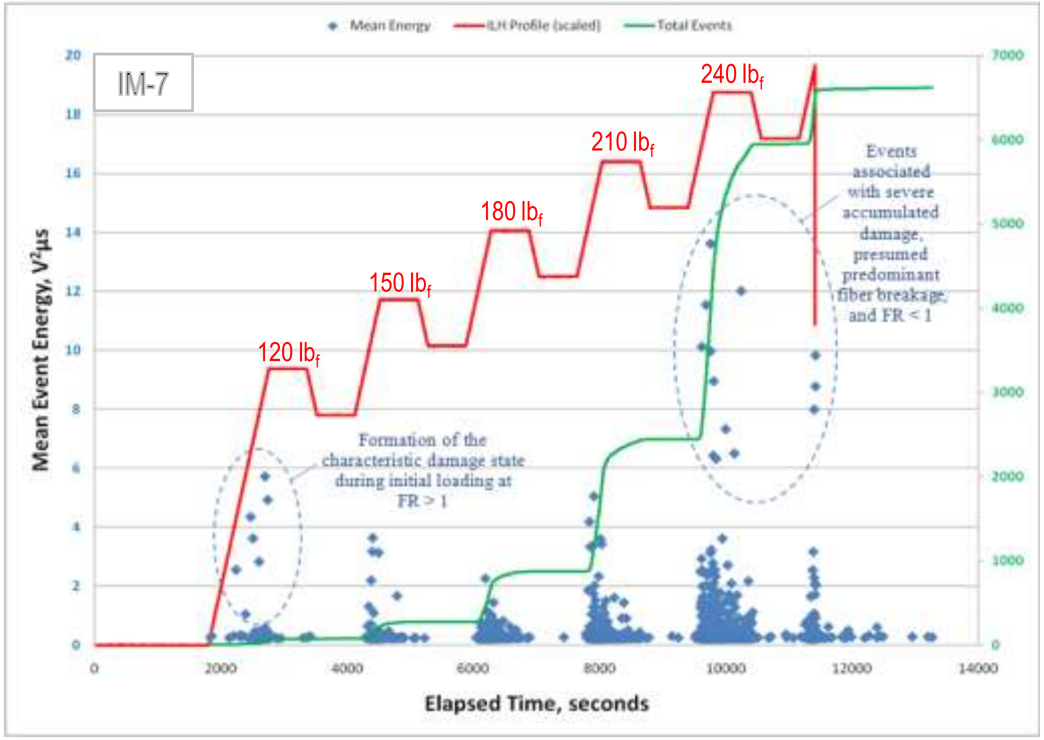




- For same material and averaging method, the slope of least squares fit is indicative of damage tolerance
  - Flatter slopes correspond to good damage tolerance (in-character behavior)
  - Steep slopes correspond to low damage tolerance (out-of-character behavior)
- Kaiser effect violated at FR<1 ⇒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

Results & Discussion
composite tow

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

Results & Discussion

composite tow

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

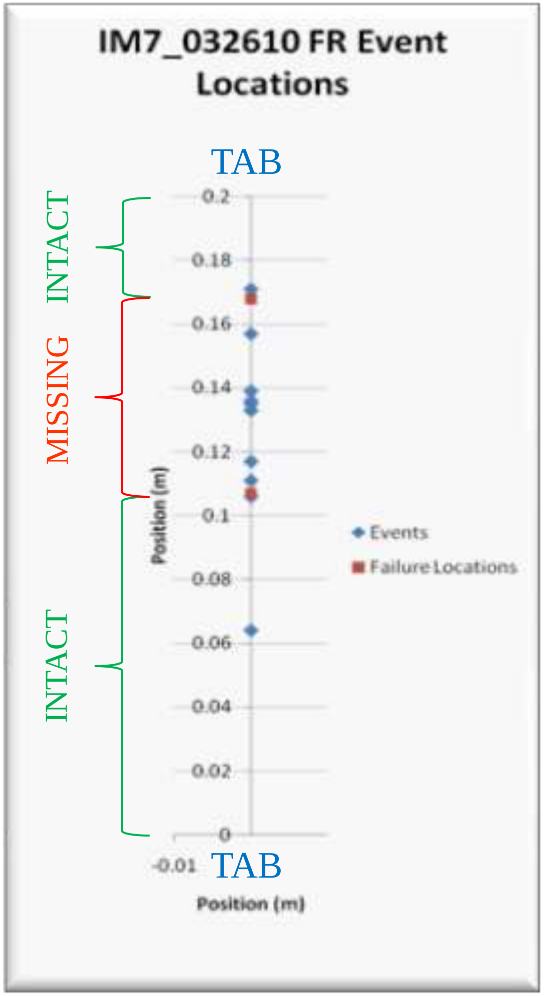
- **Let *FR** = extrapolated *FR* at rupture predicted by the least squares fit**
- ***FR** behaves like a universal parameter that varies less than the UTS**

[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

composite tow

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

Conclusions
composite tow

- 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

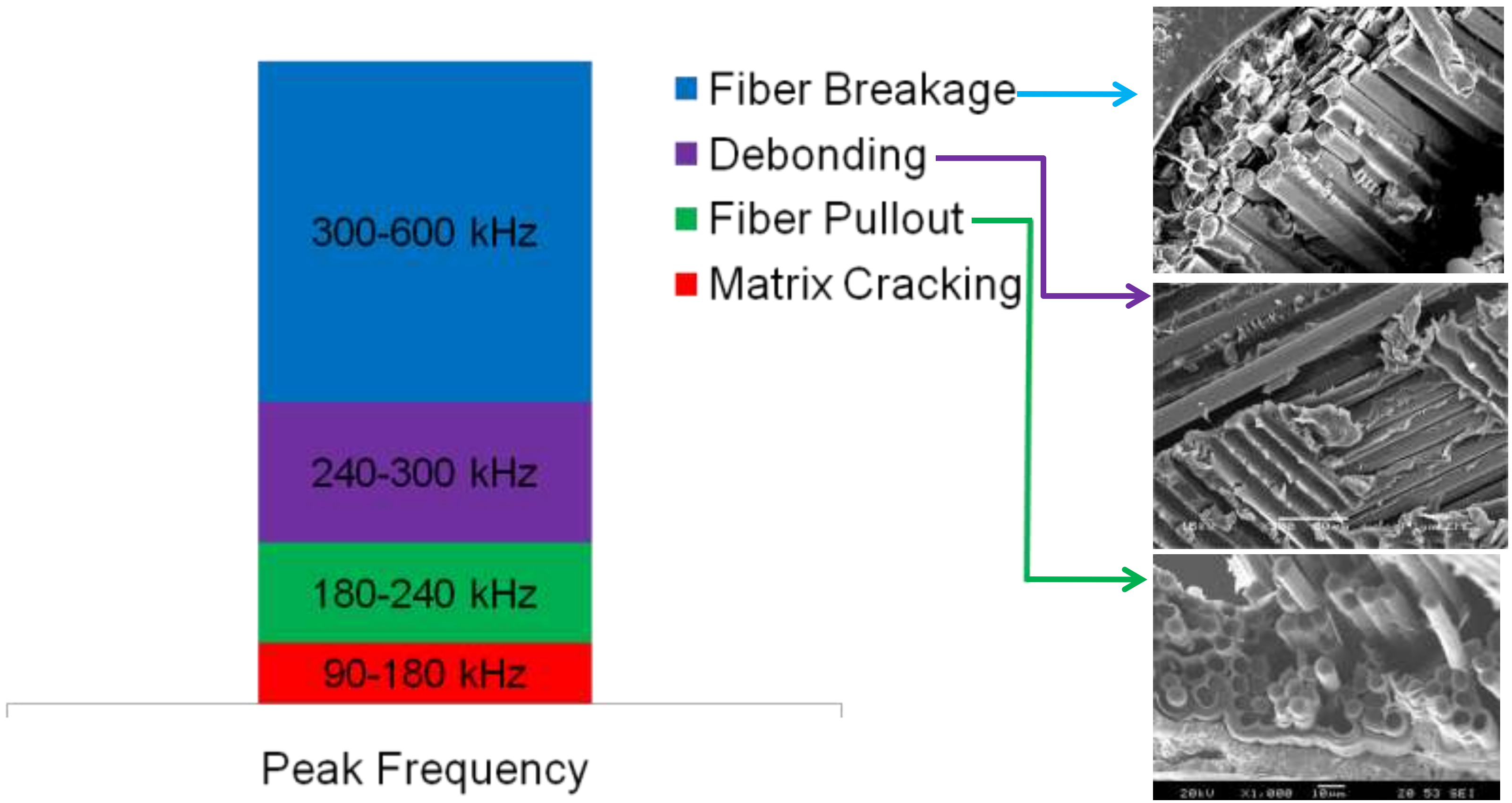
§ ASTM E1067, *Practice for Acoustic Emission Examination of Fiberglass Reinforced Plastic Resin (FRP) Tanks/Vessels*, American Society for Testing and Materials, West Conshohocken, PA, 19428 (2011).

Waveform and FFT Analysis

(IM7 & T1000 composite tow)

Results & Discussion
Waveform and FFT Analysis

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



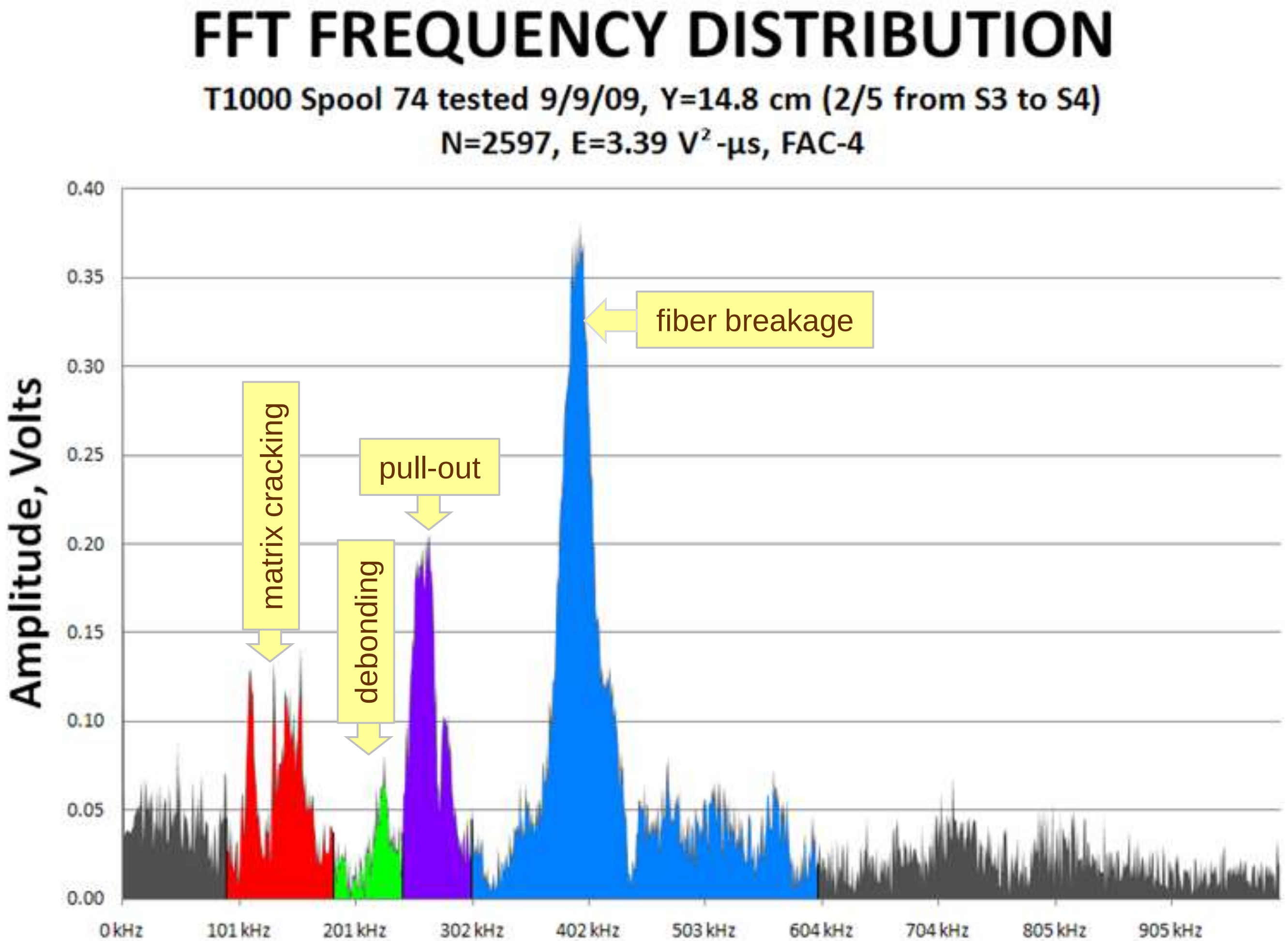
§ De Groot, P., P. Wijnen, and R. Janssen, “Real-time Frequency Determination of Acoustic Emission for Different Fracture Mechanisms in Carbon/Epoxy Composites,” *Composites Sci. Technol.*, **55**, pp. 405-421 (1995).

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

Results & Discussion

Waveform and FFT Analysis

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

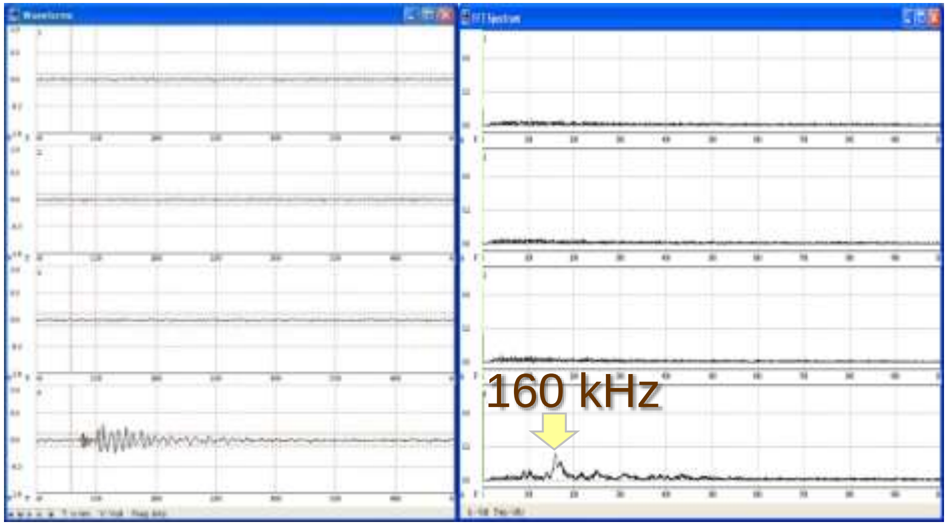


Results & Discussion

Waveform and FFT Analysis

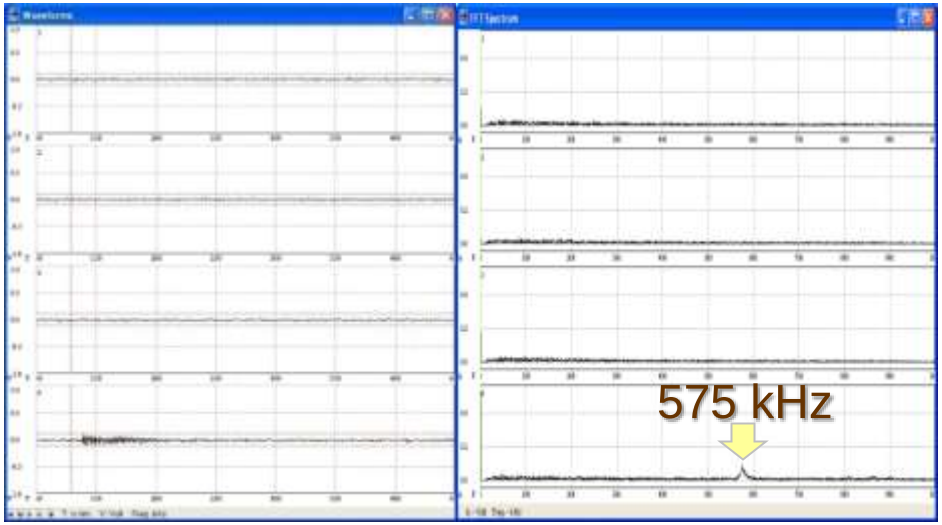
- In general, three different waveforms were observed for C/Ep

1. Matrix Cracking



waveform FFT

2. Fiber Breakage

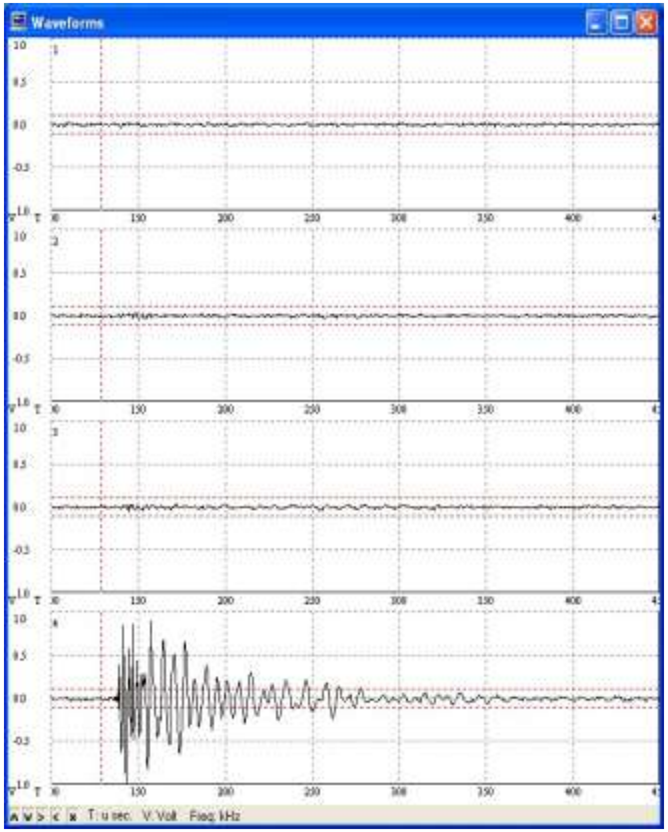


waveform FFT

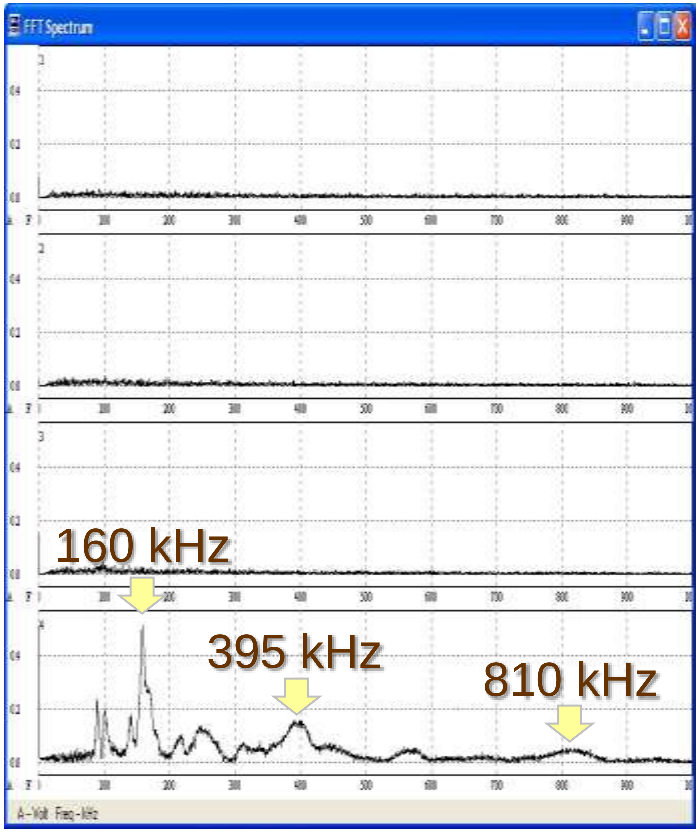
Results & Discussion
Waveform and FFT Analysis

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

3. Concerted, mixed mode failure



waveform

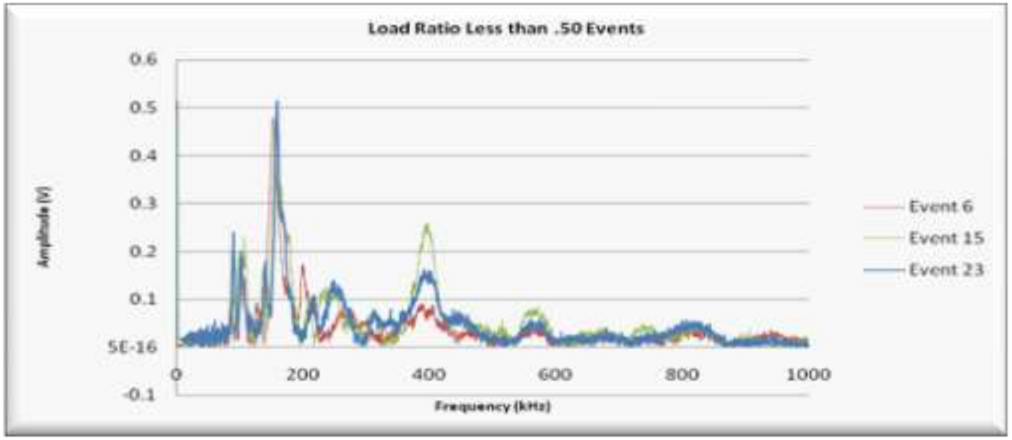


FFT

Results & Discussion
Waveform and FFT Analysis

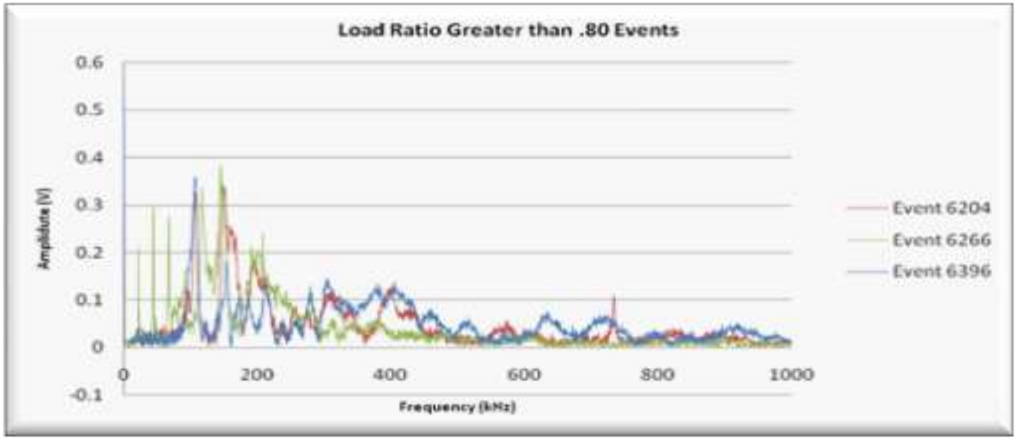
- IM-7 early *vs*. late life events

Early Life



LR < 0.5
first ILH ramp up

Late Life

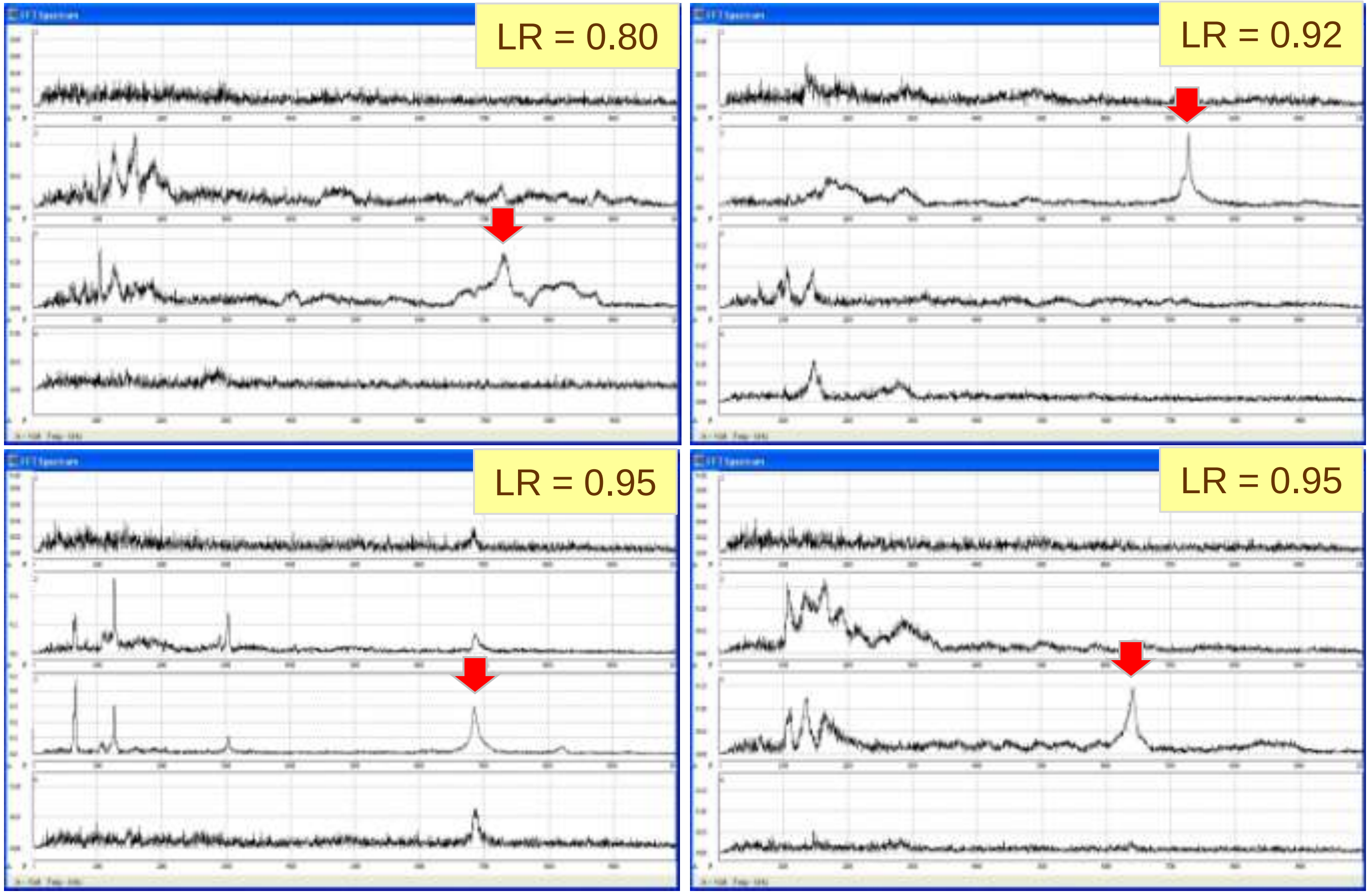


LR > 0.8
last ILH ramp up

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

Results & Discussion
Waveform and FFT Analysis

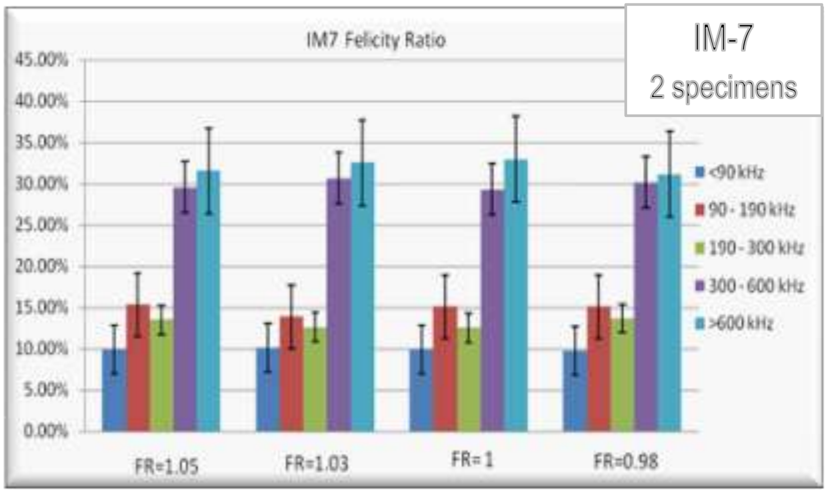
- High frequency peaks shifted downward with increasing load ratio:
  731 kHz ⇒ 728 kHz ⇒ 685 kHz ⇒ 640 kHz
- Attributed to increasing accumulated damage, hence lower modulus, causing slower stress wave propagation

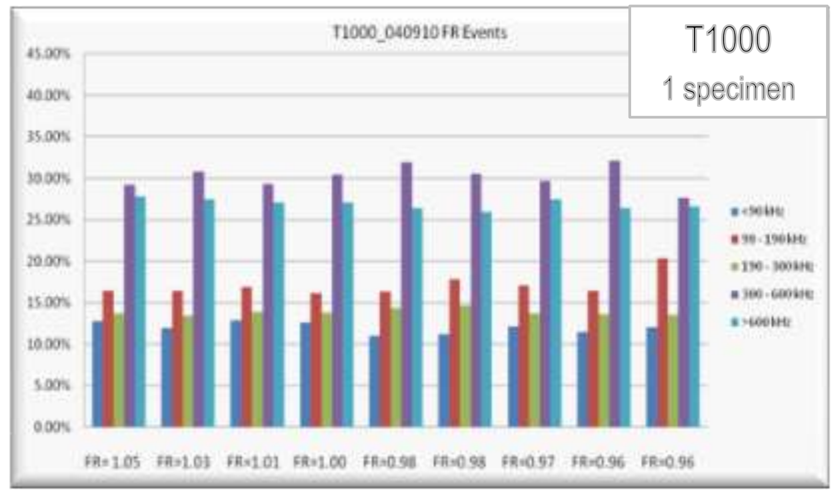


Results & Discussion

Waveform and FFT Analysis

- 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*)

Data Reduction

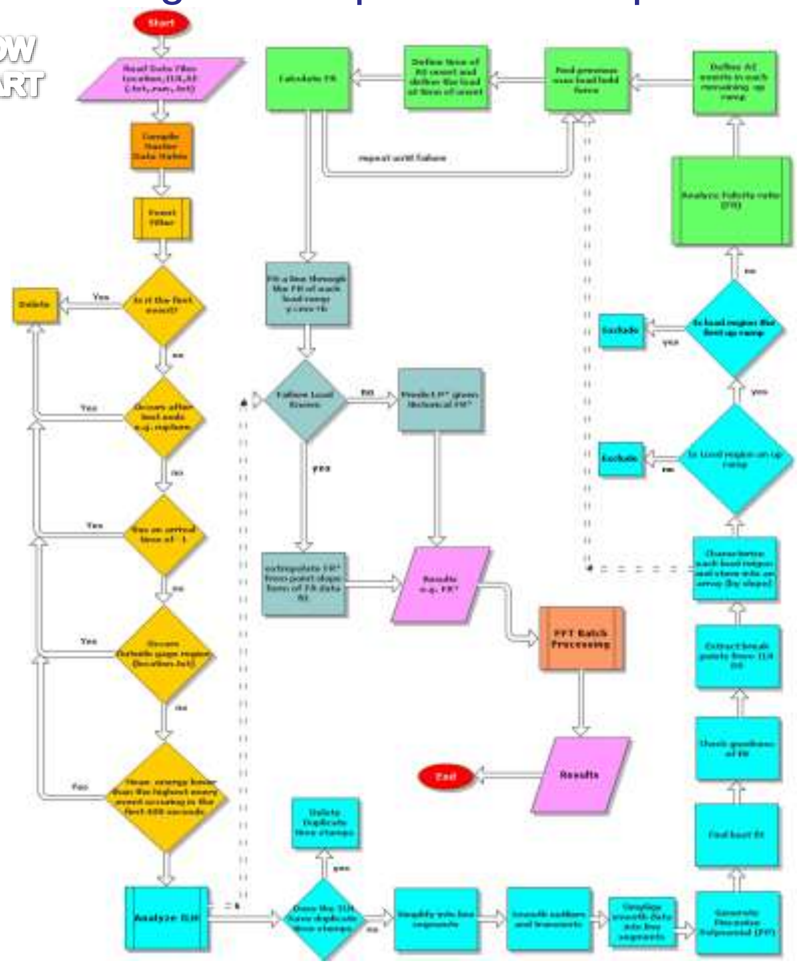
Enhancements

Results & Discussion

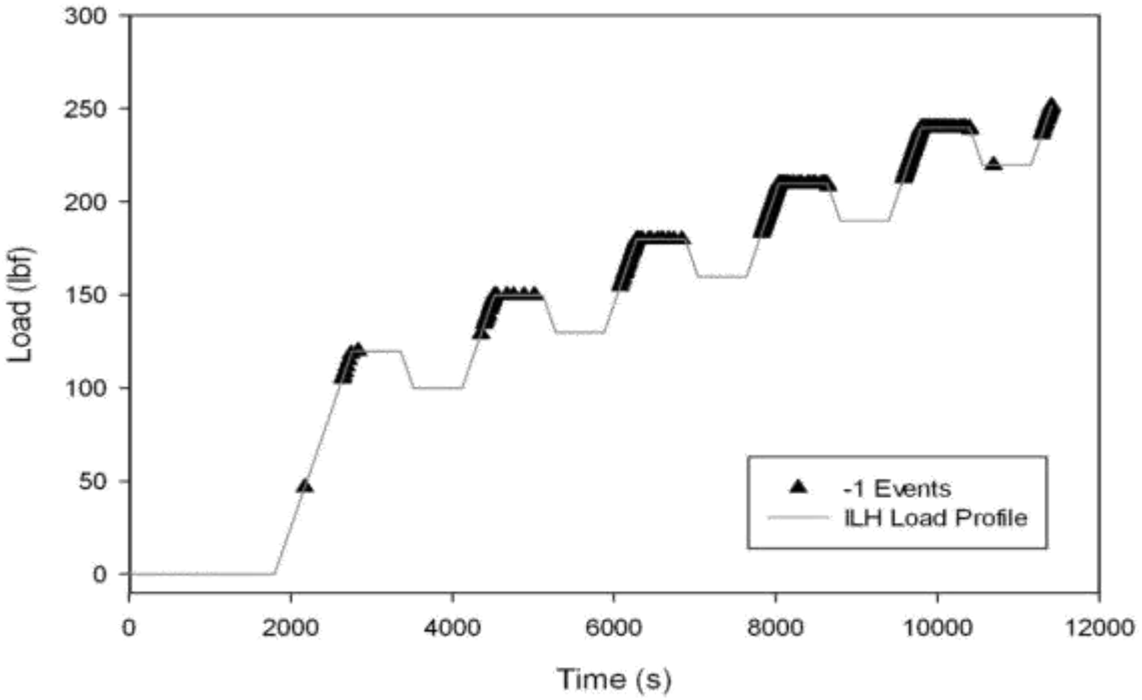
Data Reduction Enhancements

- Felicity Ratio Analysis Tool (FRAT) written
  - Automate AE data reduction
  - Optimizes best fit using least squares or bisquare fitting

FLOW CHART



Results & Discussion
Data Reduction Enhancements

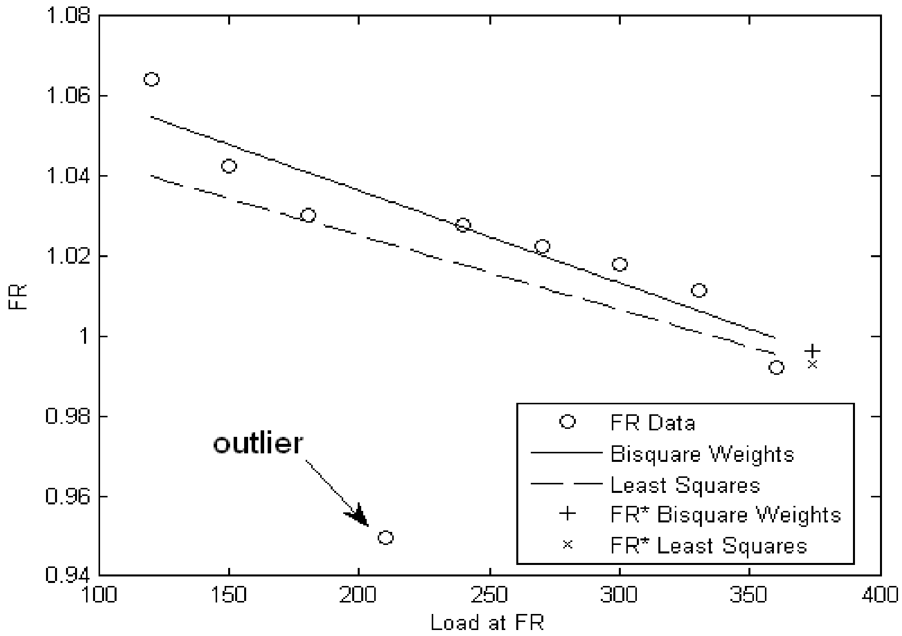


- Filtering Criteria
  - -1 arrival channel events were plotted against the load profile
  - -1 events primarily occurred on loading & upper load holds
  - No grounds for rejecting lower energy events

Results & Discussion

Data Reduction Enhancements

- Linear Least Squares (LLS)
  - Gives outliers too much leverage over fit.
  - Must manually remove outliers
  - Minimizes square of residuals
- Bisquare weighting
  - Very similar process to LLS
  - Weights residuals of each point and down weights points of high leverage.
    - Automatically remove outliers
  - Minimizes weighted square of residuals

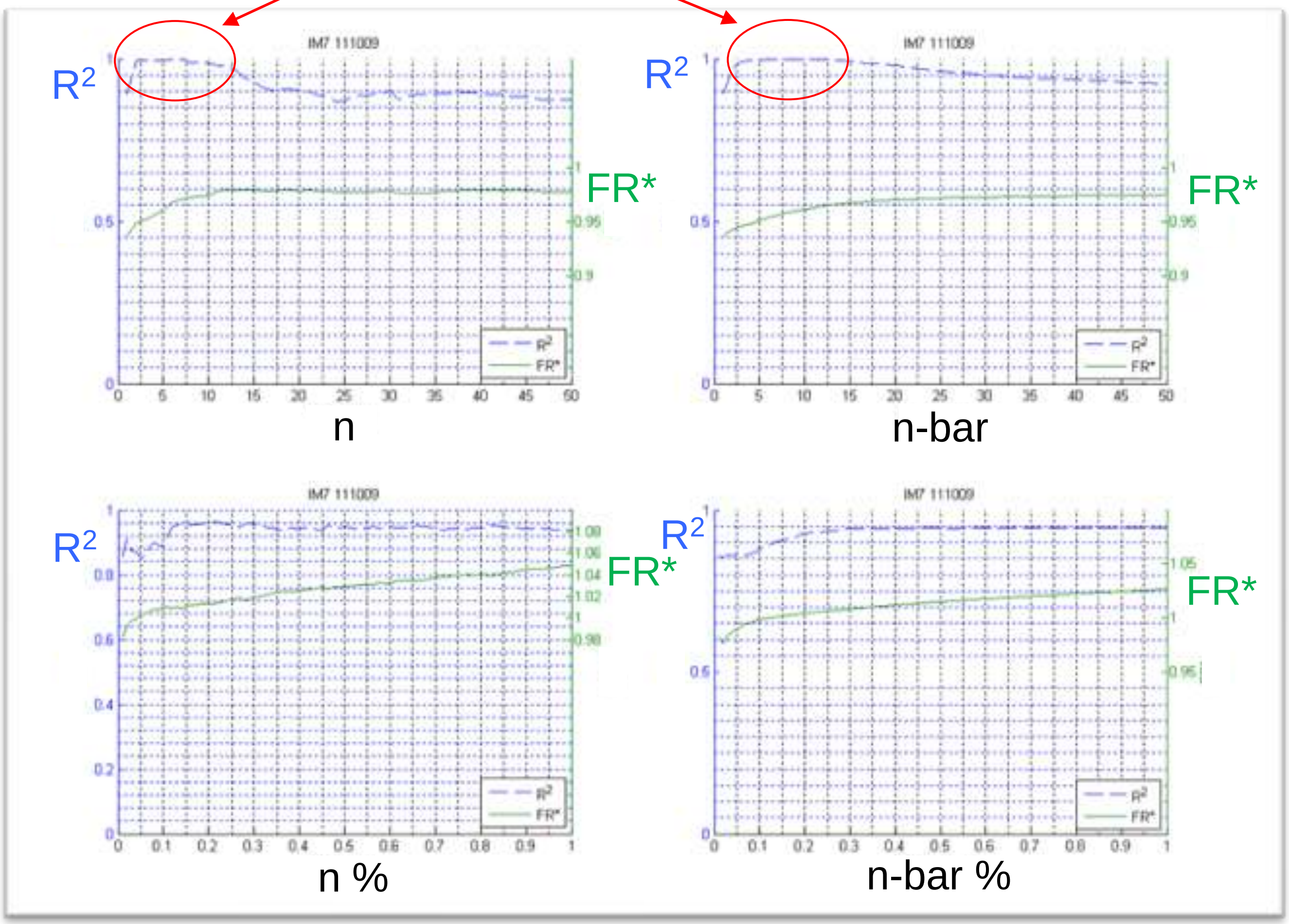


Results & Discussion

Data Reduction Enhancements

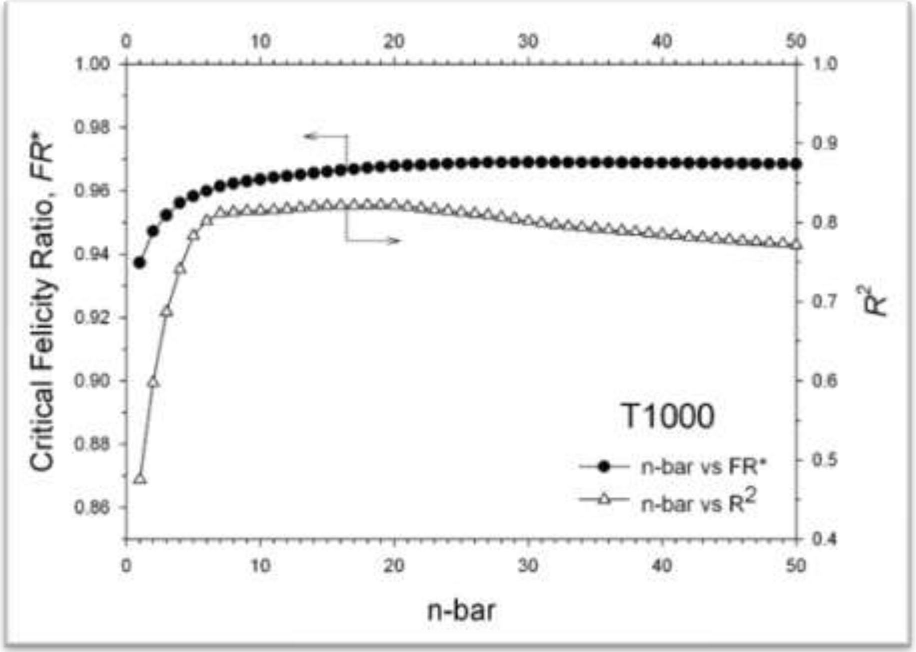
- Can optimize the linear fit for different methods for determining the onset of significant AE used to calculate the FR:

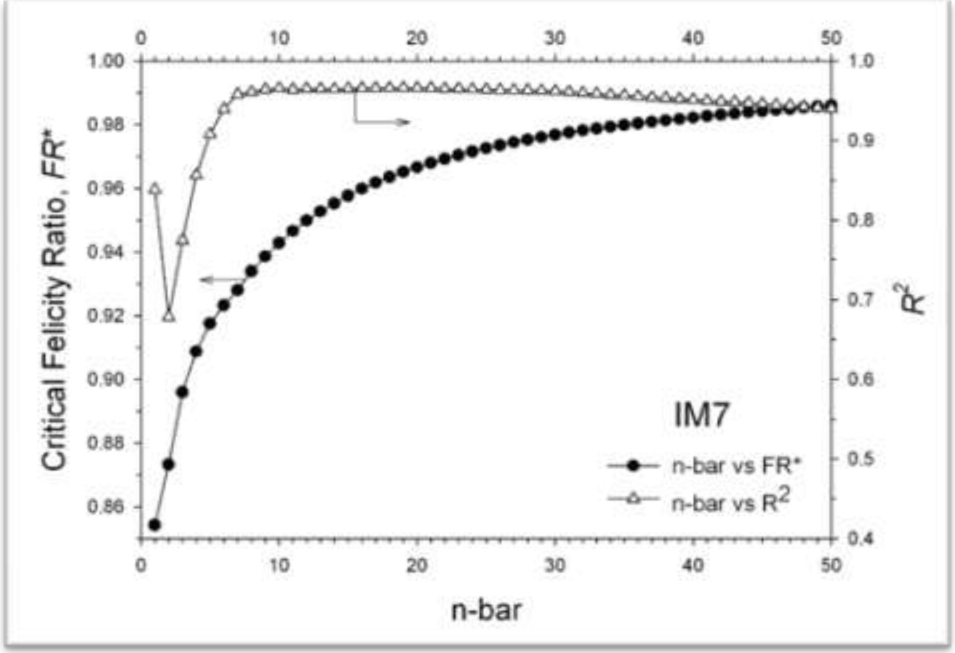
best linear fits



Results & Discussion
Data Reduction Enhancements

- Comparison of IM7 and T1000 tow showing the variation of the R^2 (coefficient of determination) and FR* with *n*:



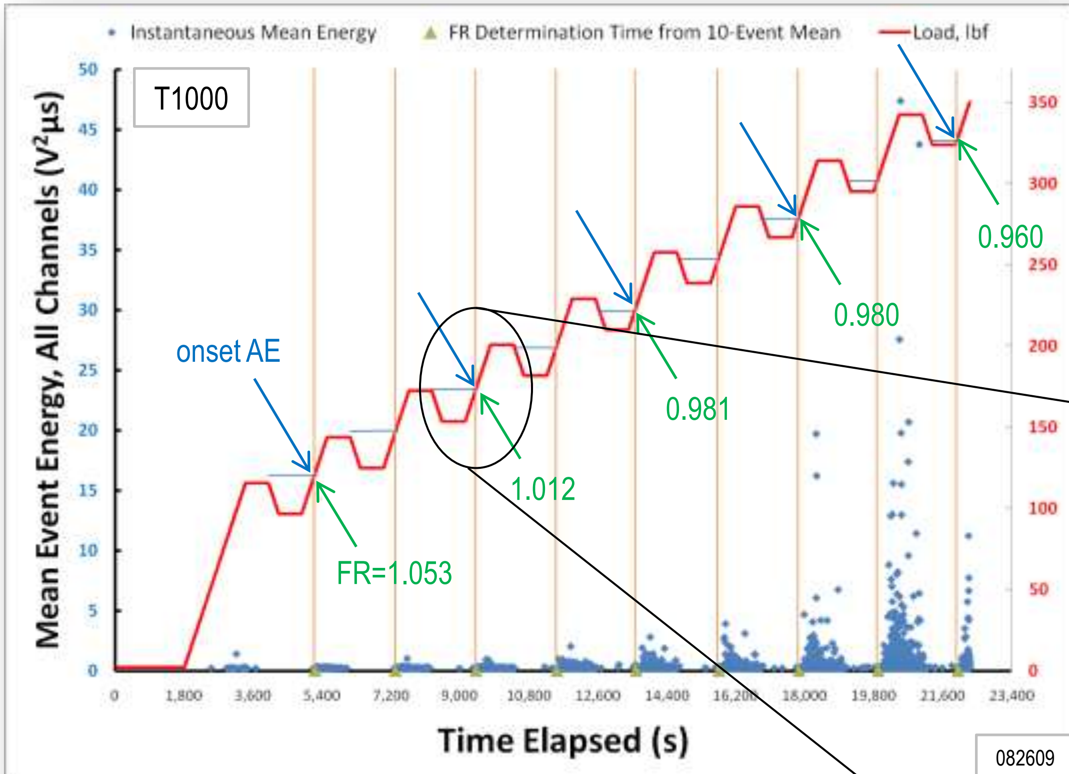


Note: lower values of R^2 for T1000 as compared to IM7

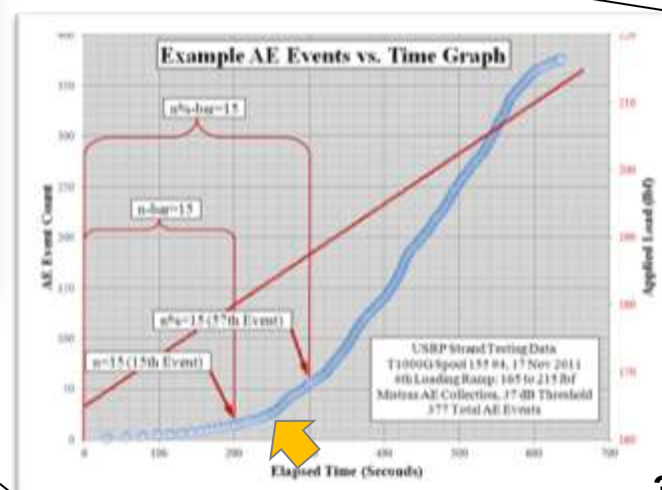
Results & Discussion

Data Reduction Enhancements

- Analytical identification of the 'knee' § in the AE events vs. time curve using an exponentially weighted moving average (EWMA) method:



4th Felicity ramp

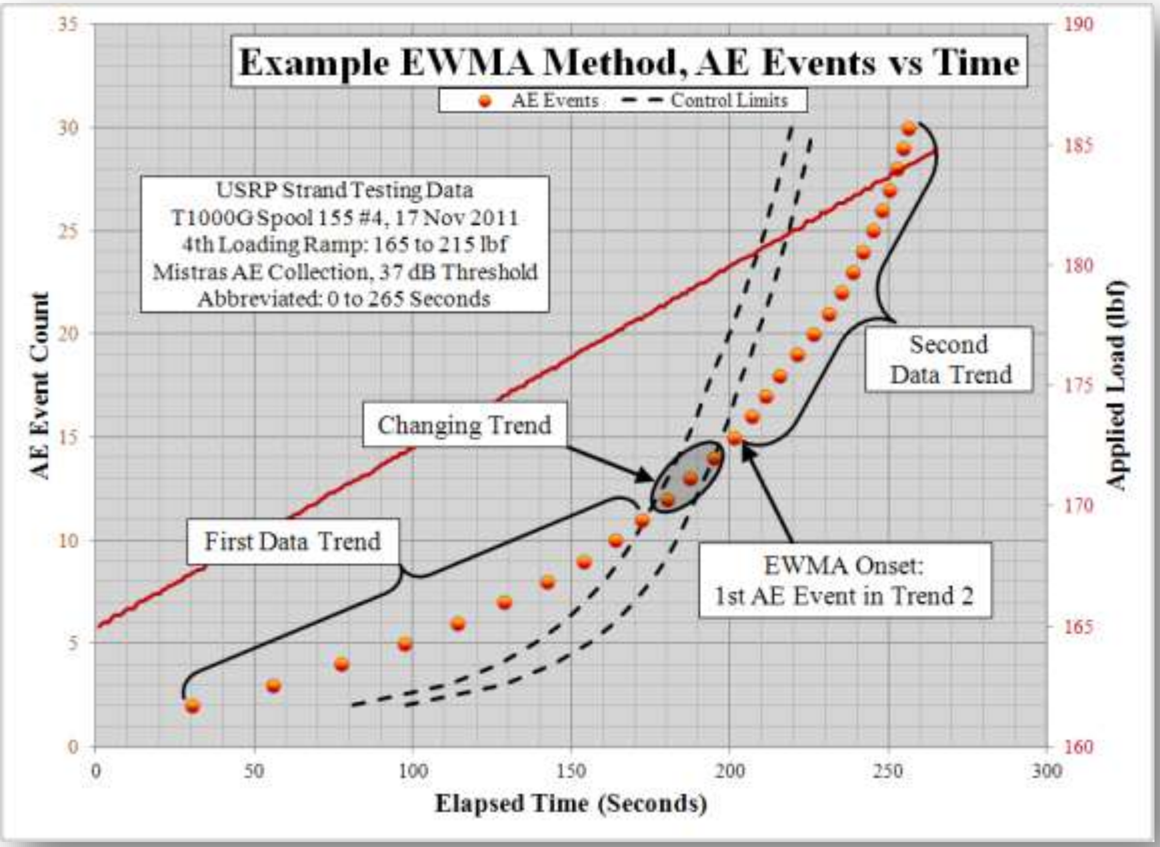


§ *knee in the curve* — a dramatic change in the slope of the cumulative AE (MARSE or Signal Strength) versus time curve (ASTM E2478).

Results & Discussion

Data Reduction Enhancements

- The 'knee' is not analytically defined in ASTM E2478[§] necessitating development of an exponentially weighted moving average (EWMA) method:



plotted statistic:

$$z_i = \lambda x_i + (1-\lambda) z_{i-1}$$

control limits:

$$Limits = T \pm L \frac{S}{\sqrt{n}} \sqrt{\frac{\lambda}{2-\lambda}\left[1-(1-\lambda)^{2i}\right]}$$

standard deviation:

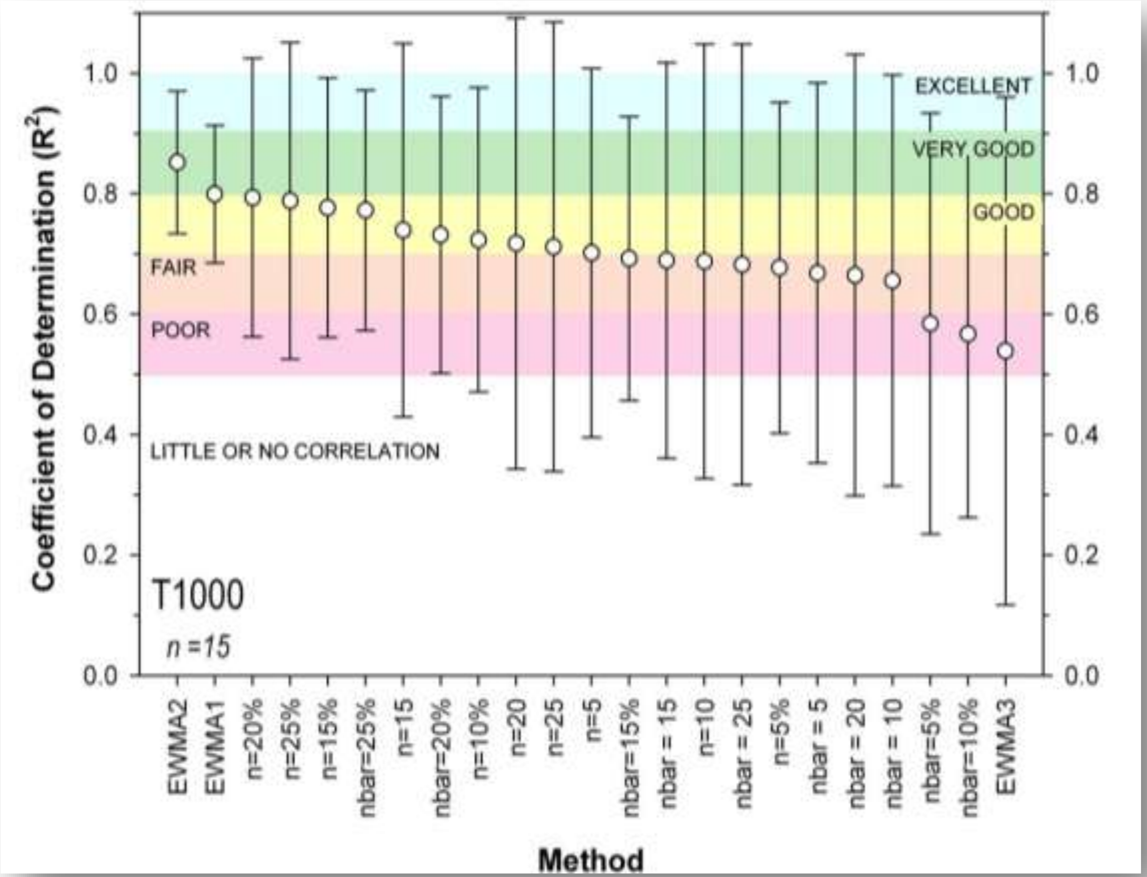
$$\hat{\sigma} = \frac{\sum_{i=0}^{i} MR_i}{(i-1) d_2(2)}$$

§ ASTM E2478, *Standard Practice for Determining Damage-Based Design Stress for Glass Fiber Reinforced Plastic (GFRP) Materials Using Acoustic Emission*, American Society for Testing and Materials, West Conshohocken, PA, 19428 (2011).

Results & Discussion

Data Reduction Enhancements

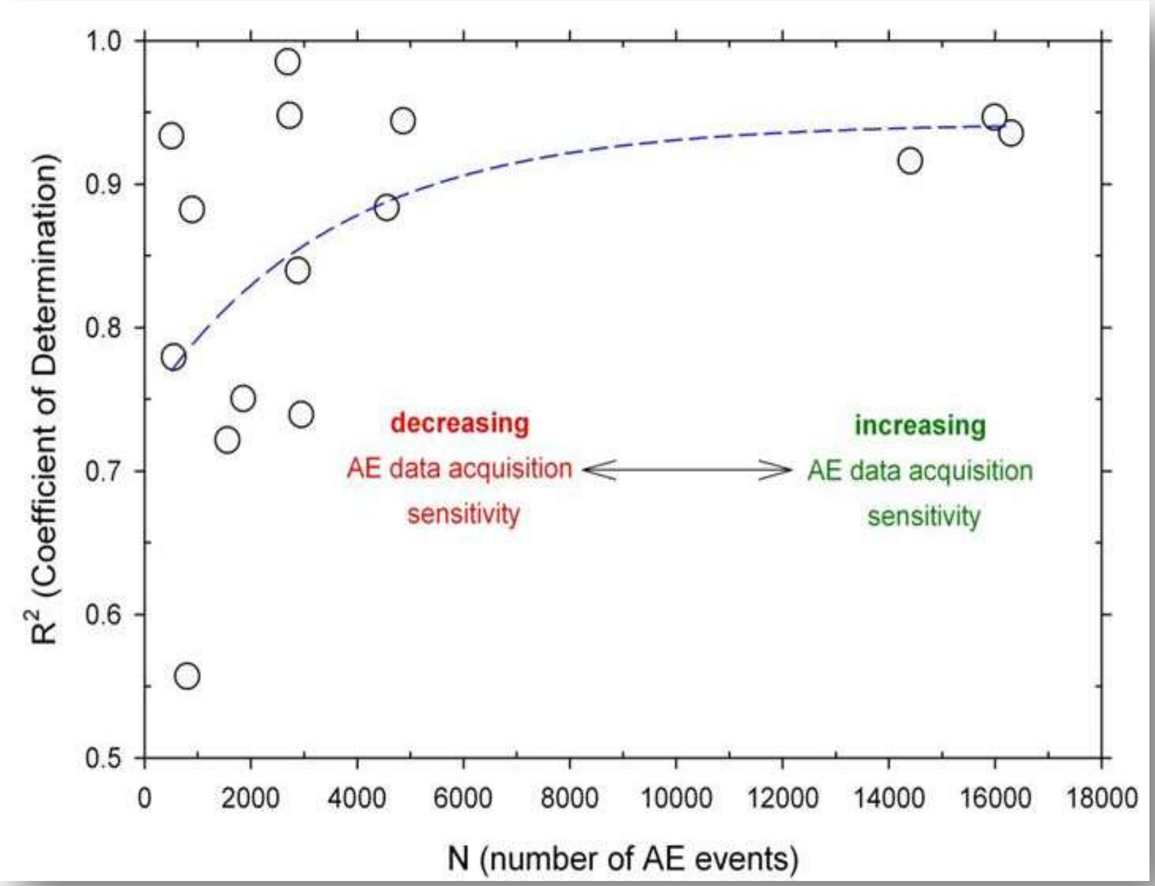
- EWMA method found to yield better (more linear) results than other methods <u>for</u> T1000 tow:



better linear fit

Results & Discussion
Data Reduction Enhancements

- Effect of data richness on FR trend analysis linearity showing that more sensitive AE DACS setting produce better linearity:



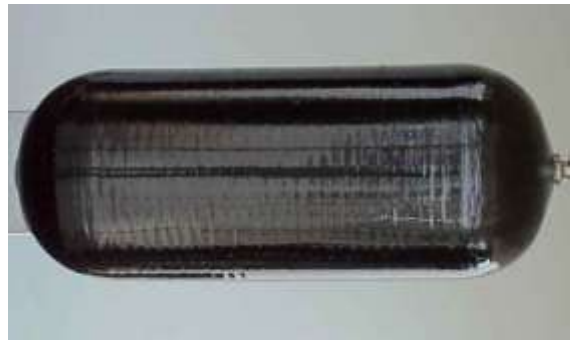
For example, 30 dB sensitivity is better than 50-60 dB sensitivity for FR analysis or quantification of fiber breakage events

Application to
Composite Overwrapped Pressure Vessels
(COPVs)

Results & Discussion

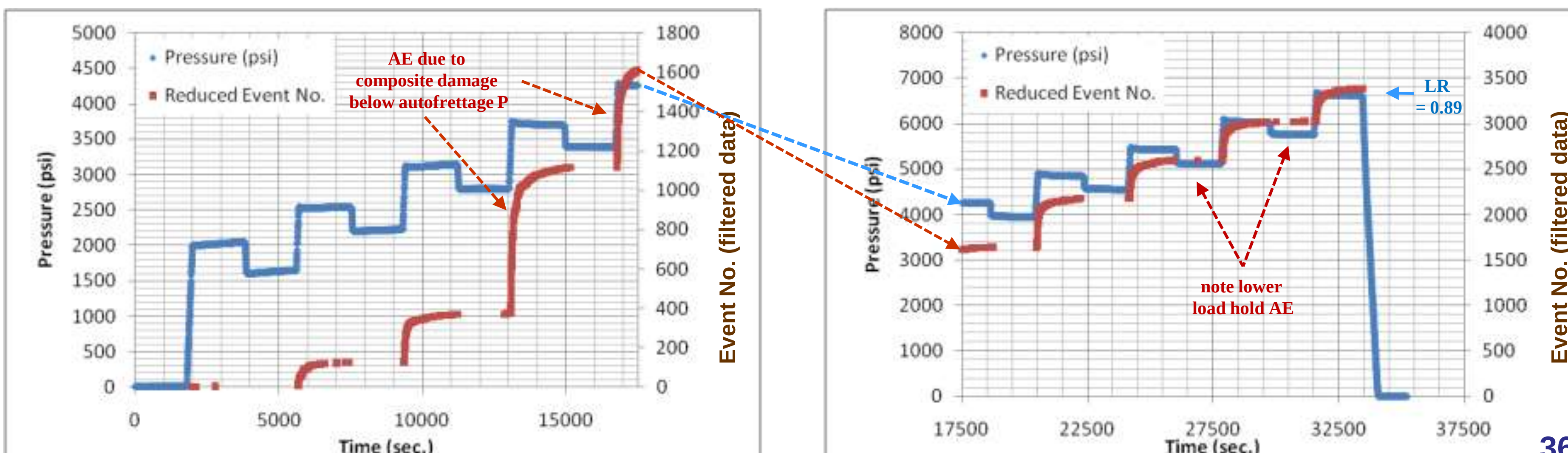
COPVs

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



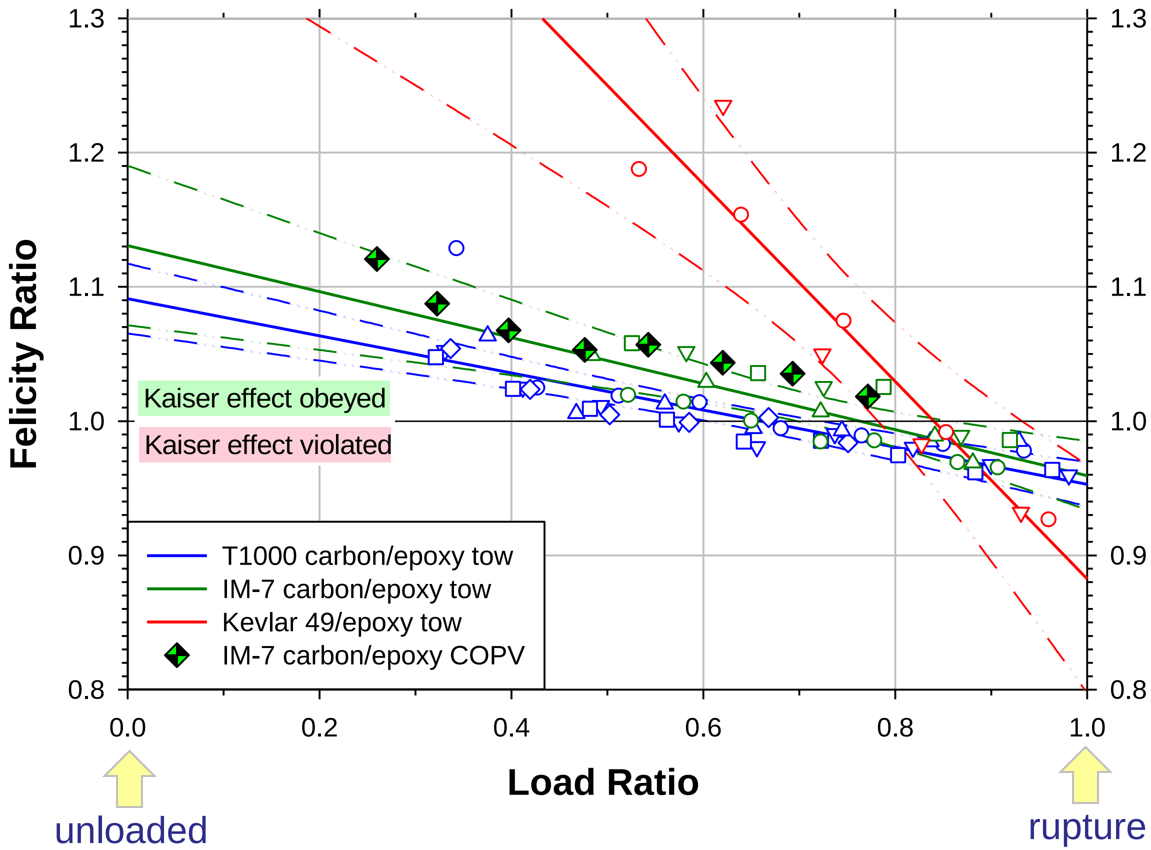
Pressure & Events vs. Time

0 to 17500 s **17500 to 37500 s (cont.)**



Results & Discussion
COPVs

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

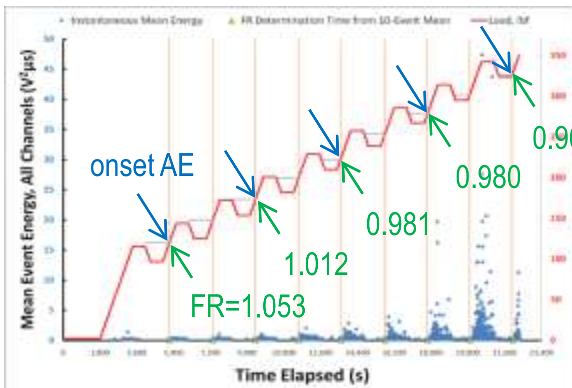


Least squares fits (solid lines) and 99 % confidence intervals (dash-dot-dot lines) also shown for T1000 and Kevlar® 49

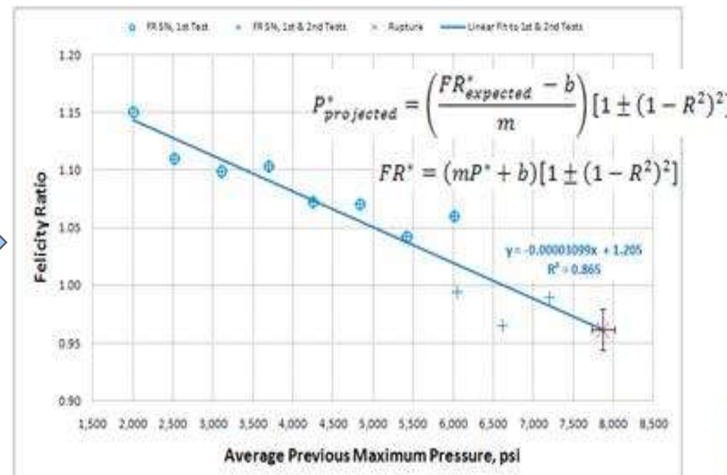
Results & Discussion
COPVs

- Lifetime (burst pressure) prediction of a COPV using a semi-empirical *FR* approach §

Subject C/Ep composite to intermittent load hold pressure schedule (red)



Measure cumulative composite damage by monitoring approach of *FR* to *FR**



First 8 Data Points:

$FR^* = 1.000 \pm 0.010$

$P^*_{projected} = 7{,}540 \pm 75$ psi in agreement with hydroburst data (below)

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

All Data:

$FR^* = 0.961 \pm 0.018$

$P^*_{projected} = 7{,}870 \pm 144$ psi higher than initially projected 7,540 psi value

$P^*_{observed} = 7{,}869$ psi higher than 7,529 psi value based on hydroburst data on 2 identical COPVs

1. Nichols, C., J. Waller, and R. Saulsberry, "Acoustic Emission Lifetime Estimation for Carbon Fiber/Epoxy Composite Overwrapped Pressure Vessels," *USRP Final Report*, NASA-JSC Whites Sands Test Facility, Las Cruces, NM, August 2010.

2. Waller, J., C. T. Nichols, D. J. Wentzel, and R. L. Saulsberry, *Use of Modal Acoustic Emission to Monitor Damage Progression in Carbon Fiber/Epoxy Composites*, QNDE Conference, July 18 – 23, 2010, San Diego, CA.

Results & Discussion
COPVs

AE event decay rate analysis on load holds using ASME Section X, Appendix 8 §

$$y = Ae^{Bt} + C$$

Acceptance criteria from ASME Section X Mandatory Appendix 8 and NB10-0601 Supplement 9 :

Acceptable Event Stability:

-0.1 < ***B*** < -0.0001 & $R^2 \geq 0.80$

Observed acceptance criteria in WSTF IR&D IM7 COPV tests (more stringent):

Acceptable Event Stability:

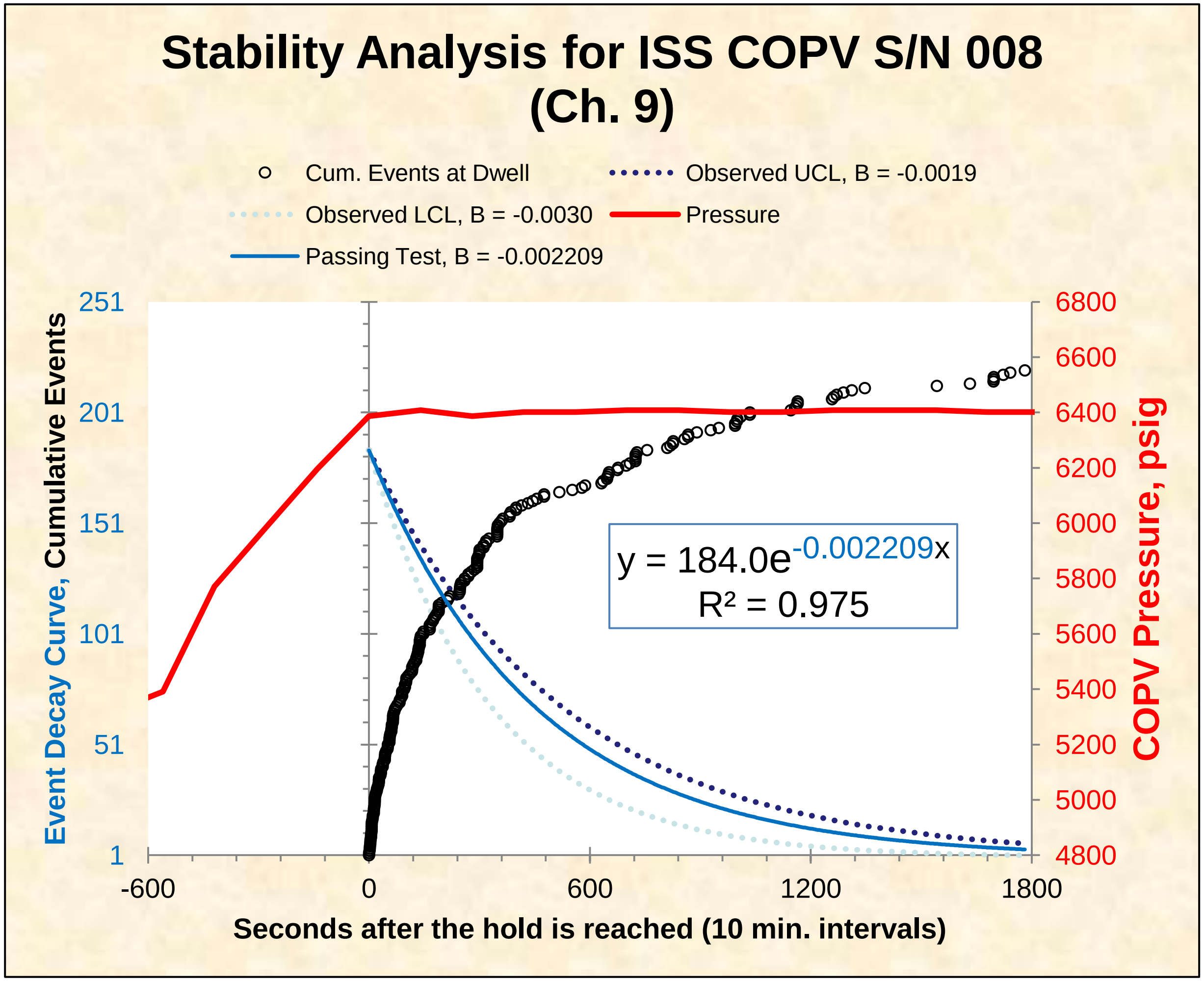
-0.0030 < ***B*** < -0.0019 & $R^2 \geq 0.90$

Shape factor B can also be expressed as the time required for the structure to emit 99% of events on a dwell.

$$t_{99\%} = \frac{\ln(0.01)}{B}$$

25 to 40 minutes

(1535 to 2424 sec)



§ ASME Boiler and Pressure Vessel Code, Section X: Fiber-Reinforced Plastic Pressure Vessels, Section X, Appendix 8-620 Supplementary Examination Requirements (latest revision).

Conclusions
COPVs

- 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 AE events are low energy (<2 $V^2\mu s$)
  - FR AE events occur close to the observed failure locus
  - FR AE events consist of more than 30% fiber breakage (>300 kHz)
  - FR AE 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
- EWMA knee methods for determining the ‘knee’ look very promising
- Exponential AE event decay rate analysis on load holds look very 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)

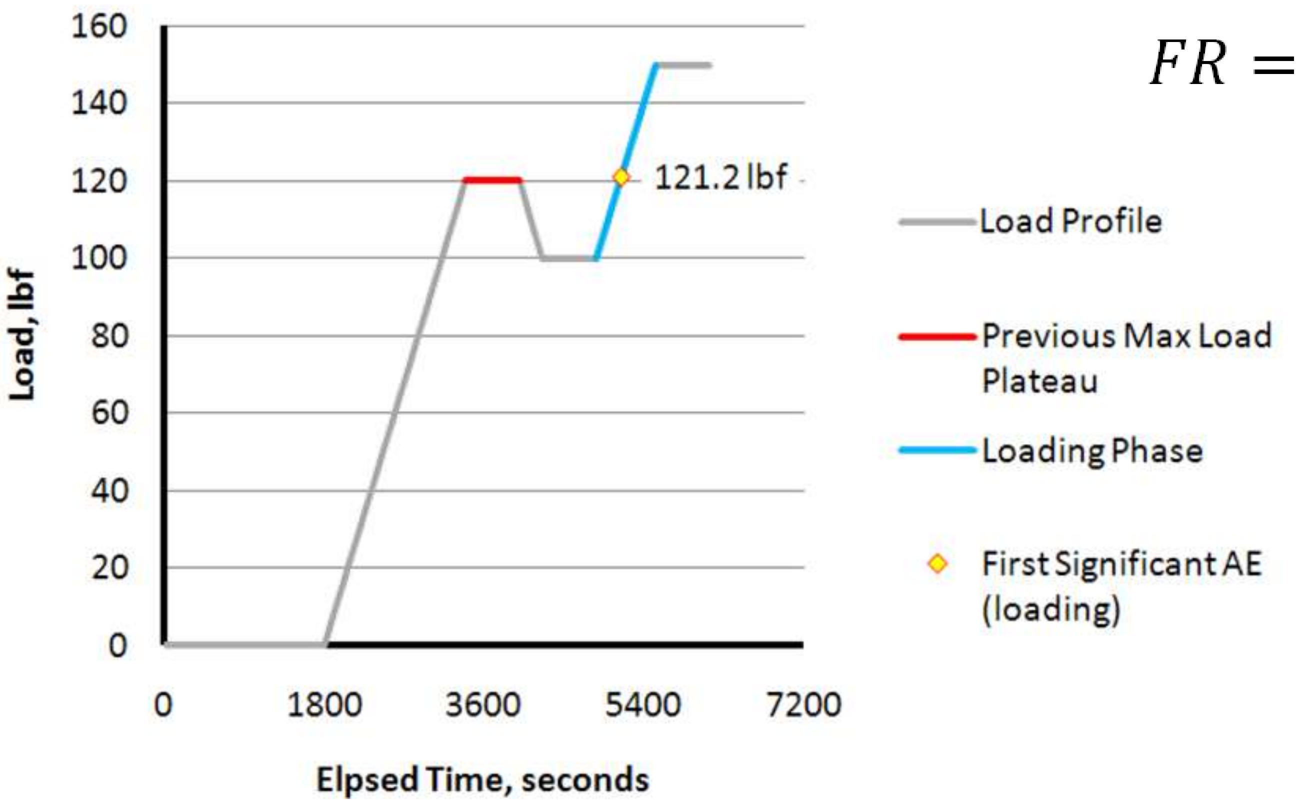
Support to develop AE methods specific to K/Ep and C/Ep

(NDE of composite micromechanics)

Back-Ups

Felicity ratio (FR)

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

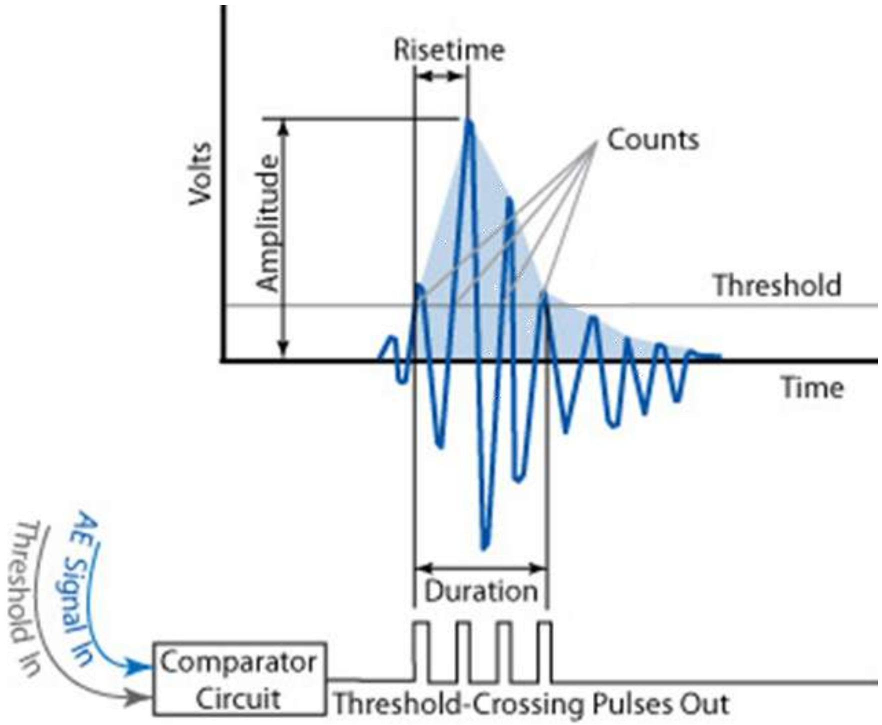


$$FR = \frac{121.2}{120} = 1.01$$

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/)

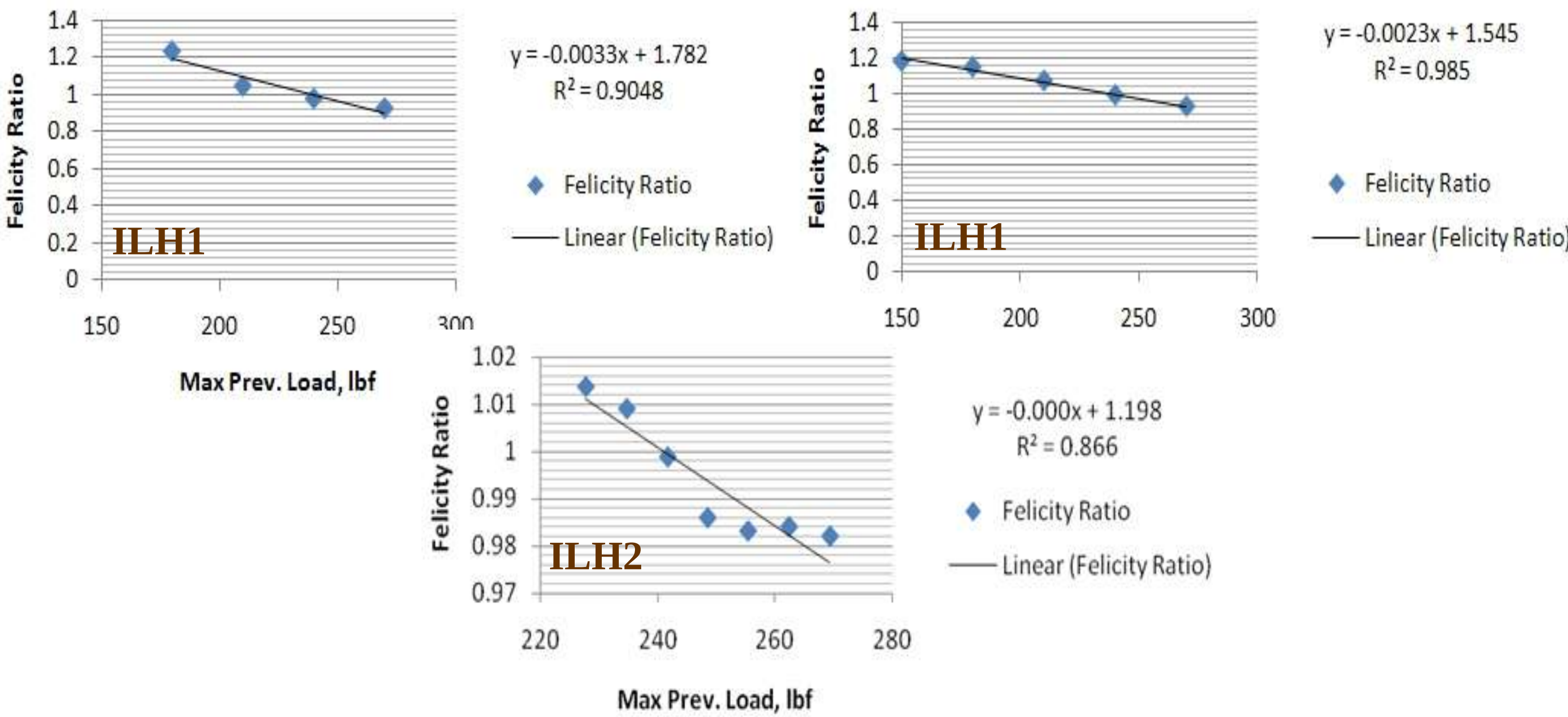


•100 mV

Results & Discussion

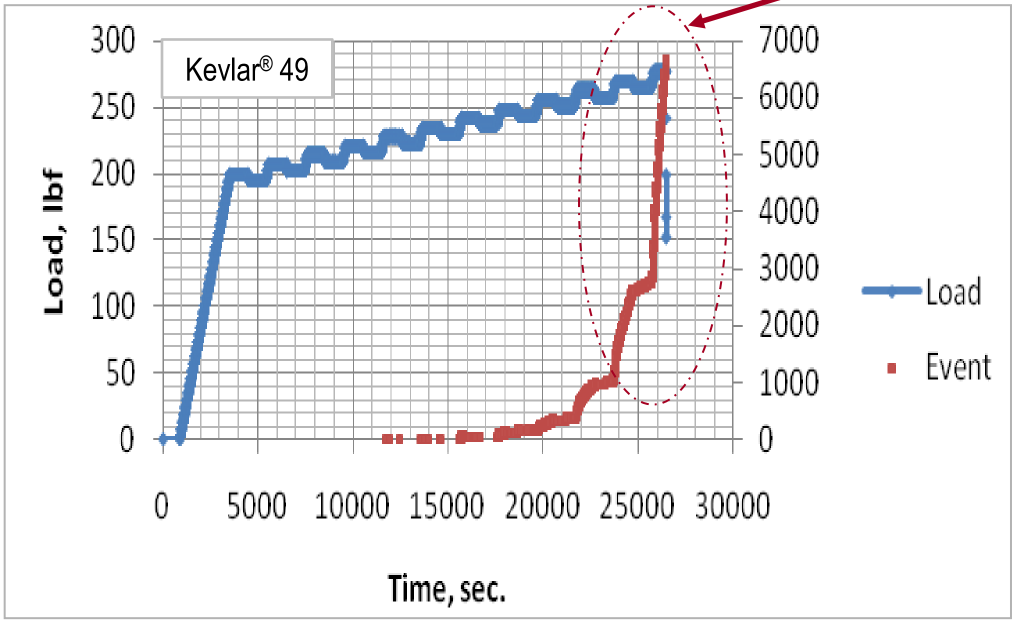
- For Kevlar-epoxy 4650 denier tow, correlation coefficients for ILH1 & 2 methods indicated good (R^2 = 0.866) to excellent (R^2 = 0.985) agreement:

Kevlar® 49-epoxy



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:

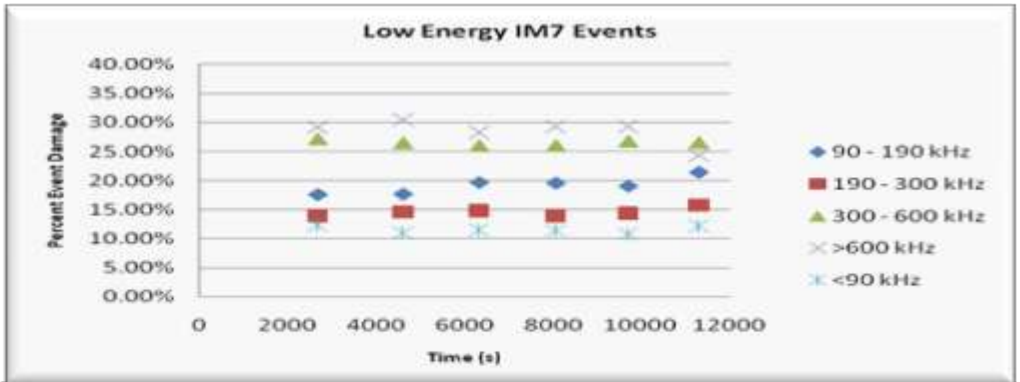


  – Areas of critically intense AE activity also showed greatest violation of Kaiser effect, hence, the lowest FR values

Results & Discussion

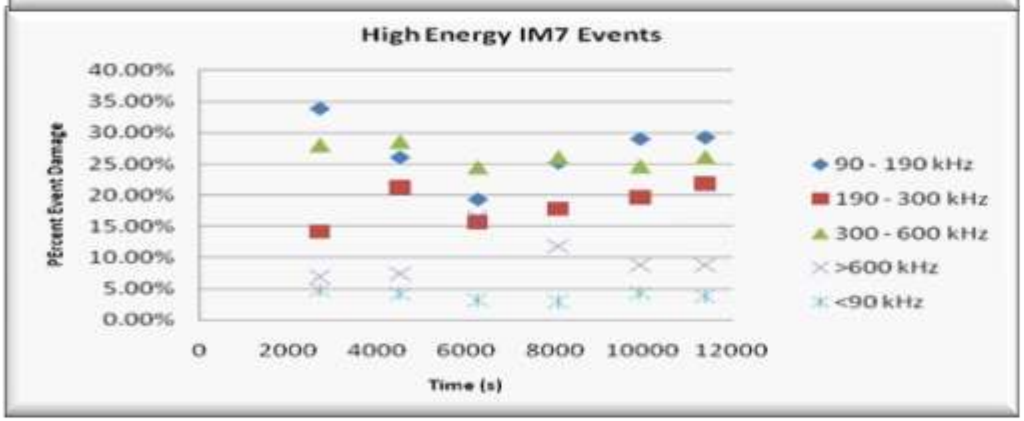
- IM-7 low *vs*. high energy events

Low Energy



E < 2 V^2-μsec

High Energy

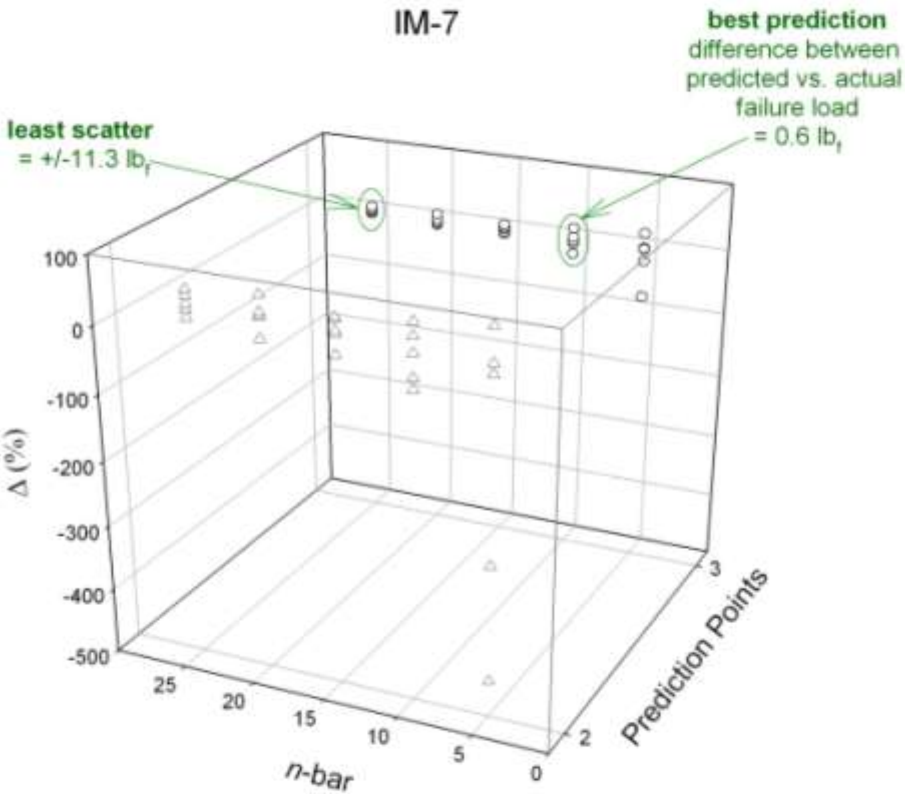


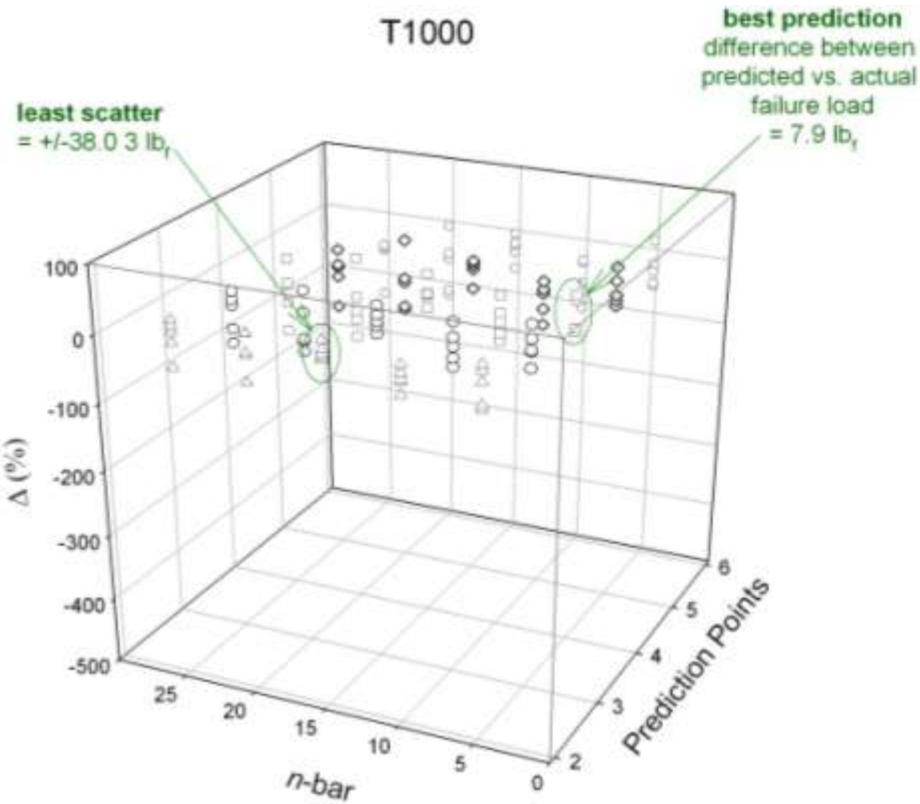
E > 2 V^2-μsec

- 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
composite tow

- Response surface generated using rotating mean method to identify where during in damage cycle best prediction of catastrophic failure can be made:



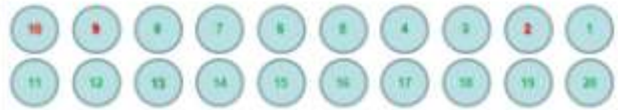


$$Delta = Actual - Predicted\ Failure\ Load$$

Results & Discussion

COPVs

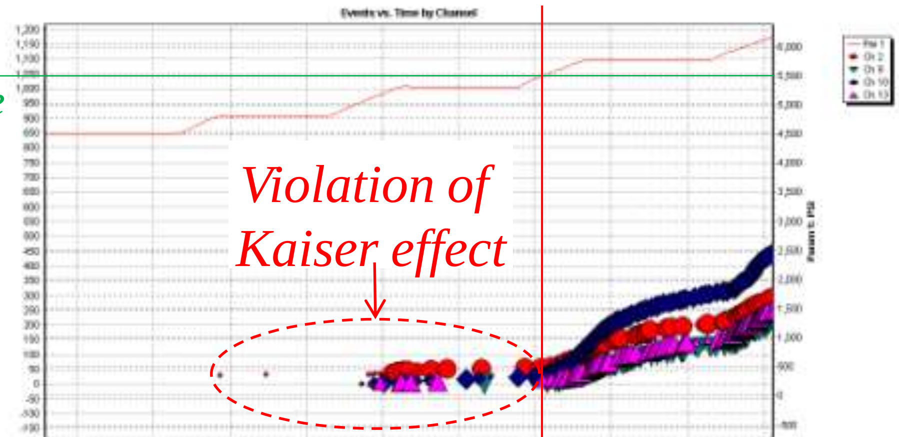
WSTF Area 270 Test, Week 1, T1000 COPVs (General Dynamics)



ca. 5500 psi

autofrettage pressure

burst or impending burst vessel



ca. 5500 psi

autofrettage pressure

survivors