CONCLUDING TASKS

White Sands Test Facility

Task

In-Situ Nondestructive Evaluation of Kevlar® and Carbon Fiber Reinforced Composite Micromechanics for Improved Composite Overwrapped Pressure Vessel Health Monitoring
(Concluded in FY11)

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Background

NASA has been faced with recertification and life extension issues for epoxy-impregnated Kevlar® 49 (K/Ep) and carbon (C/Ep) composite overwrapped pressure vessels (COPVs) used in various systems on the Space Shuttle and International Space Station, respectively. Each COPV has varying criticality, damage and repair histories, time at pressure, and pressure cycles. COPVs are of particular concern due to the insidious and catastrophic burst-before-leak failure mode caused by stress rupture (SR) of the composite overwrap. SR life has been defined [1] 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.” SR has none of the features of predictability associated with metal pressure vessels, such as crack geometry, growth rate and size, or other features that lend themselves to nondestructive evaluation (NDE). In essence, the variability or “surprise factor” associated with SR cannot be eliminated. C/Ep COPVs are also susceptible to impact damage that can lead to reduced burst pressure even when the amount of damage to the COPV is below the visual detection threshold [2], thus necessitating implementation of a mechanical damage control plan [1]. Last, COPVs can also fail prematurely due to material or design noncompliance. In each case (SR, impact or noncompliance), out-of-family behavior is expected leading to a higher probability of failure at a given stress, hence, greater uncertainty in performance.

For these reasons, NASA has been actively engaged in research to develop NDE methods that can be used during post-manufacture qualification, in-service inspection, and in-situ structural health monitoring. Acoustic emission (AE) is one of the more promising NDE techniques for detecting and monitoring, in real-time, the strain energy release and corresponding stress-wave propagation produced by actively growing flaws and defects in composite materials [3, 4, 5, 6, 7, 8]. To gain further insight into the mechanisms responsible for composite rupture, broadband ‘modal’ acoustic emission analysis was used. Also, since AE data reduction proved to be very time consuming, specialized data reduction software was written to automate the process.

Objectives

Currently there are no integrated NDE plans for baselining and monitoring defect levels in fleet COPVs, much less for performing life-cycle maintenance inspections either in a traditional remove-and-inspect mode or in a more modern *in situ* structural health monitoring (SHM) mode. However, even before an NDE plan can be implemented, quantitative accept-reject criteria have to be developed that correlate known levels of damage with and a measurable NDE 'damage' parameter.

Therefore, the first objective of this project was to demonstrate proof-of-concept that an AE-determined 'damage' parameter exists, and that this parameter can be used either as an accept-reject criterion, or to predict catastrophic failure. Two AE-based 'damage' parameters were examined in this project: 1) the Felicity ratio (*FR*), and 2) cumulative fiber breakage as determined by Fast Fourier Transform (FFT)-batch processing. Other AE-determined 'damage' parameters such as the change in AE activity upon proof loading (Dunegan's corollary, [9]) or unloading (the calm ratio, [10]) were not examined.

This project develops enabling technologies for NDE of space exploration systems containing composite materials, in particular, COPVs. Upon further refinement these enabling technologies can served as the basis of engineering controls expressed in the form of accept-reject criteria or real time burst prediction. Implementation of these controls can in turn mitigate or eliminate the catastrophic hazards associated with load bearing composite materials and components such as COPVs that pose a risk to existing and future exploration spacecraft and crews. This is especially important for the COPVs now used on the International Space Station, some of which have unfavorable long term reliability risk factors. This project also improves safety for COPVs that have been compromised due to impact, radiation damage on orbit, or whose safety margins have otherwise been reduced by increased performance or weight saving demands, for example, the push to thin-walled metal liners. The impetus behind this project was the pull of AE equipment of complex structures, in this case COPVs, to Technology Readiness Level (TRL) 3 (perform research to demonstrate feasibility of technology applied to a complex structure; namely, a COPV), ultimately opening up the possibility of autonomous inspection during service.

Approach

Overall: This project is a subtask of a multi-center project to develop state-of-the-art NDE techniques capable of evaluating composite damage in K/Ep and C/Ep materials of construction and COPVs. In this subtask, broad band AE was used to characterize micromechanical damage progression in uniaxial Kevlar® 49 K/Ep tows, IM7 and T1000 C/Ep tows, and IM7 COPVs [11, 12, 13, 14].

The basic outline of this project was to use a building block approach to first demonstrate proof-of-concept for well-characterized composite material classes (single tow reinforced with intermediate or high modulus carbon fiber, or high modulus aramid fiber). This proof of concept was then extended to a more complex structure, in this case IM7 COPVs.

Testing of single tows allowed the *FR* to be correlated with increasing damage. Damage progression was also followed by analyzing the FFT of each AE event. Manual summation of FFTs occurring during the Felicity ramps, for example, gave a damage 'fingerprint' for the damage occurring during that part of the loading profile. Using this approach, it was possible to determine the relative amounts of different types of microscopic damage occurring at any particular time during the load profile (e.g., matrix cracking, fiber-matrix debonding, fiber pull-out, and fiber breakage [5]).

Stress Profiles: Tows and COPVs were subjected to intermittent load hold (ILH) stress or pressure profiles (or similar) based on the pressure tank examination procedure described in ASTM E 1067 [15], and also referred to as the manufacturer's qualification test in ASTM E 1118 [16]. Stress profiles (Figure 1) were applied to tow specimens using an Instron® 5569 Series Electromechanical Test Instrument equipped with a 50 kN (11,200 lb_f) capacity load cell. Pressure profiles (Figure 2) were applied to IM7 COPVs using a high pressure hydraulic pump. COPVs were pressurized at 0.07-0.08 *LR*/min (~10 psi/sec) (*LR* = load ratio = instantaneous pressure divided by the anticipated or actual burst pressure). Depressurization rates were controlled using a motor operated needle valve. The pressurant media was a 95% water 5% soluble oil solution. Blast containment consisted of a Lexan® burst enclosure and tire mats. Pressure was measured using pressure transducers located upstream of the COPV. To insure safety, in-house software capable of remotely controlling the pressure profile was used.

AE data acquisition and reduction: Details about Digital Wave Corporation (DWC, Centennial, CO,) FM-1 AE system, data reduction, software, adhesive, and system verification checks are discussed elsewhere [17]. For tow tests, four DWC B1080 piezoelectric sensors (DWC, 50 kHz to 1.5 MHz frequency range) spaced approximately 4 cm (1.6 in.) from each other were used (Figure 3, right). For COPV tests, six DWC B1025 sensors (100 kHz to 3.0 MHz range) placed at the equator (2) and boss ends (4) were used. The sensor spacing was determined to be acceptable for picking up high frequency AE caused by fiber breakage.

For COPV tests, a PCI-2 AE system equipped with AEWIN™ PCI2-24 software (Mistras Group, Inc. (MGI), Princeton, Junction, NJ) was also used. For COPV tests, two WD wideband differential sensors (MGI) placed about 2.5 cm (1-in.) below the transition COPV region. Peak frequency vs. time analyses consisted of monitoring the cumulative number of hits versus the time they occurred for 5 distinct frequency bands. The frequency band distribution was divided as follows: 40-60 kHz, 61-125 kHz, 126-200 kHz, 201-265 kHz, and 266-400 kHz.

FFTs were obtained directly from DWC WaveExplorer™ or MGI AEWIN™ software. To expedite DWC data analysis, procedures for isolating the direct wave and removing reflections [17] were not used. To minimize loss of the high frequency component attributable to fiber breakage, and dispersion effects due to increasing damage, only the FFT from the first arrival channel (DWC FM-1) was analyzed. DWC FFTs were exported to Microsoft Excel™ and the area under the frequency curve was calculated using a right Riemann sum and a step size equal to the default interval between frequencies (0.49 kHz). DWC frequency ranges were assigned as follows: matrix cracking, 90-190 kHz; fiber pullout and debonding, 190-300 kHz; fiber breakage, >300 kHz [5].

Materials: Unidirectional 3817 denier Torayca® T1000G ($v_f \approx 0.676$ to 0.707) and 3775 denier HexTow™ IM7 (5000) ($v_f \approx 0.670$ to 0.708) 12,000 filament composite tows were used. The epoxy matrix consisted of a UF3323-102 resin (ATK Space Systems, Ogden, UT). Curing was accomplished by wrapping the prepreg tow over a steel mandrel and subjecting it to the same cure cycle as used for the IM7 COPVs. Each tow specimen had a ribbon-like geometry with irregular width and thickness. Tow ends were secured inside the cardboard end tabs with 3.5 g (0.12 oz.) of 1:1 Hardman® Extra-Fast Setting Epoxy (Ellsworth Adhesives, Germantown, WI) which was cured at room temperature for at least 24 h before test. Cardboard end tabs were designed with two stacked $l \times w \times h = 25 \times 1 \times 1$ mm (1 × 0.079 × 0.079 in.) cardboard spacers on the outside edge of each tab, thus increasing the amount of epoxy held within the tab (Figure 3, left). This tabbing procedure reduced the amount of tow pullout and crushing (fewer grip failures) and reduced grip noise (amount of data filtered from raw AE data sets decreased to < 10 percent).

COPV fabrication was conducted at HyPerComp Eng. Inc. (Westlake, CA) and consisted of wrapping a cylindrically shaped aluminum liner (Samtech International, Inc.) with the same IM7 prepreg tape described above. The COPV had a nominal outer diameter of 16.0 cm (6.3 in.), length of 50.0 cm (19.8 in.) and a wall thickness of 2.0 mm (0.080 in.). The helical wraps, denoted by “H”, consisted of 2 plies oriented at ±13.8° and ±17.1° with an average angle of 14.9° relative to the vessel’s axial direction, while the cirque or “hoop” wrap, denoted by “C”, consisted of 1 ply (wrap pattern 3H/15C). Hydroburst tests gave a burst pressure of 51.91 ± 1.01 MPa (7529 ± 147 psi).

Co-Funding

Funding was received from the Undergraduate Student Research Project (USRP) program to pay for interns engaged in all aspects of this NASA Nondestructive Evaluation Working Group (NNWG)-sponsored project, including but not limited to AE method refinement and writing in-house *FR* analysis software for tows and COPVs, thus increasing the technical readiness level of promising AE methods slated for use in the “Smart COPV” project (NNWG FY12 New Start).

Funding was also received for a NASA-JSC Internal Research and Development proposal for COPV-level testing during FY11 (completed) and FY12 (on-going). This testing involves subjecting IM7 and T1000 C/Ep COPVs to a stress level indicative of the autofrettage pressure (nominal *LR* = of 0.6 to 0.8), whereupon the burst pressure was predicted based on *FR* trendline analysis.

Customers

This project directly targeted the Reliability/Life Assessment/Health Monitoring in the NASA Office of the Chief Technologist Roadmap TA12, Materials, Structures, Mechanical Systems and Manufacturing Materials, Structures, Mechanical Systems and Manufacturing and is crosscutting to other discipline road maps. For example, TA07, Human Exploration Destination Systems discusses the criticality of having integrated health monitoring/management systems to free up the crew to cope with other mission issues. The necessary specialized data reduction software development for this is also deemed critical. Similarly, TA02, In-Space Propulsion Technologies discusses the criticality of having integrated systems health management. This project was the first step in promulgating and developing the necessary real time NDE methods that will be used in these integrated health management systems. The International Space Station program, the NESC Composite Pressure Vessel Working Group, future manned and unmanned NASA space exploration programs, the Office of the Chief Technologist; plus commercial, Department of Defense, and Department of Transportation concerns utilizing composite overwrapped pressure vessels will benefit from this work.

Metrics

Progress was reported on an annual basis and peer reviewed by the NNWG members. Comparisons were made to the original project plan and schedule, providing a measure of project progress.

External NASA peer review was considered to be essential and was obtained by communicating findings to E. Madaras of NASA LaRC and J. Walker of NASA MSGC. Non-NASA peer review was obtained from the American Society of Testing and Materials (ASTM) International Committee E07 on Nondestructive Testing, the American Society of Nondestructive Testing (ASNT), and the Acoustic Emission Working Group (AEWG).

Products

This project has produced quantitative AE methods that can be used to monitor progressive damage accumulation in K/Ep and C/Ep tows, and C/Ep COPVs, including optimization of AE data acquisition parameters, sensor selection and sensor spacings. Products also include a more reliable tabbing method for K/Ep and C/Ep uniaxial single tow specimens (valid for tows with breaking forces up to 1800 N (400 lb_f)). To reduce the labor needed for AE data reduction, increase analysis throughput, and to eliminate operator subjectivity, this project has also produced an in-house AE data reduction software that has utility for both tow and COPVs. Features of the software include:

1) calculate the *FR* automatically as a function of load (pressure), representing a vast improvement in labor savings
2) optimize the *FR* using various consensus methods for determining the onset of significant AE
3) calculate pressure breakpoints from noisier COPV pressure schedule data, overcoming errors caused by pressure spikes and overshooting of pressure set points
4) discard *FR* outliers using a robust fitting method

Last, this task led to modifications of the NDE Wave Image Processor (NDEWIP) program developed by NASA GRC, so that it can better accommodate vendor (e.g., DWC) AE data files.

Major Technical Accomplishments

Mentoring and Reporting: Six USRP interns were mentored in support of this task, leading to eight internally published USRP papers, all related to AE. Two conference papers summarizing findings were given at the 2009 and 2010 Quantitative Nondestructive Evaluation (QNDE) Conference [11, 12], and a conference presentation was given to the 2010 ASNT Conference and Quality Testing Show [18]. Two NASA-JSC Biennial Research and Technology Development Reports were published in 2011 as well [13, 14]. Coupled with annual presentations given to the NNWG members, dissemination of technical results helped to insure proper peer review of findings was obtained.

FR Analysis of Composite Tow: The most significant finding in current work was the observation of a predictable, linear decrease in the *FR* with increasing *LR* as composite tow and COPV are taken to failure. While acceptable linearity (R^2>0.9) was obtained on K/Ep tow [11] by using first significant AE event to determine the onset of significant AE, acceptable linearity could only be obtained on C/Ep tow using the first 5 or more significant events. A new parameter referred to as the critical Felicity ratio, or

*FR**, was developed, which is simply the extrapolated value of *FR* at failure (*LR* = 1.0). C/Ep tow gave an *FR** close to 0.96 with an observed scatter between 1 and 2 percent. By comparison, the ultimate tensile strength of C/Ep exhibited more scatter, varying between 5 and 8 percent, indicating that the *FR* is more independent of failure mode than the tensile strength. By analogy, COPV burst pressure would be expected to be more scattered than the *FR** for a given COPV population. Data also revealed T1000 is more damage tolerant than IM7, and C/Ep is more damage tolerant than K/Ep (Figure 4).

FR Analysis of COPVs, Representative Case: Representative IM7 COPV data (filled green and black symbols, Figure 4) were found to overlap IM7 tow data (open green symbols, Figure 4) using *FR*s calculated the same way (first 15 events averaged). This overlap suggested that the IM7 COPV was trending towards failure in much the same way IM7 composite tows were. To determine if this was true, the IM7 COPV was taken to burst in a subsequent ILH test. Stitching the two COPV ILH data sets together (Figure 5) initially proved difficult. However, acceptable agreement was obtained by using a new *FR* determination method based on sampling the first 5 percent of the total number of AE events occurring on any given Felicity ramp.

Predicting COPV burst pressure, *P**, has proven to be difficult given the wide Weibull variability exhibited by composites; however, if the *FR* dependence on *LR* is known for a group of COPVs of the same design and materials-of-construction, it should then be possible to use *FR** derived from the population to predict *P** of an unknown using the following semi-empirical expressions:

$$FR^* = (mP^* + b)[1 \pm (1 - R^2)^2] \tag{1}$$

$$P^* = \left(\frac{FR^* - b}{m}\right)[1 \pm (1 - R^2)^2] \tag{2}$$

where *m* is the *FR* vs. *LR* slope and *b* is the hypothetical *FR* at zero load. In the IM7 COPV test, Eq. (1) gave an *FR** of 0.96 ± 0.02 ($m = -3.10 \times 10^{-5}$, $b = 1.21$, $R^2 = 0.87$) using the stitched COPV data. Assuming that COPV *FR**s exhibit the same scatter as tow *FR**s (1 to 2 percent), the *FR* method should be able to predict the burst pressure with similar accuracy. Application of Eq. (2) to all the data (Figure 5) verified this, giving a predicted *P** of 54.3 ± 1.0 MPa (7870 ± 144 psi), with a 1.8 percent (144 psi) error, and which is virtually identical to the observed *P** of 54.25 MPa (7869 psi) (Figure 5). The initial 8-point data set (filled green and black symbols, Figure 4) acquired during ILH pressurization of the COPV to 46.9 MPa (6800 psi), led to a more conservative predicted *P** of 51.98 MPa (7540 psi). Accurate prediction of the burst pressure of a COPV without taking the COPV to burst opens up the possibility of autonomous in-situ performance verification checks on materials and components such as COPVs, as long as suitable pressure profiles can be applied during service that allow the Felicity effect to be measured.

Waveform Analysis: Analysis of DWC data on C/Ep tow revealed the presence of three characteristic waveforms, each differentiated on the basis of amplitude, duration and frequency. The first characteristic waveform exhibited low amplitude signals with lower frequencies and short durations attributed to matrix cracking. The second waveform exhibited moderate amplitudes with high frequencies and short durations attributed to fiber breakage. The final and most common waveform exhibited high amplitudes and a wide range of frequencies and long durations attributed to concerted failure including all modes of micromechanical damage (Figure 6).

FFT Analysis: In the case of uniaxial loading parallel to the fiber axis, microdamage attributable to tranverse matrix cracking would be not be expected to be as important, while microdamage attributable to fiber breakage caused by preexisting fiber flaws or fiber misalignment would be expected to be more important. FFT data show this is exactly what occurs in IM7 and T1000 C/Ep tow. More specifically, FFT analysis of the *FR* events responsible for the onset of significant AE during the ILH up ramps revealed that the frequency distribution for these events was invariant with respect to *FR,* or analogously *LR* (Figure 7), and essentially the same for T1000 versus IM7 tow (data not shown). Also, fiber breakage (>300 kHz) was the most predominant failure mode for both IM7 and T1000. T1000 had slightly more low frequency damage associated with matrix cracking than IM7, which might be due to to the higher fiber strengh of the T1000, thus causing stresses to be localized more within the matrix at equivalent *LR*s.

Custom Software Development: Analyzing AE data without the aid of sophisticated software has been very time consuming and occasionally introduces human error due to the complexities involved. To circumvent these issues, an algorithm was developed to reduce the AE data and to predict the critical rupture point automatically (Figure 8). An extensive validation process has been undertaken to increase the versatility and accuracy of the algorithm. Specific features of the algorithm include automatic AE data filtering and synchronization of the AE and pressure data. The algorithm also checks the linearity of the *FR* vs. previous highest pressure data using several different averaging methods for determining the onset of significant AE (*FR* nominator), selecting the best (optimal) averaging method to use for the existing AE data set. Five averaging methods are considered: 1) method: AE event = onset; 2) method: mean of the first AE events = onset; 3) % method: the AE event % into all of the AE events for that ramp = onset; 4) % method: the mean of the first % of the AE events for that ramp = onset; and 5) an exponentially weighted moving average (EWMA) method. A Ramer-Douglas-Peucker subroutine is used to determine the previous maximum load (*FR* denominator). To assess the goodness of fit of the generated *FR* data, both coefficient of determination, R^2, and least absolute residual approaches are used to assess goodness of fit, eliminating outliers when necessary. Data output consists of R^2 vs. *FR* plots for each averaging method (Figure 9). Data suggest that by developing a master *FR* curve for a family of identical composite materials, a rapid assessment of in-family or out-of-family performance can be made solely on the basis of comparing the initial *FR* vs. *P* curve. It is intended that future revisions of the algorithm will include this feature.

Future Work

Further work is needed to improve the use of *FR* as an analytical damage parameter. The effect of the activation time (the time a composite as allowed to rest unloaded, prior to re-application of load) on the *FR* must be better understood. The dependence of cumulative fiber breakage on applied load must be understood, which will require batch processing of FFTs using, for example, the NDEWIP software developed at NASA-GRC. Specimen-to-specimen reproducibility and damage progression within a given material or design class must be better understood, which could be facilitated by using commercially available pattern recognition software that differentiates individual acoustic emission events on the basis of energy, duration, and rise time. More work is needed to show whether *FR* trends consistently predict *P** and out-of-family behavior. It is anticipated the significant progress in these areas will be accomplished in FY12 under the “Smart COPV” project (NNWG FY12 New Start).

Upon successful completion of these tasks, the utility of the methods that are developed should be extended to similar material and design classes of composites to determine the merits of universal lifetime prediction using AE-based approaches.

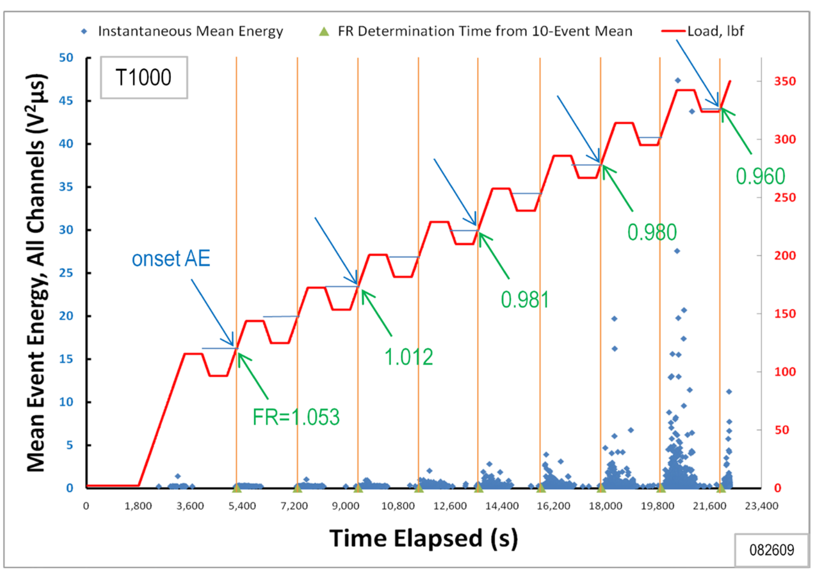

Figure 1 Representative intermittent load hold stress schedule used for a T1000 carbon-epoxy composite tow showing decrease in onset AE relative to previous highest load, and Felicity ratio with time (right y-axis units are in lb_f)).

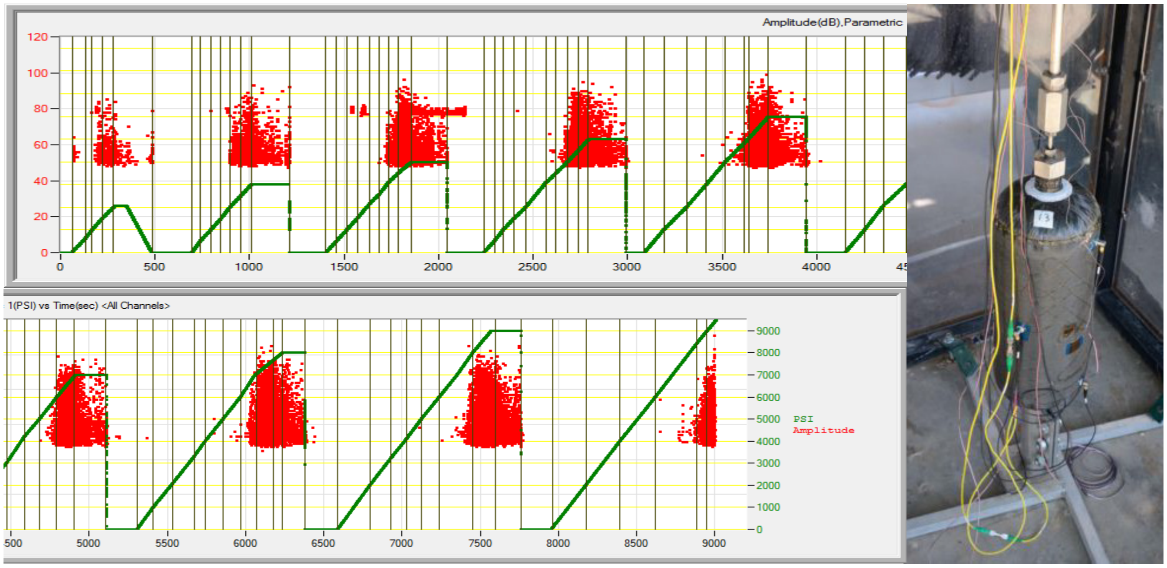

Figure 2 a) Representative pressure schedule (green line) used for an IM7 carbon-epoxy composite overwrapped pressure vessel (right) showing cumulative acoustic emission activity (red data) as a function of applied pressure (pressure cycles were from 1000 to almost 9000 psi in 1000 psi increments.

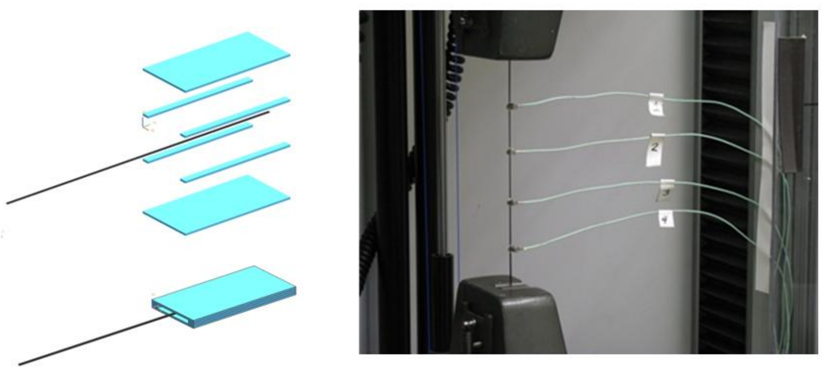

Figure 3 Cardboard end tabs before and after assembly (left, adhesive not shown), and a 25-cm gauge length 12k carbon-epoxy tow specimen mounted in grips showing four B1080 AE sensors (right, end tabs hidden in grips).

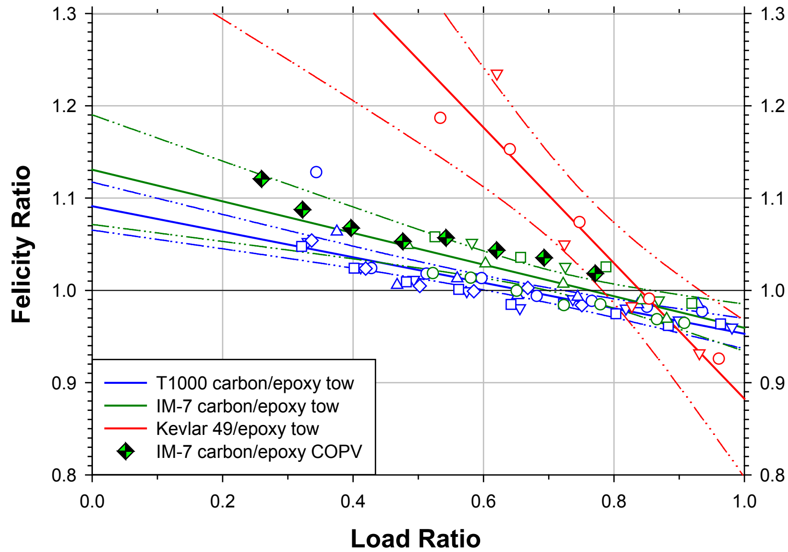

Figure 4 Drop in Felicity ratio with increasing load showing least squares fits and 95 % confidence intervals for T1000 carbon-epoxy tow (blue data), IM7 carbon-epoxy tow (green data), Kevlar®-epoxy composite tow (red data), and an IM7 composite overwrapped pressure vessel (filled green and black symbols), (NOTE: Felicity ratio determined using the first AE event for Kevlar®-epoxy tow, and the mean of the first 15 events for T1000 tow, IM7 tow, and the single IM7 COPV.)

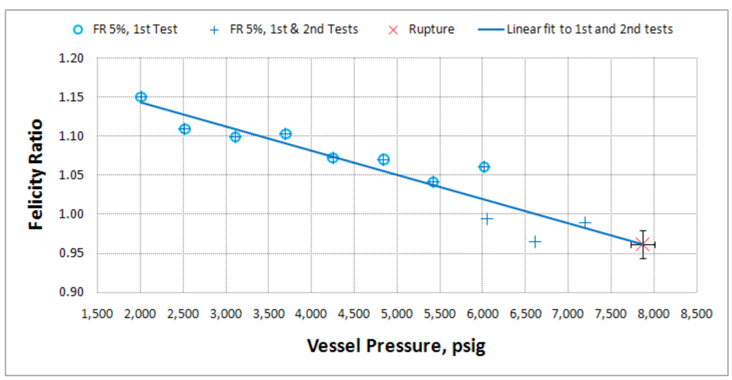

Figure 5 Drop in Felicity ratio with increasing pressure for an IM7 composite overwrapped pressure vessel (large crossed hexagonal symbols). (NOTE: Felicity ratio determined using the first 5 percent of total number of AE events.)

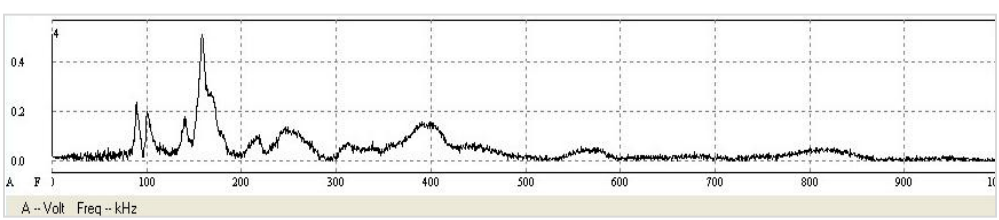

Figure 6 Fast Fourier transform an acoustic emission event taken from a 12k carbon-epoxy tow showing peaks attributable to (left to right) matrix cracking, fiber-matrix debonding and pull-out, and fiber breakage the (unfiltered voltage(V) versus frequency (kHz))

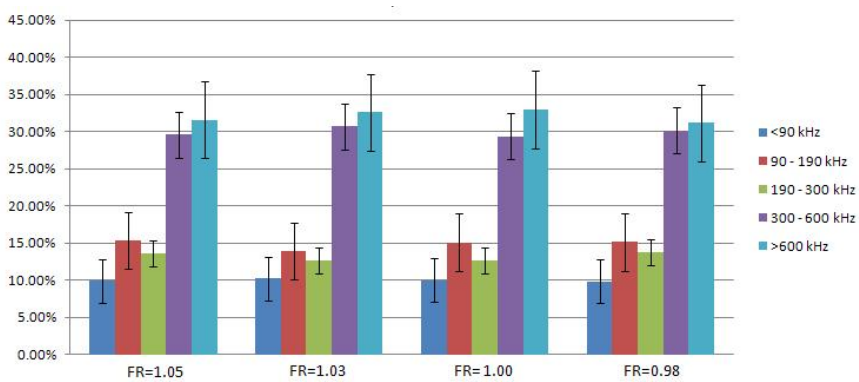

Figure 7 Damage distributions (vertical scale = percent of total damage) for Felicity ratio events for three IM7 carbon-epoxy 12k tow specimens. (NOTE: First 15 significant AE events used for each FR/specimen.)

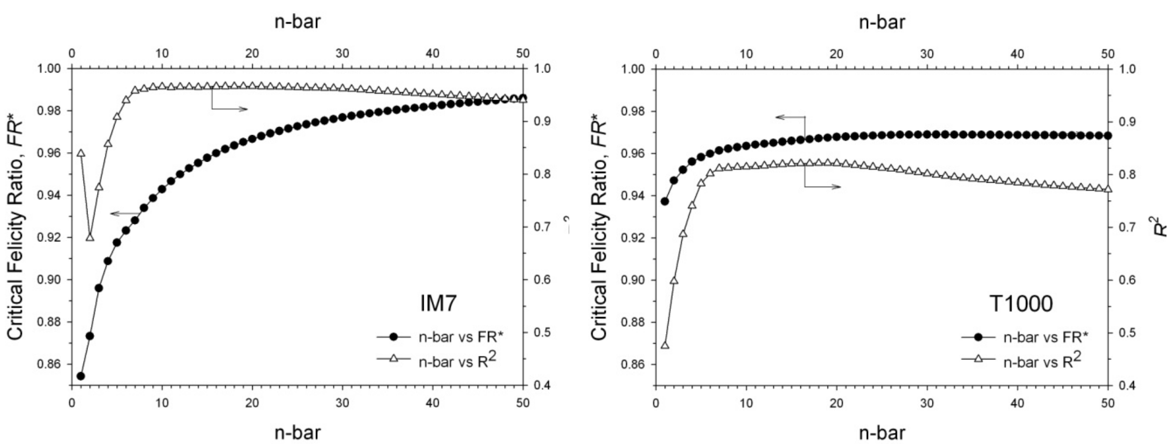

Figure 8 The dependence of the coefficient of determination, R^2, and critical Felicity ratio, FR^*, with the mean of the number of acoustic emission events used to determine the onset of significant acoustic emission for IM7 carbon-epoxy tow (left), and T1000 carbon-epoxy tow (right).

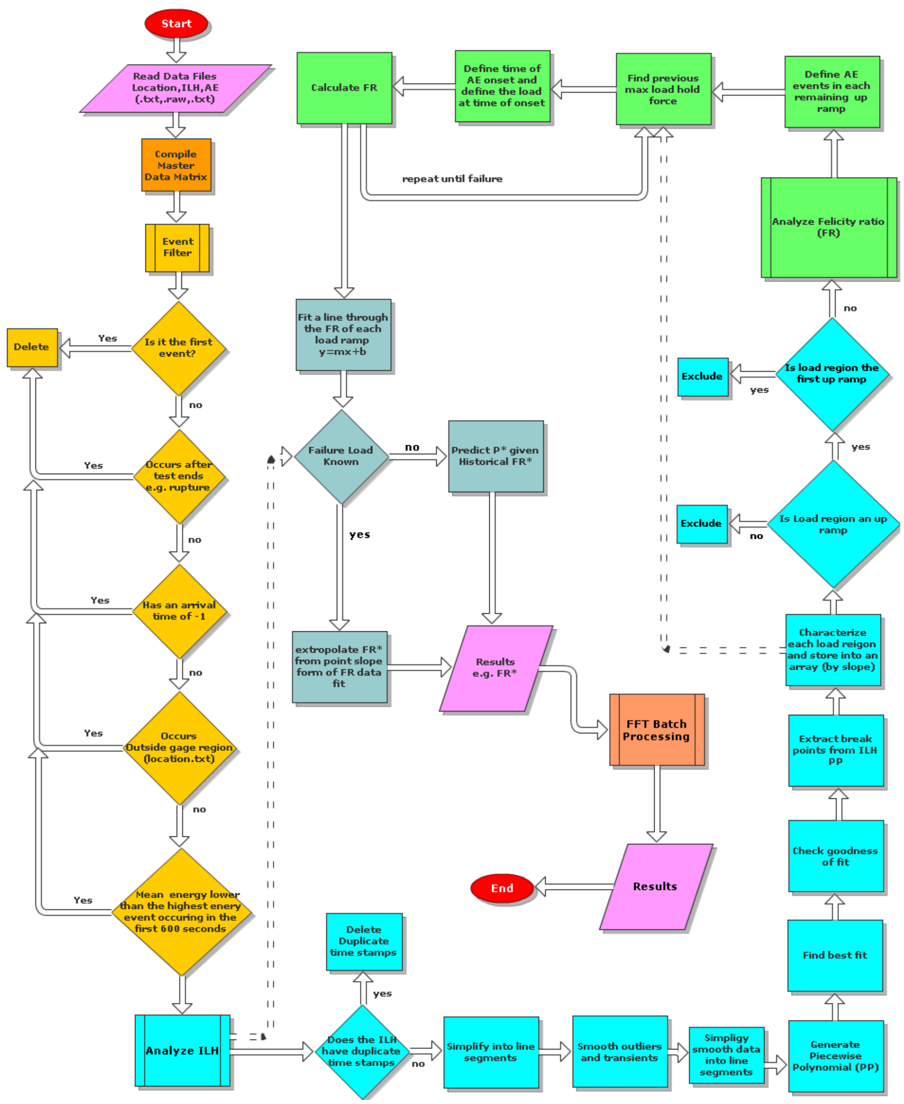

Figure 9 Felicity ratio optimization program flow chart (does not show Robust fitting routine)

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