Acoustic Emission and Development of Accept-Reject Criteria for Assessing Progressive Damage in Composite Materials

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NASA has been faced with recertification and life extension issues for epoxy-impregnated Kevlar® 49 (K/Ep) (E.I. du Pont de Nemours and Company, Wilmington, Delaware) 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 of the composite overwrap. Stress rupture life has been defined 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." Stress rupture 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 stress rupture cannot be eliminated. Consequently, NASA has devoted much effort to develop NDE methods that can be used during post-manufacture qualification, in-service inspection, and structural health monitoring of COPVs. One of the more promising NDE methods for detecting and monitoring actively growing flaws and defects in composite materials is acoustic emission (AE). It is hoped the AE procedures being developed will lay the groundwork for establishing quantitative accept-reject criteria of composite materials and components such as COPVs so that precautionary or preemptive engineering steps can be implemented to minimize or obviate the risk of stress rupture.

Experimental

Testing was performed at NASA Johnson Space Center's White Sands Test Facility. Unidirectional 4560 denier Kevlar® 49 composite strands (manufactured in 1987 and prepared per American Society for Testing and Materials [ASTM] D 2343) had an ultimate tensile strength of 3.74 ± 0.19 gigapascal (GPa) (542 ± 28 kilopound per square inch [ksi]); the epoxy matrix was LRF-092 resin. Unidirectional 3817 denier T1000 and 3775 denier IM7 12,000-filament composite tows had an ultimate tensile strength of 6.81 ± 0.37 GPa (988 ± 53 ksi) and 3.96 ± 0.31 GPa (575 ± 45 ksi), respectively; the epoxy matrix was UF3323-102 resin from TCR Composites (Ogden, UT). Each tow specimen had 25 × 51-mm (1 × 2-in.) cardboard end tabs with spacers on the outside edge (figure 1, left). Tow ends were secured inside the end tabs with a fast-setting epoxy. This tabbing procedure reduced the amount of tow pullout and crushing (fewer grip failures) and reduced grip noise. IM7 unidirectional composite tow results were then compared to a 15.7-cm- (6.2 in.)-diameter cylindrical IM7 COPV with an aluminum liner, manufactured from the same IM7 material-of-construction (same carbon fiber spools, epoxy matrix, cure cycle, and fiber volume fraction).

AE measurements were taken using a DWC FM-1 system (Digital Wave Corp., Centennial, Colorado). Each channel was connected to a DWC PA-0, 0 dB gain preamplifier. Tabbed specimens were configured with four DWC B1080 piezoelectric sensors (50 kilohertz [kHz] to 1.5 megahertz [MHz] frequency range) positioned approximately 4 cm (~ 1.6 in.) apart (figure 1, right).

Intermittent load hold (ILH) tensile stress profiles (figure 2) based on the pressure tank examination procedure described in ASTM E 1067 (also referred to as the manufacturer's qualification test in ASTM E 1118) were applied. Accumulated composite damage was monitored by the decrease in the Felicity ratio (*FR*), given by:

$$FR = \frac{load\ at\ onset\ of\ significant\ AE}{previous\ maximum\ load}$$

Fast Fourier transforms (FFTs) were obtained using DWC WaveExplorer™ software. FFTs were exported to Microsoft Excel® (Microsoft Corporation, Redmond, Washington), 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). Frequency ranges were assigned to micromechanical damage mechanisms per de Groot's "Real-time Frequency Determination of Acoustic Emission for Different Fracture Mechanisms in Carbon/Epoxy Composites" as follows: matrix cracking, 90 to 190 kHz; fiber pullout and debonding, 190 to 300 kHz, and fiber breakage, > 300 kHz.

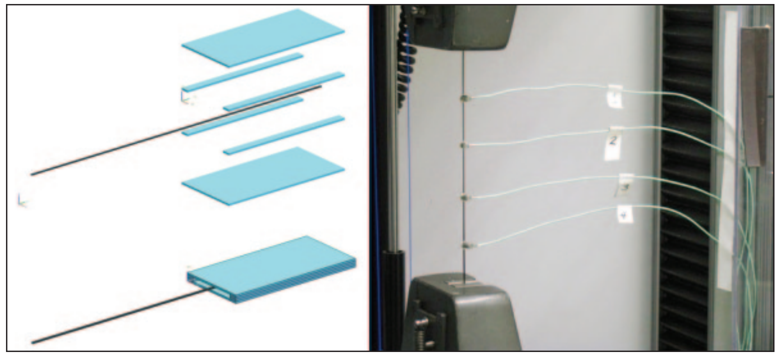

Fig. 1. *Cardboard end tabs and a mounted carbon epoxy tow specimen showing four B1080 acoustic emission sensors.*

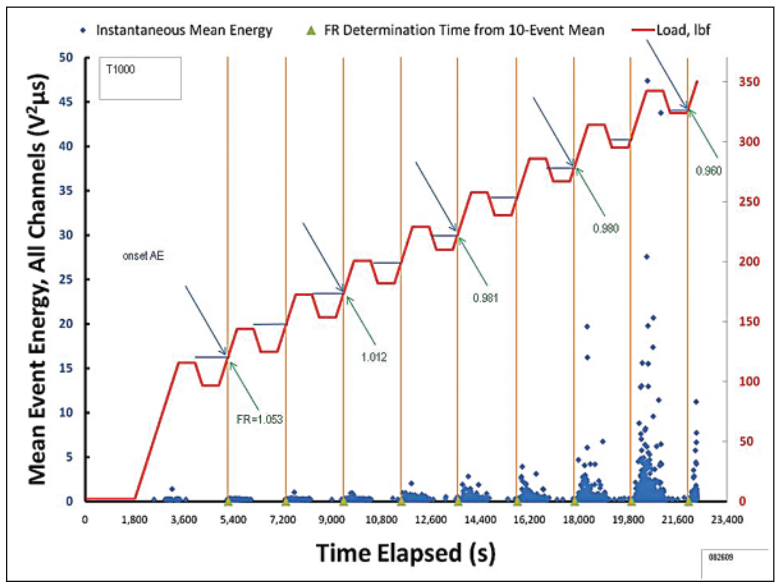

Fig. 2. *Representative intermittent load hold stress schedule used for T1000 carbon epoxy tow. Right y-axis units are in lbf.*

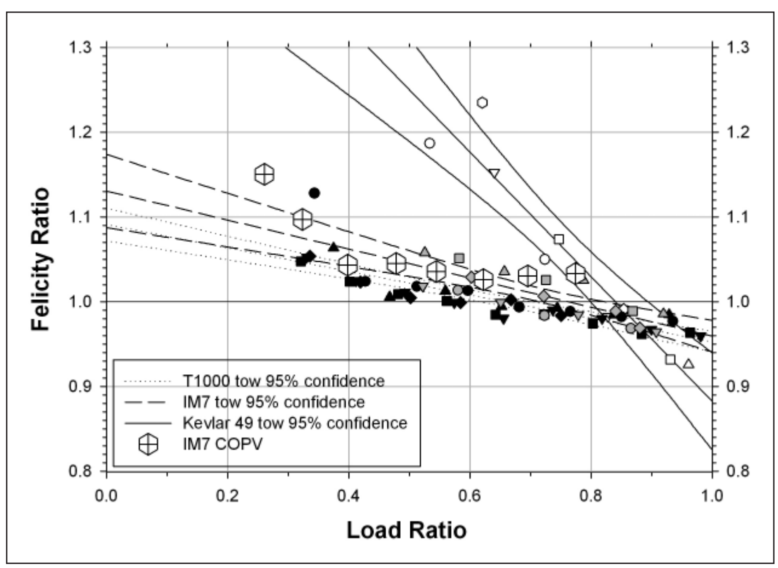

Fig. 3. *Least squares fits and 95% confidence intervals.*

Results and Discussion

The most significant finding was the observation of linear decreases in the *FR* with increasing load during ILH testing. While acceptable linearity ($R^2 > 0.9$) was obtained on K/Ep tow by using the first significant AE event to determine onset of significant AE, acceptable linearity could only be obtained on C/Ep tow using the first five or more significant events. All K/Ep, all C/Ep IM7, and most (four of six) C/Ep T1000 tow specimens failed explosively. A new parameter, referred to as the critical Felicity ratio (*FR**), was developed, and is simply the extrapolated value of *FR* at failure. K/Ep tow gave an *FR** close to 0.88 (scatter not determined). Both IM7 and T1000 gave an FR* near 0.96 with an observed scatter between 1.2% and 1.4%. Based on the rate of progressive damage accumulation, T1000 was found to be more damage tolerant than IM7, and C/Ep (T1000 or IM7) was found to be more damage tolerant than K/Ep (figure 3).

By comparison, IM7 and T1000 gave an ultimate tensile strength with an observed scatter between 5.3% (T1000) and 7.9% (IM7), indicating that *FR* behaves more like a universal damage parameter, while the tensile strength is more sensitive to surface flaws and strength variation over the cross-sectional area. By analogy, COPV burst pressure would be expected to be more scattered than the *FR** for a given COPV population. For example, taking *FR* = 1 as the threshold for significant accumulated damage, a damage threshold of 743 newtons (N) (167 pounds force [lb_f]) was obtained for IM7, and 939 N (211 lb_f) for T1000. Similarly, the slope of the *FR* vs. load ratio (*LR*) line was flatter for T1000 than for IM7 (figure 3). K/Ep, although quieter than C/Ep, undergoes a steeper and more dramatic drop in *FR* upon loading and therefore is less damage tolerant. This seems counterintuitive (quieter = lower damage tolerance) until it is realized that the

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continued

AE events associated with K/Ep damage, while fewer, tended to be more energetic than those associated with C/Ep damage.

IM7 COPV data acquired using the same ILH profile as used in the above unidirectional tow tests were found to overlap IM7 tow data (figure 3) (Note: *FR*s were calculated the same way; i.e., the first 15 events were averaged). This overlap suggests that the IM7 COPV was trending toward failure in much the same way the IM7 tows were proven true in a subsequent two-part ILH test to burst (Nichols et al., this publication).

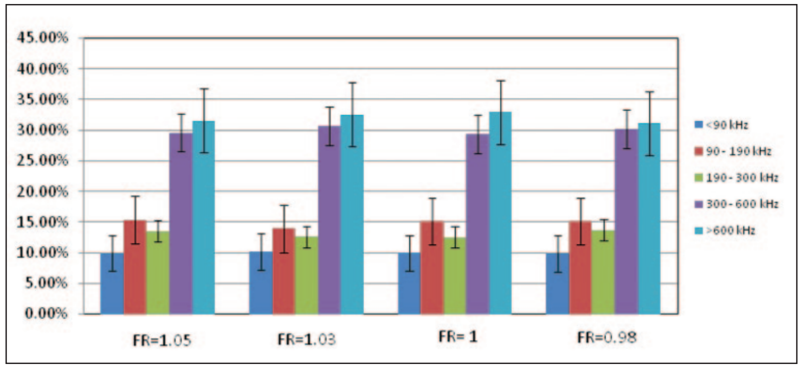

Fig. 4. Fast Fourier transform frequency distributions for IM7 Felicity ratio events at increasing levels of accumulated damage.

Waveform and Fast Fourier Transform Analysis

Three characteristic waveforms were identified in the C/Ep tow tests, each differentiated on the basis of amplitude, duration, and frequency: low amplitude signals with lower frequencies and short durations attributed to matrix cracking; moderate amplitudes with high frequencies and short durations attributed to fiber breakage; and high amplitudes with a wide range of frequencies and long durations attributed to concerted failure including all modes of micromechanical damage (most common).

In the case of uniaxial loading parallel to the fiber axis, microdamage attributable to tranverse matrix cracking would not be expected to be as relevant as microdamage attributable to fiber breakage. C/Ep FFT data show this is exactly what occurred in IM7 and T1000 tow specimens. More specifically, FFT analysis of the *FR* events responsible for the onset of significant AE (due to creation of new damage sites or growth of existing ones) during the ILH up ramps revealed that the frequency distribution for these events was invariant with respect to applied load (figure 4). Also, the frequency distribution noted for T1000 (data not shown) was essentially the same as for IM7. For example, fiber breakage (> 300 kHz) was the most predominant failure mode for both IM7 and T1000 at all applied loads. Lastly, T1000 had slightly more low-frequency (90-190 kHz) damage associated with matrix cracking than IM7, perhaps due to the higher fiber strengh of the T1000, thus causing stresses to be localized more within the matrix at equivalent loads.

Summary

Linear decreases in *FR* were observed with increasing stress during ILH testing of K/Ep tow, C/Ep T1000 and IM7 tow, and an IM7 COPV. Tests on C/Ep tow further showed that *FR** behaves like a universal damage parameter that exhibits less scatter than the tensile strength or, analogously in the case of a COPV, the burst pressure. An FFT analysis of the *FR* events responsible for the onset of significant AE revealed that the frequency distribution was invariant with respect to applied load, hence the amount of accumulated damage. These observations lay the groundwork for using both the *FR* and accumulated amount of composite damage (e.g., fiber damage) determined from FFTs as analytical accept-reject criteria for composite materials and components subjected to periodic stress over their service lifetime (e.g., cyclic loading for structural composites, or refill cycles for COPVs).