Acoustic Emission and Microscopic Characterization for Improved Composite Overwrapped Pressure Vessel Health Monitoring

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We are currently evaluating composite overwrapped pressure vessels (COPVs) to better quantify their reliability and likelihood of failure due to creep rupture, impact damage, and age-dependent issues. More specifically, NASA White Sands Test Facility (WSTF) is focusing attention on the Kevlar® fiber reinforced epoxy matrix (K/E) COPVs used on the space shuttle and on the carbon fiber reinforced epoxy matrix (C/E) COPVs used on the International Space Station. Together, these COPVs have varying criticality, time at pressure, number of pressure cycles, and damage and repair histories.

A group of nondestructive evaluation (NDE) experts is evaluating appropriate NDE methods for COPV health monitoring. One method showing particular promise in characterizing the failure of K/E and C/E composite materials is acoustic emission (AE). We have developed modal AE (mAE) techniques to correlate microscopic AE events such as matrix cracking and filament rupture with their corresponding mAE signatures. Of interest and reported here is the correlation of strain rate data with mAE data during progressive creep rupture degradation.

WSTF performed tensile creep rupture investigations on two COPV composite materials of construction: impregnated K/E strand (1140 denier Kevlar® 49), and impregnated C/E strand (12K T1000 and IM-7). As a control, the facility also performed investigations on unimpregnated carbon fiber tow (12K T1000). All tests were conducted under ambient conditions using two methods: an in-house accelerated stepped stress method (SSM), and ASTM [American Society for Testing and Materials] D 7337 *Standard Test Method for Tensile Creep Rupture of Fiber Reinforced Polymer Matrix Composite Bars*. We investigated the test results microscopically and by environmental scanning electron microscopy (ESEM) (figure 1).

A full description of the testing, including details of the test configuration and specimen preparation, is available on the NASA NDE Working Group Website, http://nnwg.org/current/, under White Sands Test Facility ("In-Situ NDE Characterization of Kevlar and Carbon Composite Micromechanics for Improved COPV Health Monitoring").

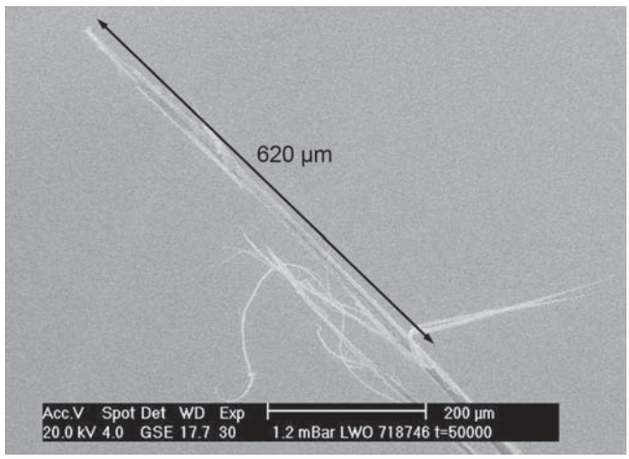

Fig. 1. *Post-creep rupture ESEM micrograph of a failed Kevlar® 49 filament.*

Determination of the ultimate tensile strength (UTS) allowed the load ratio (LR) to be calculated during SSM and ASTM D7337 creep tests. In the SSM case, the applied stress was stepped from LR = 0.6 to LR = 1.0 in 0.1-LR increments, each lasting 10,000 seconds. This allowed us to investigate the creep rupture behavior of the above materials of construction in an accelerated (overnight) test. In the ASTM D 7337 case, we subjected a representative specimen to a fixed LR of 0.8. Depending on the LR used, the expected creep rupture test duration could vary from 101 hours (high LR) to 104 hours (low LR). In both cases, we monitored strain and mAE activity simultaneously (ex.: figure 2). To elucidate microscopic and macroscopic changes, we also analyzed the specimens by optical microscopy, ESEM, and videography.

One of the interesting features observed for K/E strand specimens was that the strands did not always break in a single catastrophic event, but often in more than one partial rupture events. This may have important implications for the creep rupture behavior of K/E COPVs presently used in the shuttle fleet.

Creep data acquired for K/E using ASTM D 7337 (LR=0.8) demonstrated the feasibility of conducting 103- to 104-minute creep tests with AE sensors attached on creep specimens anchored by poured epoxy tabs, thus providing a straightforward way to estimate the creep rupture

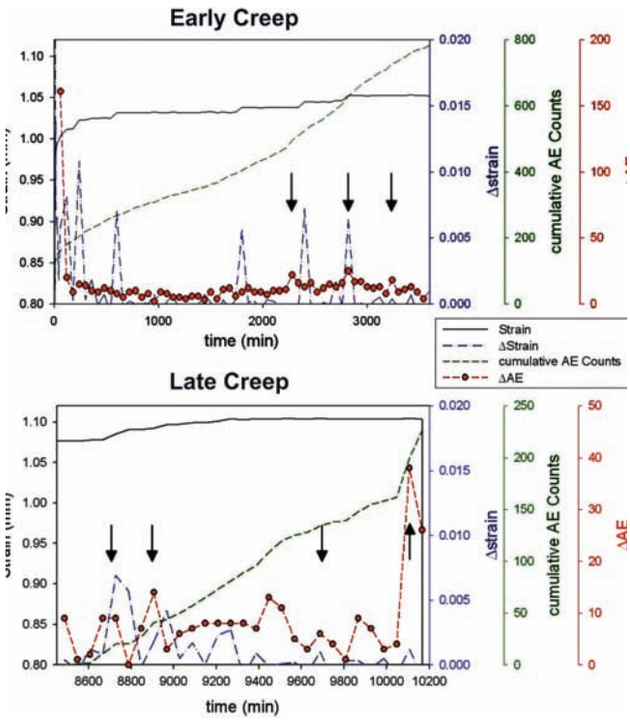

Fig. 2. *AE and strain data during early and late creep in a K/E fixed stress creep test.*

lifetime of COPV composite materials of construction. Due to time constraints, however, we performed most creep rupture tests using the quicker SSM approach.

ESEM images acquired at 20× magnification during SSM testing revealed very few changes aside from Poisson elongation and reduction of width, even at LR exceeding 0.9 relative to the UTS. ESEM at 500× revealed areas of higher electron contrast at LR = 0.92 that did not appear at lower LRs; however, more thorough ESEM analysis is needed to ensure these were not imaging artifacts.

Inspection after rupture during SSM testing of the K/E specimen clearly revealed a skin/core morphology in the Kevlar® filaments. Scientists observed fiber split lengths of 620 mm (about 50 times the 12-µm diameter (D) of a single Kevlar® filament) (figure 1). For a Kevlar 49 molecule, failure at chain ends within the core of a single filament would result in a fiber split length of approximately 150D; therefore, the 50D split lengths suggest chain scissioning is operative, which is expected for strands undergoing creep rupture at a LR > 0.7.

Optical microscopy at 60× magnification during testing of a K/E strand showed discernable macroscopic changes occurring during the SSM loading profile. Like the ESEM results, optical microscopy showed that creep damage is very subtle in terms of giving any indication of impending creep rupture, even during late creep.

Real-time mAE data acquired on a K/E strand during an ASTM D 7337 fixed-stress creep test revealed both an increase in the amplitude (energy) and in the number of events as we approached a critical creep rupture point (figure 2). Discrete creep events, as revealed by strain increases, often corresponded with increases in AE activity during early and late creep (see arrows in figure 2).

To date, WSTF has performed NDE on K/E strand, C/E strand, and carbon fiber tow. NDE measurements made so far include mAE, stress (load cell), strain (fiber Bragg grating and linear variable differential transformer), ESEM and optical microscopy, and low-magnification videography. An example of videography of an unimpregnated 12K T1000 carbon fiber tow appears in figure 3. The video frames show progression of creep rupture degradation from unloaded to post-rupture conditions. The explosive failure of the carbon tow was similar to the type of catastrophic and sudden failure observed in the K/E composite strand.

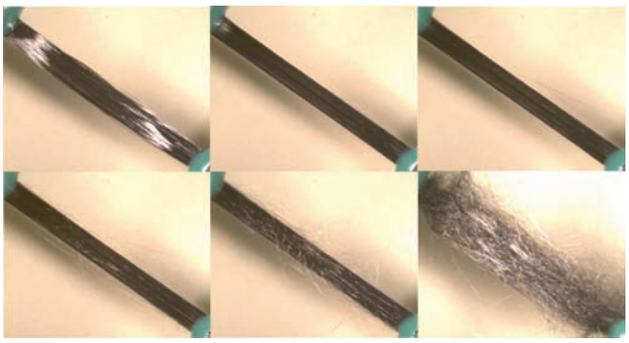

Fig. 3. *Creep rupture progression in an unimpregnated 12K T1000 carbon fiber tow.*

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