Update on the Progress of Hygrothermal Aging of Triaxial Braided Carbon/Epoxy Composites

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Material Fabrication

- **Materials**
  - **Fiber:** Torayca® T700S standard modulus carbon fiber
  - **Matrix Materials:** 4 ranging from brittle to toughened
    - Hexcel 3502
    - Cytec 5208
    - EPIKOTE Resin 862/EPIKURE Curing Agent W
    - Cytec PR520

- **Processing**
  - Resin transfer molding (RTM) for both resin and composite
    - Final cure at 350°F (177°C) for 2 hr
    - Resin glass transition temperature, $T_g \geq 300°F (149°C)$
  - 6 plies, $[+60°/0°/-60°]$ 2D triaxial braid preform
    - 24k axial tows, 12k bias tows
    - Equal fiber volume in all directions

- **Cured composite properties**
  - 0.125 in thick, 2’x2’ panel
  - ~56% fiber volume fraction
Hygrothermal Aging Cycle

Cure temperature, $T_c = 350^\circ$F (177$^\circ$C)

Minimum glass transition temperature, $T_g \sim 300^\circ$F (149$^\circ$C)

Runway hot/wet soak
85°F (29.4$^\circ$C) / 85% RH

250°F (121.1$^\circ$C)

$T_g - 50^\circ$F (27.8$^\circ$C)

Descent

Ascent

-65°F (-53.9$^\circ$C) at cruise

Temperature ($^\circ$C)

Time (Hours)
Composite Mechanical Property Test Methods

- ASTM D 3039 Tension
- ASTM D 3410 Compression
- ASTM D 7078 Modified V-Notch Rail Shear

ASTM D 7078 V-Notched Rail Shear “H” Specimen

Test plan limitations
- The number of tests per aging condition was limited by material availability
- The ASTM D 3039 test method does not provide an accurate measure of transverse tensile strength for braided composites
  - Used only to provide an indication of aging effects
  - Improved test methods are being developed
Ballistic Impact Test Method

Test method considerations

- Blunt impact allows large deformation before failure
- Simple method enables easier use in other labs
- 12 in X 12 in panel size provides efficient use of material
Previously Reported E862 Results
E862 Resin/Composite to 2 Years of Aging

• E862 Resin properties
  – Chemical structure
    • Surface oxidation identified
    • Limited continuation of cure identified
  – Physical properties
    • Glass transition temperature was not affected
    • Physical aging resulting in embrittlement and volume loss identified
  – Mechanical properties
    • Resin tensile strength reduced and ductile response eliminated

• Composite properties
  – Microcracking was observed
  – Mechanical properties
    • Tensile strength was not reduced
    • Compression strength was reduced
    • Shear had not yet been tested
  – Impact penetration threshold was not found to change
Post-cure effect of the first cycle causes an increase in plateau stress.

Aging causes a reduction in strain to failure.

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E862 Composite Microcracking

Microcracks visible on a painted surface

Overlaid image of braid architecture showing crack locations within fiber tows

Contrast enhanced X-ray CT images of microcracks in interior plies

0 cycles

217 cycles (4 months)

739 cycles (12 months)

739 cycles – different ply (12 months)
E862 Ballistic Impact Results

- No reduction in penetration threshold
- More brittle resin failure observed for the longest aging time

Impact Velocity (ft/sec)

- Penetrate
- Contain

No Aging
344 Cycles (6 months)
1149 Cycles (1.6 years)
New Mechanical Results

3502, 5208, E862, PR520
3502 Composite Mechanical Results (12 Months)

- Possible transverse tension strength reduction
- Shear strength reduction
- Compression strength reduction
- Likely due to observed microcracks initiating failure
- Aging to 24 months is ongoing
5208 Composite Mechanical Results (24 Months)

- Transverse tensile strength reduction (correlates with shear reduction because of failure mode)
- Shear strength reduction
- Compression strength reduction
- Likely due to observed microcracks initiating failure
E862 Composite Mechanical Results (24 Months)

- No significant tensile strength changes
- Compression strength reduction likely
- Possible shear strength reduction
- Changes are likely due to observed microcracks initiating failure
No significant strength changes observed
Very little evidence of microcracking was observed
Aging to 24 months is ongoing

Aged PR520/T700s Tensile Strength (MPa)

Aged PR520/T700s Compression Strength (MPa)

Aged PR520/T700s Shear Strength (MPa)
Summary of Mechanical Results

- Microcracking has been identified as the most likely cause of strength reduction in compression, shear, and transverse tensile strength.
- Previous work with E862 indicated that resin embrittlement can occur.
- Preliminary studies indicate that microcracking is occurring during the low temperature part of the cycle as a result of differences in thermal expansion coefficients of fiber and resin.
New Ballistic Impact Test Results

3502, PR520
Ballistic Impact Results: 3502

- No aging effect was observed.
Ballistic Impact Results: PR520

- No aging effect observed.
- Panel variation effect was observed.

![Cross Section Location](image)

![Graph](image)
Impact Damage and Axial Tow Nesting: PR520 Aged

- Nesting is observed by sectioning the panel near the edge, in line with the axial tows passing through the panel center.
- More damage is observed on the side opposite the impact in panels with less axial tow nesting.

540 ft/s
Back of Panel

536 ft/s
Back of Panel

524 ft/s
Back of Panel

515 ft/s
Back of Panel

○ Penetrate  ● Contain
Summary of Ballistic Impact Results

• Previous aging of the E862 showed little change in impact threshold.
• Additional results on 3502 and PR520 also show little change after 12 months of aging.
• Fiber tow nesting has an influence on impact damage and possibly impact threshold.
• Impact threshold does not correlate with static coupon tests
Conclusions

• Hygrothermal aging has resulted in small reductions in transverse tensile, shear, and compression strengths in E862, 3502, and 5208 composite materials.
• The primary mechanism is the development of microcrack damage that initiates failure in matrix/delamination failure dominated static tests, but not in the fiber failure dominated axial tensile test.
• The impact penetration threshold does not appear to be sensitive to the presence of pre-existing microcrack damage.
• Axial fiber tow nesting can influence the extent of damage following impact and the impact penetration threshold.
• PR520 composite was not observed to be affected by aging up to 12 months; minimal microcrack development was observed.
Continuing Work

• 3502 and PR520 Composite
  – Aging to 2 years
    • Tension
    • Compression
    • Shear
    • Impact

• PR520 Resin
  – Aging to 2 years
    • Tension
    • Compression
    • Shear
    • FTIR, DSC, DMA
The End.