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

![Diagram showing temperature and time for different phases of aircraft operations](image)

- **Runway hot/wet soak**: 85°F (29.4°C) / 85% RH
- **Minimum glass transition temperature**: $T_g \approx 300°F (149°C)
- **Cure temperature**, $T_c = 350°F (177°C)$
- **Descent**: 250°F (121.1°C)
- **Ascent**: $T_g - 50°F (27.8°C)$
- **-65°F (-53.9°C) at cruise**
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

R=1.5"

R=0.998"

1.95"
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
E862 Resin Tensile Properties

Post-cure effect of the first cycle causes an increase in plateau stress.

Aging causes a reduction in strain to failure.

- 54 Cycles
- 0 Cycles
- 108 Cycles

True Stress (MPa) vs. Strain (%)

The 2012 Aircraft Airworthiness & Sustainment Conference (AA&S 2012), 2-5 April, 2012, Baltimore, MD
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**

### Aging Times
- **No Aging** (0 cycles)
- **344 Cycles** (6 months)
- **1149 Cycles** (1.6 years)

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The 2012 Aircraft Airworthiness & Sustainment Conference (AA&S 2012), 2-5 April, 2012, Baltimore, MD
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
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
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.

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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.

Less Damage

More Damage

**A**

540 ft/s

**C**

524 ft/s

**B**

536 ft/s

**D**

515 ft/s

○ Penetrate  ● Contain

Back of Panel

Back of Panel

Back of Panel

Back of Panel
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.