Summary of the effects of two years of hygro-thermal cycling on a carbon/epoxy composite material

Composite materials are beginning to be used for structures in the fan section of commercial gas turbine engines. This paper explores the type of damage that could occur within one type of composite material after exposure to hygrothermal cycles (temperature/humidity cycles) that are representative of the environment in the fan section of an engine. The effect of this damage on composite material properties is measured. Chemical changes in the matrix material were limited to the exposed surface. Microcrack formation was identified in the composite material. This damage did not cause a significant reduction in tensile strength or impact penetration resistance of the composite material. Additional data is needed to assess the effect of damage on compressive strength.
SUMMARY OF THE EFFECTS OF TWO YEARS OF HYGRO-THERMAL CYCLING ON A CARBON/EPOXY COMPOSITE MATERIAL

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Material fabrication

- **Materials**
  - Fiber: Torayca® T700S standard modulus carbon fiber
  - Matrix: EPIKOTE Resin 862/EPIKURE Curing Agent W
  - 3 additional materials are currently being aged
- **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, $[0°/+60°/–60°]$ 2D triaxial braid preform
    - 24k axial tows, 12k bias tows
    - Equal fiber volume in all directions
- **Cured composite properties**
  - 0.125 in thick
  - 56% fiber volume fraction
Hygro-thermal aging cycle

Cure temperature, $T_c = 350^\circ F$

Minimum glass transition temperature, $T_g \sim 300^\circ F$

Runway hot/wet soak (85°F/85% RH)

Ascent

Decent

-65°F at cruise

-100 -50 0 50 100 150 200 250 300 350 400

Temperature (°F)

0 1 2 3 4 5 6 7 8 9 10 11 12

Time (Hours)
Aging test plan

• Resin properties
  – Chemical structure
    • Surface
    • Interior
  – Physical properties
    • Glass transition temperature
    • Volume loss (densification)
  – Mechanical properties
    • Tensile strength and fracture surface

• Composite properties
  – Microcracking
  – Mechanical properties
    • Tension
    • Compression
    • Shear (in-progress)
  – Impact penetration threshold
Color indication of resin aging

Color change in aged tensile specimens

Small changes in chemical structure can cause a large color change
ATR/FTIR test method for chemical analysis

- **Infrared spectroscopy**
  - Light passing through or reflected from a material is absorbed by each chemical group at a unique wavelength (or wavenumber)

- **ATR - Attenuated Total Reflectance**
  - Surface reflection technique that probes a surface layer ~0.5 – 5 µm thick

- **FTIR – Fourier transform infrared spectroscopy**

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Surface layers are sanded to expose interior material

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Example infrared spectrum

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The 2011 Aircraft Airworthiness & Sustainment Conference (AA&S 2011), 18-21 April, 2011, San Diego, CA
FTIR spectra for a specimen aged 271 cycles

Amide formation is an indication of chemical degradation near the surface.

Residual uncured epoxy rings disappear near the surface.

Spectra similar to unaged baseline.

Baseline (unaged surface)

Depth from surface (mm)

Wavenumber, cm\(^{-1}\)

Absorbance

Amide formation is an indication of chemical degradation near the surface.

Residual uncured epoxy rings disappear near the surface.

Spectra similar to unaged baseline.
Glass transition temperature test methods

Dynamic mechanical analysis (DMA)
- ASTM D4065-06 – test procedure (resins)
- ASTM D7028-07 – intercept method (composites)

**Test conditions**
- Single cantilever beam specimen
- Ramp rate = 5 °C/min

Differential scanning calorimetry (DSC)

**Heat Flow**
- Reference specimen
- Test specimen

**Test conditions**
- ~ 5-10 mg specimen mass
- Ramp rate 5 °C/min (modulates with 0.5°C amplitude at 0.025 Hz)

- Endotherm peak from rapid volume increase above Tg
- (Enthalpy calculated from peak area)
- Exotherm peak for additional cure (not observed in this study)

- T_g by intercept method

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The 2011 Aircraft Airworthiness & Sustainment Conference (AA&S 2011), 18-21 April, 2011, San Diego, CA
Glass transition temperature

Results for specimens tested monthly during aging

Low values are possibly caused by differences in moisture content.
Glass transition temperature

Results for specimens tested monthly during aging

- DMA test: No low values when all specimens are dry and tested consecutively.
- DSC test: Tg increases during the first ~50 cycles then remains constant.
Densification (volume loss)

Growth of the endotherm peak above $T_g$ in DSC curves is a result of densification during aging (also called physical aging).

DSC curves measured after various numbers of aging cycles

Volume loss

Growth of endotherm
Resin tensile properties

Postcure effect of the first cycle causes an increase in plateau stress

Aging causes a reduction in strain to failure

Embrittlement correlates with aging endotherm

Fracture surfaces

0 Cycles

54 Cycles

108 Cycles
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)
Composite mechanical property test methods

ASTM D 3039 Tension
ASTM D 3410 Compression
Modified V-Notch Rail Shear (in progress)

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

Edge initiated shear failure in a transverse tensile specimen
Tensile Strength

Axial

- No reduction in strength
- Transverse tensile specimens fail by edge initiated shear
- Improved transverse test methods are being developed

Transverse

<table>
<thead>
<tr>
<th>Aging Time (years)</th>
<th>0 cycles (Unaged)</th>
<th>739 cycles (12 months)</th>
<th>1364 cycles (24 months)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>100</td>
<td>739</td>
<td>1364</td>
</tr>
<tr>
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<tr>
<td>2.5</td>
<td>-0.5</td>
<td>0.5</td>
<td>1.5</td>
</tr>
</tbody>
</table>
Compression Strength

- More test specimens and reduced scatter are needed to assess aging effects
- Improved test methods are being developed

<table>
<thead>
<tr>
<th>Aging Time (years)</th>
<th>Tensile Strength (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>-0.5</td>
<td>0 cycles</td>
</tr>
<tr>
<td>0</td>
<td>739 cycles (12 months)</td>
</tr>
<tr>
<td>1</td>
<td>1364 cycles (24 months)</td>
</tr>
</tbody>
</table>

Axial

Transverse
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
Example of ballistic impact penetration threshold

**Contained**

**Penetrated**

526 ft/sec

541 ft/sec
Ballistic impact results

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

- No Aging
- 344 Cycles (6 months)
- 1149 Cycles (1.6 years)

Impact Velocity (ft/sec)

All Penetrated

All Contained

Penetrate
Contain
Conclusions

- Chemical changes in the resin material were confined to a region within 0.2 mm of the surface.
- Densification (volume loss) occurred during early cycles and remained constant during later cycles.
- Resin embrittlement occurred during early cycles and seemed to be correlated with densification.
- Microcracking occurred in both surface and interior plies, particularly at the longer aging times.
- Aging did not cause a reduction in tensile strength.
- The effect of aging on compressive strength could not be determined because of limited test specimens and large scatter in the data.
- Aging did not cause a reduction in impact strength, although a more brittle resin failure mode was observed.