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
- $T_g - 50^\circ F$
- -65°F at cruise
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

Color change is not limited to the surface
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

Example infrared spectrum
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):
- 0 (surface)
- 0.04
- 0.20
- 0.30
- 0.35

Wavenumber, cm⁻¹:
- 1653 cm⁻¹
- 1601 cm⁻¹
- 907 cm⁻¹
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)

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

Heat flow/unit mass
- $T_g$ by intercept method
- Endotherm peak from rapid volume increase above $T_g$
  (Enthalpy calculated from peak area)
- Exotherm peak for additional cure (not observed in this study)

The Glass transition temperature test methods include Dynamic Mechanical Analysis (DMA) and Differential Scanning Calorimetry (DSC). For DMA, the methods used are ASTM D4065-06 for test procedures on resins and ASTM D7028-07 for the intercept method on composites. The test conditions for DMA include a single cantilever beam specimen with a ramp rate of 5 °C/min. For DSC, a specimen mass of ~5-10 mg is used with a ramp rate of 5 °C/min that modulates with 0.5°C amplitude at 0.025 Hz. The glass transition temperature ($T_g$) is measured by the intercept method, and there is an endotherm peak due to a rapid volume increase above $T_g$. Additionally, an exotherm peak for additional cure is observed, which is not observed in this study.
Glass transition temperature

Results for specimens tested monthly during aging

Temperature (°C)

Cycles

DMA test

Low values are possibly caused by differences in moisture content

DSC test
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

Growth of endotherm

Volume loss

The 2011 Aircraft Airworthiness & Sustainment Conference (AA&S 2011), 18-21 April, 2011, San Diego, CA
Resin tensile properties

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

Aging causes a reduction in strain to failure.

Physical Aging Endotherm

Embrittlement correlates with aging endotherm.

Fracture surfaces

0 Cycles

54 Cycles

108 Cycles

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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
The 2011 Aircraft Airworthiness & Sustainment Conference (AA&S 2011), 18-21 April, 2011, San Diego, CA
Compression Strength

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

- 0 cycles (Unaged)
- 739 cycles (12 months)
- 1364 cycles (24 months)

Aging Time (years)

Tensile Strength (MPa)

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

Impact Velocity (ft/sec)

- Penetrate
- Contain

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

- All Penetrated
- All Contained
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