Effects of Moisture Diffusion in Sandwich Composites

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<u>Outline</u>

- I. Brief summary of a numerical study funded by Boeing in 2003
- II. Experimental studies at UW initiated in 2008
- III. Proposed AMTAS project

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- Assumptions:
 - 'Pristine' (undamaged) sandwich structure; moisture ingression solely due to diffusion
 - Core = Nomex honeycomb
 - Initial moisture content = 0%. This implies:
 - Initial out-of-autoclave (or out-of-hot-press) moisture content of composite face sheets = 0%
 - Initial out-of-autoclave (or out-of-hot-press) relative humidity within core region = 0%



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- <u>Overall objective</u>: Predict whether liquid water will accumulate in core region of 'pristine' sandwich structures following long exposure to 'realistic' service environments
- <u>Created a computer program called MOIST, based on:</u>
 - Fourier heat conduction equation \rightarrow used to predict through-thickness temperature profiles
 - Fick's diffusion equations → used to predict through thickness moisture content resulting from cyclic changes in external temperature and humidity
 - Clapeyron equations → used to predict dew point of water vapor in core (if current temperature < dew point, condensation occurs)

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• Overall Conclusions:

- Core humidity levels will increase with time
- Under realistic service conditions liquid water does not accumulate within core region of pristine structures (i.e., core will not fill with water)..... <u>but</u>
- (For transport aircraft flight profiles) as core humidity is increased water will condense-freeze-thaw-evaporate during ascent-cruise-descent cycle

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- Overall Conclusions:
 - Core humidity levels will increase with time...only question is: how long?
 - Under 'realistic' service conditions liquid water does not accumulate within core region of pristine structures (i.e., core will not fill with water)..... <u>however</u>
 - (For transport aircraft flight profiles) humidity increase implies water will condense-freeze-thaw-evaporate during ascent-cruise-descent cycle
- <u>Hypothesis</u>: Condense-freeze-thaw-evaporate cycle is detrimental (e.g., decrease in face sheet-core bond strength)

Predicting Moisture Diffusion *Typical Result: Constant Temperatures and Humidity*



Definition of a Cycle



1 Hydrothermal Cycle

Typical Analysis Cyclic Changes in Temperature and Humidity



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Experimental Studies at UW Initiated in 2008

Objectives:

- Measure relative humidity in core region of a flat sandwich panel exposed to constant temperature and humidity on both sides
- Compare measurements with predictions

Test Panel Fabrication



[0/45/90/-45]_s Gr/Ep facesheets:

- Hexcel prepreg w/ M46JB fibers and M70 epoxy resin
- 350°F cure system

Type 410 Nomex honeycomb core (0.50 in thick, 0.20 in cell size, 2-mil thick paper)



Test Panel Fabrication



Pocket for embedded humidity sensors and thermocouples milled in core

First facesheet bonded to one side of panel using thinfilm adhesive

Core sized to fit within aluminum frame



Test Panel Fabrication



Leadwires inserted through honeycomb and aluminum frame



Initial installation of embedded sensors

Test Panel Fabrication



Honeycomb 'caps' placed over instrumented sites

Leadwire passage in aluminum frame sealed with epoxy



Test Panel Fabrication



- Completed panel mounted in test chamber; exposure began on Aug 5
- Initial measurements (40°C = 104°F): Panel hum sensor 1: 25.0 %RH Panel hum sensor 2: 23.4 %RH (...higher than anticipated in '03...)



Measurements

Obtained between 5 Aug to 20 April = 258 days



Measurements vs Predictions If external conditions remain constant for ~ 2yrs



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 - Change in bending stiffness, El_{eff} (measure using 4-pt bend)
 - Change in G_I and G_{II} (measure using methods being developed by Adams et al @ Univ of Utah)

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 - 8 specimens (4 autoclave, 4 hot press): cycled between room temp and -55°C, then measure properties

- Require about 12-18 mos to complete
- Good MS Thesis Project

Mechanical Engineering

Backup Slides

Temperature predictions

 Through-thickness temperature distribution assumed to be governed by the Fourier heat conduction equation:

$$\frac{\partial Q}{\partial t} = -K_z A \frac{\partial T}{\partial z}$$

- where: $\partial Q / \partial t$ = heat transfer rate
 - K_z = thermal conductivity (z direction)
 - A = area
 - T = temperature
 - t = time
- Material properties allowed to vary through thickness; heat conduction equation solved numerically using finite-differences

Core Thermal Conductivity

- *K*_{core} estimated using rule-of-mixture approach
- Volume fractions of air and paper within the core calculated using hexagonal repeat unit



Core Thermal Conductivity

Given the cell size (c), paper ribbon thickness (w), and core thickness (t), it can be shown that the volume fractions are given by:



Core Thermal Conductivity

$$K_{core} = (V_{air})(K_{air}) + (V_{paper})(K_{paper})$$

Example:

Honeycomb core with 0.20 in cell size, produced using 2-mil thick DuPont Type 410 Nomex paper:

 $K_{paper} = 0.715 \text{ BTU-in/hr-ft}^2 \text{-}^{\circ}\text{R}$ $K_{air} = 0.166 \text{ BTU-in/hr-ft}^2 \text{-}^{\circ}\text{R}$

Calculated quantities:

 $V_{paper} = 0.027$ $V_{air} = 0.973$

$K_{core} = 0.181 \text{ BTU-in/hr-ft}^2 \text{-}^{\circ}\text{R}$

* Nomex properties: http://www.matweb.com Air properties: *Marks' Standard Handbook for Mechanical Engineers*, 8th Ed (1978)

Typical Properties

Material	Thermal Conductivity, K (BTU-in/hr-ft ² -°R)	Thickness (in)
Graphite/Epoxy	4.0*	0.005 (ply)
Honeycomb Core	0.181	0.50

* <u>Note</u>: Typical through-thickness K for Gr/Ep is listed; in-plane K values typically > 400 BTU-in/hr-ft2-^oR

Predicting Moisture Diffusion

 Through-thickness (1-D) diffusion of moisture assumed to be governed by Fick's first and second laws:

$$\phi = D_z \frac{\partial c}{\partial z} \qquad \qquad \frac{\partial c}{\partial t} = \frac{\partial}{\partial z} \left[D_z \frac{\partial c}{\partial z} \right]$$

 ϕ = rate of diffusion ("moisture flux"): units = mass/(area * time) c = concentration : units = (mass/volume)

$$D_z = diffusivity : units = area/time$$

z = direction of diffusion : unit = length

t = time

Predicting Moisture Diffusion

 From an experimental point of view it is easier to deal with percent moisture by weight (*M*), rather than the concentration of moisture (*c*). Fick's first and second laws are restated as:

$$\phi = \frac{D_z \rho}{100} \frac{\partial M}{\partial z} \qquad \qquad \frac{\partial M}{\partial t} = D_z \frac{\partial^2 M}{\partial z^2}$$

 $\rho = \text{density, mass/volume}$ M = "moisture content" $M = \frac{(\text{current weight}) - (\text{dry weight})}{(\text{dry weight})} \times 100\%$

Predicting Moisture Diffusion

 Temperature dependency of diffusion coefficient for solids (i.e., ply and core paper) assumed to follow a Arrhenius-type relationship:

$$D = D_o \exp\left(-\frac{E}{T}\right)$$

where: D_o , E = known material constants (differ for ply and core paper) T = absolute temperature

Predicting Moisture Diffusion

 Temperature dependency of diffusion of H₂0 vapor in air assumed to follow a power law of the form*:

$$D_{air} = 0.03376 \left(\frac{T(^{\circ}R)}{491.67(^{\circ}R)}\right)^{1.81} \frac{in^2}{\sec}$$

* Massman, W.J., *Atmospheric Environment*, Vol 32 (6), pp 1111-1127 (1998).

Predicting Moisture Diffusion Estimated Core Density and Diffusivity

$$\rho_{core} = (V_{air})(\rho_{air}) + (V_{paper})(\rho_{paper})$$

$D_{core} = (V_{air})(D_{air}) + (V_{paper})(D_{paper})$

Predicting Moisture Diffusion

The moisture content (*M*) of any surface layer in contact with air can be related to the relative humidity according to (Springer, 1980):

$$M = M_u \left(\frac{\% RH}{100}\right)^b$$

- constant M_u = material property
- exponent b = 1 for most materials
- relationship used to define the boundary condition at all ply interfaces

Predicting Moisture Diffusion

- Preceding relations allows forward-difference solution to Fick's equations; summary
 - (At all interior ply interfaces) moisture flux leaving ply k must equal moisture flux entering ply k+1

• (Boundary conditions):
$$M = M_u \left(\frac{\% RH}{100}\right)$$

- (Initial conditions): Initial through-thickness moisture content assumed uniform (assumed = zero in '03)
- Time step increment of 1 minute

Properties Used in '03

Property	Gr/Ep (typical values)	Type 410, 2-mil Nomex (www.matweb.com)
D _o	0.010 <i>in²/sec</i>	0.006 <i>in²/sec</i>
E	10300 °R	9000 °R
M _u	0.02	0.03
Density, ρ	0.054 <i>lbm/in</i> ³	0.026 <i>lbm/in</i> ³

<u>*Note*</u>: Properties reported for Gr/Ep vary widely. For example:

 $0.005 < D_o < 0.040 \text{ in}^2/\text{sec}$

Predicting Condensation Having calculated the moisture content and temperature within core following Step 1, then

1. Calculate relative humidity within core volume (based on rule of mixtures approximation)

$$\% RH = M_{core} / M_{u}^{core} = \left(\frac{M_{core}}{V_{air} + M_{u}^{pap} V_{pap}}\right)$$

Predicting Condensation (cont'd)

2. Use Clapeyron equation to estimate saturated vapor pressure (P_{svp}) at Step 1 core temperatures (T_{cor}^{s1})

$$P_{svp} \approx P_{svp}^{ref} \exp\left[\frac{h_{fg}(T_{cor}^{s1} - T^{ref})}{(RT^{ref})T_{cor}^{s1}}\right]$$

 T^{ref} = ref temperature (e.g., T^{ref} = 70°F = 529.67°R) P_{svp}^{ref} = saturated vapor pressure at T^{ref}

 $= 0.3632 \text{ psi at } T^{ref} = 529.67^{\circ}R$

 h_{fg} = enthalpy of vaporization at T^{ref}

= 1054 BTU/lbm = 820E3 ft - lbf / lbm at $T^{ref} = 529.67^{\circ}R$ R = gas constant for water vapor = 85.76 ft - lbf / lbm - R

Predicting Condensation (cont'd)

3. Calculate partial pressure of water vapor at Step 1 temperatures:

$$P_{pp} = (\% RH)(P_{svp})$$

Predicting Condensation (cont'd)

4. Calculate dew point temperature based on reference temperature and partial pressure at step 1 temperatures:

$$T_{dewpt} = \frac{h_{fg}T^{ref}}{h_{fg} - \ln\left(\frac{P_{pp}}{P_{svp}^{ref}}\right)RT^{ref}}$$

Predicting Condensation (cont'd)

5. Finally, condensation is predicted if core temperatures during step 2 become lower than calculated dew point temperature

$$T_{cor}^{s2} < T_{dewpt} \Longrightarrow$$
 Condensation

Experimental Measurements



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← Assembled Test Stand

 $\begin{array}{c} \text{Stainless-Steel} \\ \text{Chamber} \rightarrow \end{array}$





← Temperature Controller

Insulated Water Bath \rightarrow



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- Chamber instrumented with:
 - 2 type K thermocouples
 - 2 Ohmic Instruments Model HC-610 capacitive humidity sensors:
 - 5-95 %RH
 - -40 to 185°F operating range
- Test begun on 5 Aug:
 - Temp controller set @ 40°C (104°F)
 - Insulated bucket filled with distilled water (~2 liters added every 48 hrs since)
 - Data recorded every 30 mins (using Labview)

HC-610 Thermoset polymer capacitive humidity sensor. Hybrid electronics. Linear output. Range 5 to 95 %RH 2%. Temp. – 40 to 185 °F. Supply voltage 4.0 - 5.8 VDC



PDF Man/Instructions

www.ohmicinstruments.com/





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- As of 26 Jan (174 days = 5.8 mos after test initiation):
 - Ave chamber temp: 39.8 ± 0.50 °C
 - Ave chamber %RH: 54.8 ± 2.8%
- Chamber temperature and relative humidity for a typical 48-hr period plotted at right

data from: 7:50 am on 3 Nov to 7:50 am on 5 Nov





Typical Analysis Constant Temperatures, Non-uniform Humidity

- 12-ply Gr/Ep inner and outer facesheets
- 0.50 in honeycomb core with 0.20 in cell size
- Outer: T=90F; RH = 100% (constant)
- Inner: T=90F; RH = 0% (constant)



Typical Steady-State Temperature Profiles

- 12-ply Gr-Ep facesheets (0.060 in thick)
- Nomex honeycomb core (0.50 in thick)
- <u>Step 1:</u> Inside temp = 70F Outside temp = 85F

Step 2: Inside temp = 65FOutside temp = -65F

