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JAMS

# VARTM Variability and Substantiation

D. Heider, C. Newton, J. W. Gillespie



June 21<sup>st</sup> 2006

**UNIVERSITY OF DELAWARE**  
**CENTER FOR COMPOSITE MATERIALS**  
*INTERNATIONALLY RECOGNIZED EXCELLENCE*



The Joint Advanced Materials and Structures Center of Excellence



# FAA Sponsored Project Information

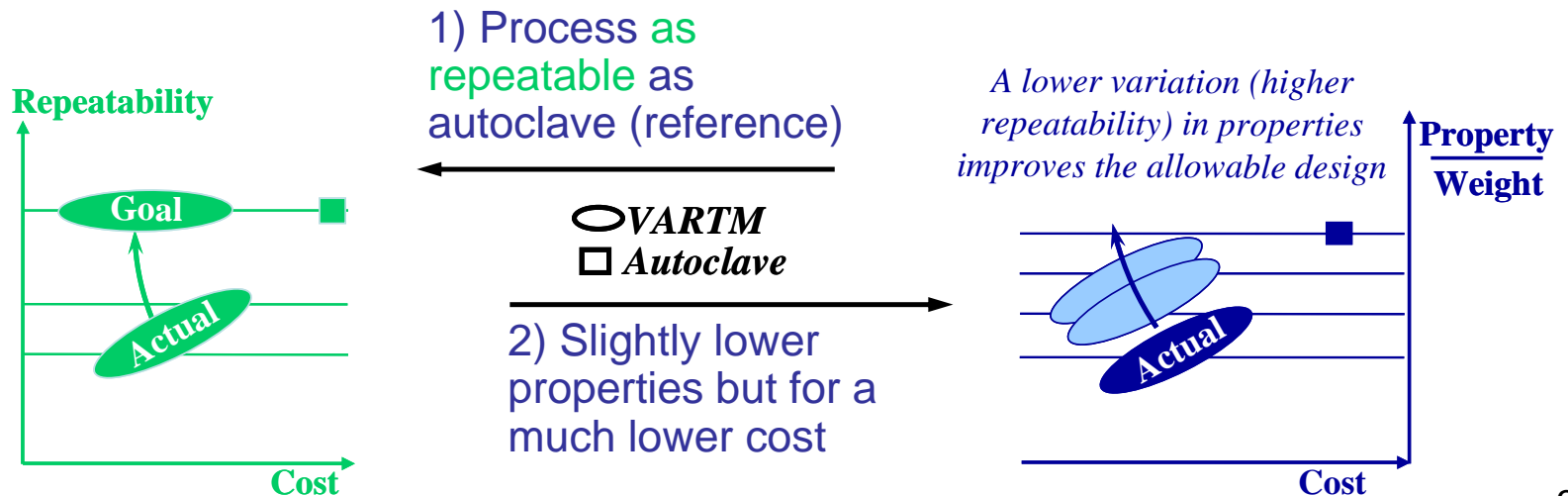


- Principal Investigators & Researchers
  - Dirk Heider
  - John W. Gillespie, Jr.
  - Crystal Newton
- FAA Technical Monitor
  - Curtis Davies
- Industry Participation
  - Gore (Munich, Germany)
    - Provided membrane materials, access to instrumentation and technical input
  - Hexcel (Seguin, Texas)
    - Provided resin and fabric material and technical input
  - Cytec (Anaheim, CA)
    - Provided resin and fabric material and technical input
  - EADS (Augsburg, Germany)
    - Provided technical and financial input
  - Boeing (Philadelphia, PA)
    - Provided technical input



# MOTIVATION

- VARTM process: +/-
  - Main advantages: **low cost, high fiber volume fraction, large scale parts**
  - Still some limitations
    - High variability compared to autoclave process
      - From part to part
      - In the same part
- Following conditions have to be met to make VARTM viable for high-performance aerospace applications:





# APPROACH



- Three VARTM processes will be evaluated on process repeatability, part quality, and mechanical performance
- Establish the fundamental understanding of the membrane/resin interaction
- Conduct model experiments to understand infusion and post-infusion stages of various VARTM processes
  - Implement novel characterization equipment
  - Model the thickness variation
  - Model the void formation
- Establish an elevated temperature VARTM workcell for toughened epoxies

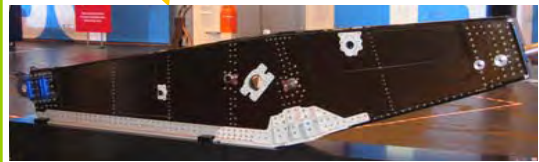


# VARTM Process Variations

1. Seemans Resin Infusion Molding Process (SCRIMP)
  - Use of Distribution Media
  - Patent held by TPI Inc.
2. Vacuum-Assisted Processing (VAP)
  - Use of an additional membrane
  - Patents held by EADS
  - Reduces Void Content, Improves Process robustness
3. Controlled Atmospheric Resin Infusion Process (CAPRI)
  - Reduced pressure differential
  - Patent held by the Boeing Co.
  - Reduces thickness gradient, improves fiber volume fraction variation



# AEROSPACE VARTM'D COMPONENTS



Flap tracks for the A380



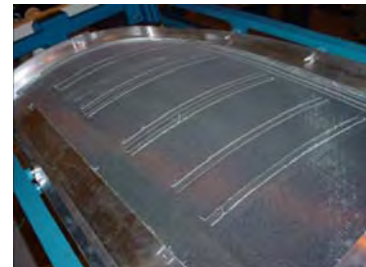
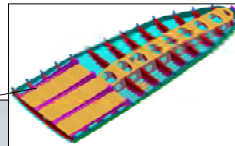
Pressure Dome



CH-47 Chinook Forward Pylon



A400M CFC Cargo Door



C-17 Main Landing Gear Door



Other BOEING Components

- LAIRCOM panels
- Leading edge 787
- Rear Bulkhead 787



# Liquid Injection Molding Simulation



- **Developed since 1987**
  - Simulation of Liquid Composite Molding (LCM) processes such as
    - Resin Transfer Molding (RTM)
    - Vacuum-Assisted Resin Transfer Molding (VARTM)
  - Finite Element Model (FEM) allows
    - Simulation of large-scale complex structures
    - Optimization of injection and vent locations
    - Study of dry-spot and high void content areas
    - Integration of various materials (fabric, core, metal meshes, etc.)
    - Simulation of cure behavior
- **Successful virtual VARTM product development examples include**



**BOEING**

**CYTEC**

*Technology ahead of its time™*



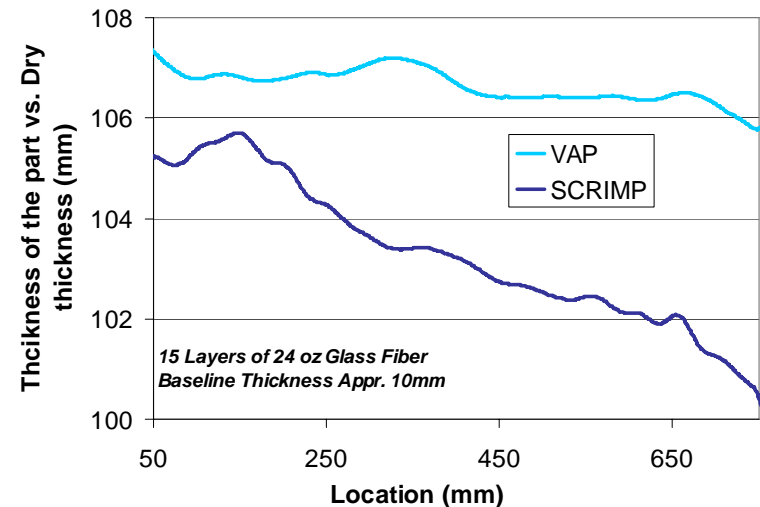
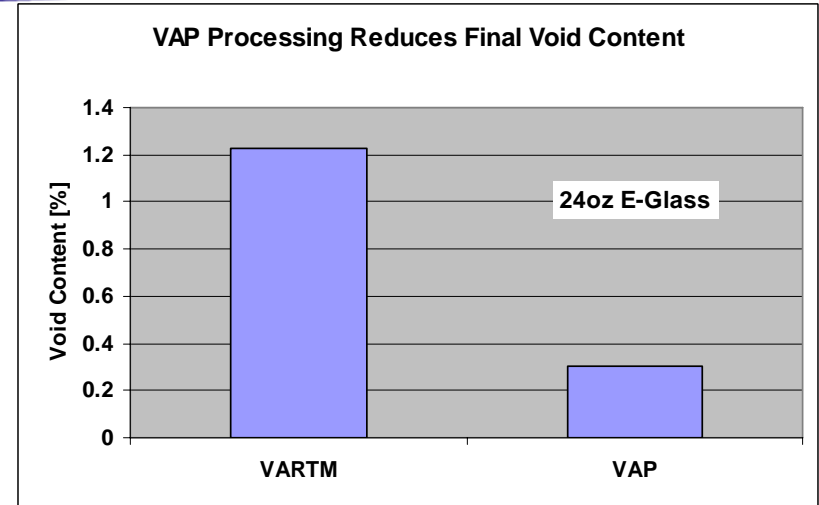
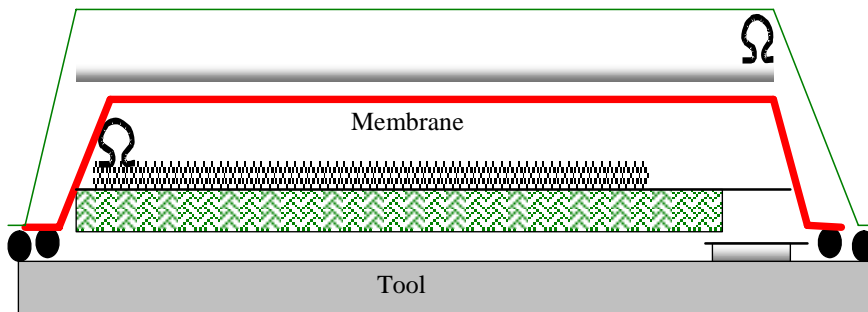
**AMTECH CORPORATION**

**GENERAL DYNAMICS**  
Land Systems

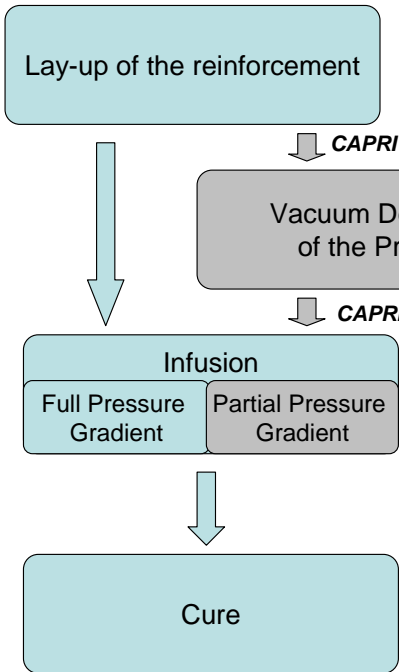


# MEMBRANE-BASED VARTM PROCESSING (VAP)

- Utilize membrane cover to allow continuous degassing and uniform vacuum pressure during VARTM processing
  - Reduces void content
  - Improves uniformity (fiber volume fraction, thickness)
  - Eliminates dry-spots







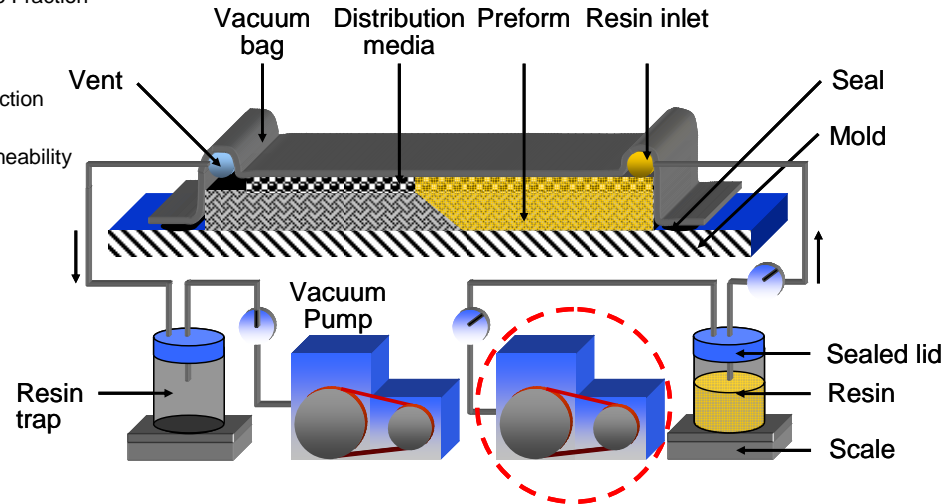
- Controlled Atmospheric Pressure Resin Infusion
- Adds vacuum debulking and reduce pressure differential to setup

### Advantages

- Increased Fiber Volume Fraction
- Reduced Gradients
  - Pressure
  - Thickness
  - Fiber Volume Fraction

### Disadvantages

- Decrease in fabric permeability
- Increase in flow times
- Increase in lead length



## CAPRI Patent held by Boeing

Woods, J., Modin, A. E., Hawkins, R. D., Hanks, D. J., "Controlled Atmospheric Pressure Infusion Process", International Patent WO 03/101708 A1.

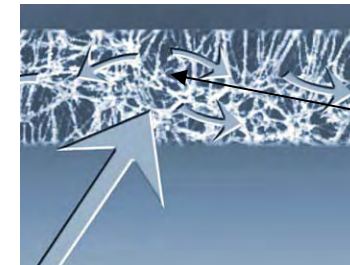
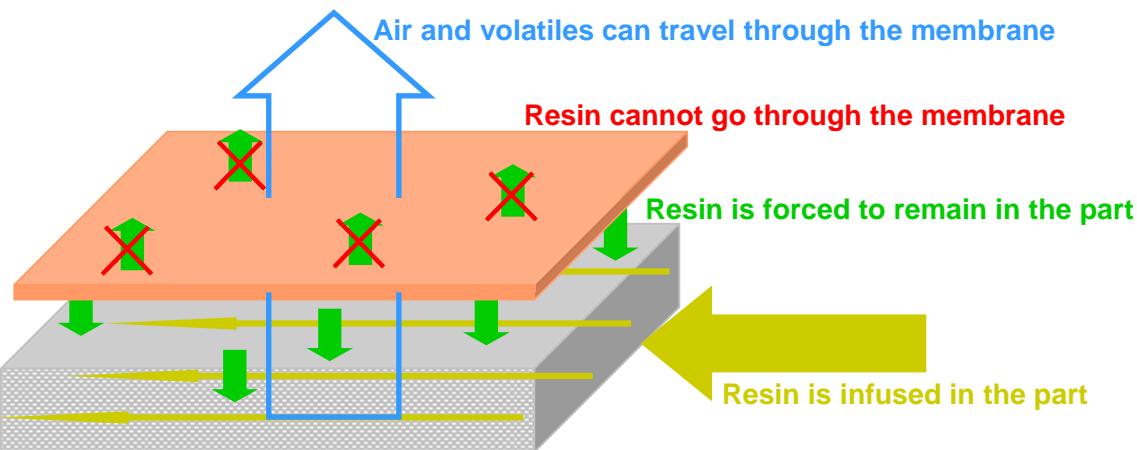
# MAIN REQUIREMENTS OF THE MEMBRANE

## •Desirable Characteristics for a membrane used in VARTM:

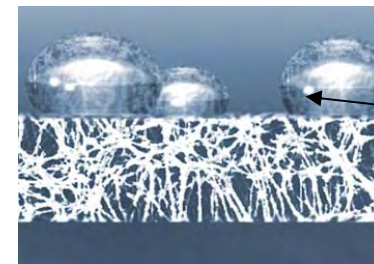
- Gas permeable material
  - OR High air permeability through the thickness
- Resin-proof material
  - OR Low liquid/resin permeability through the thickness

## •Compatibility with resin

- Compatible: The resin does not go through the membrane and is forced into the part
- Incompatible: The resin penetrates the membrane



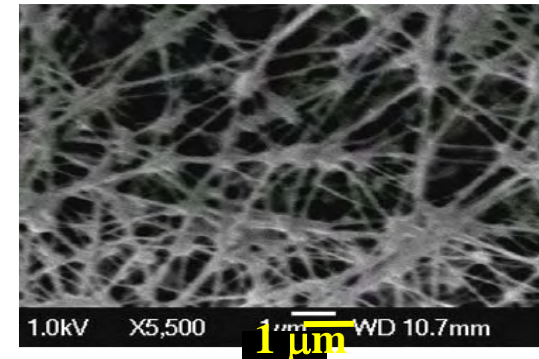
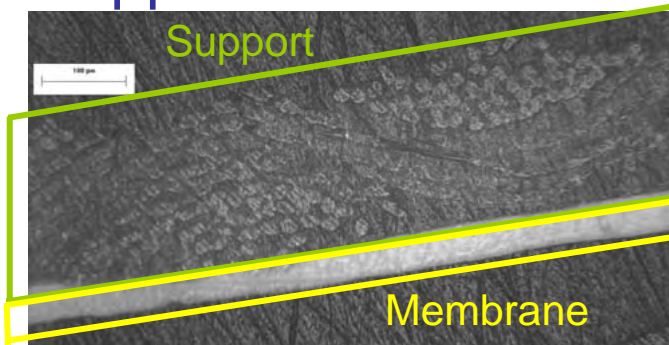
[www.gore-tex.co.uk](http://www.gore-tex.co.uk)





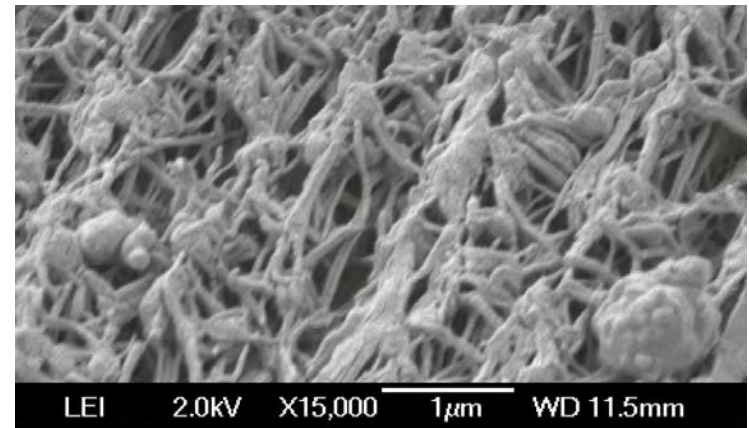
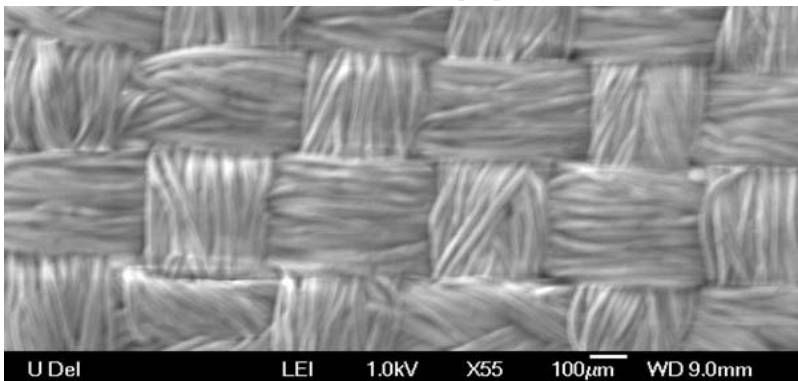
# Membrane (from W. L. Gore & Associates, GmbH)

- Optical microscope
  - The membrane is mounted on a support
- SEM of the membrane
  - Top surface



- Cross-section

- SEM of the support



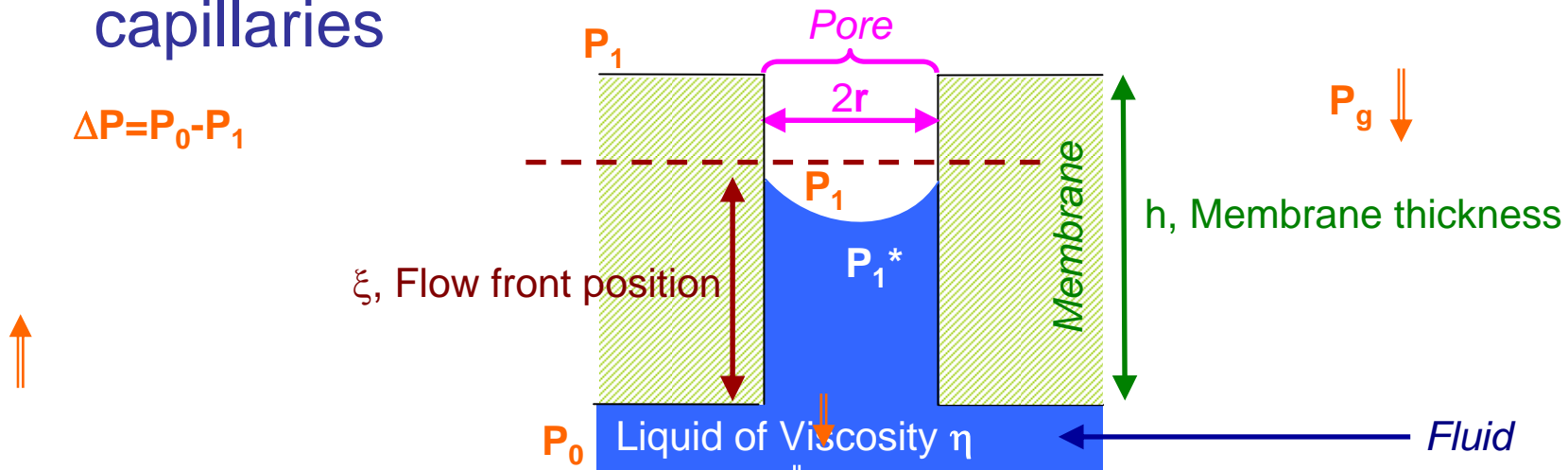


# Modeling of Resin Transport Through Membrane

- Goal

- Predict the impregnation time of the membrane by the resin to make sure that:  $t_{\text{impregnation}} \geq t_{\text{gel}}$

- Geometry: thin, straight and cylindrical pores, called capillaries



- Forces acting on the system

- Vacuum applied during the VARTM process ( $\leq 10^5$  Pa)
- Capillary effect
- Gravity force



# Expression of the Pressures

- Gravity:  $P_g = \rho g \xi$

- Pressure in the fluid below the meniscus  $P_1^*$ :

$$P_1^* = P_1 + P_c$$

- And  $P_c = -\frac{2\gamma \cos \theta}{r}$  (Laplace Equation), so:

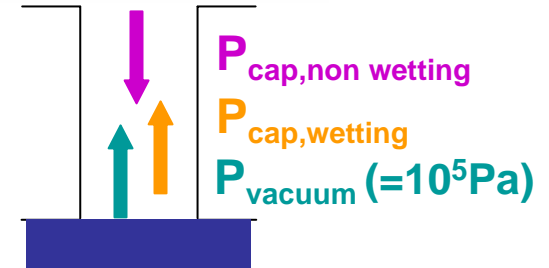
$$P_c = -\frac{2\gamma \cos \theta}{r}$$

$$P_1^* = P_1 - \frac{2\gamma \cos \theta}{r}$$

- $P_c$ : capillary pressure (Pa)
- $\gamma$ : resin surface tension (N/m)
- $\theta$ : contact angle membrane/resin of interest
- $r$ : pore radius (m)
- $\rho$ : resin density ( $\text{kg/m}^3$ )
- $g$ : gravitational acceleration ( $\text{m/s}^2$ )
- $\xi$ : position of the flow front in the pore (m)

# Capillary pressure vs. nanoscale

- From the capillary pressure expression, interesting information about pore size vs. wetting characteristics can be provided



Wetting fluid  $\Leftrightarrow \theta < 90^\circ$

$\Rightarrow P_{cap} > 0 \Rightarrow$  Spontaneous wetting

NON-Wetting fluid  $\Leftrightarrow \theta > 90^\circ$

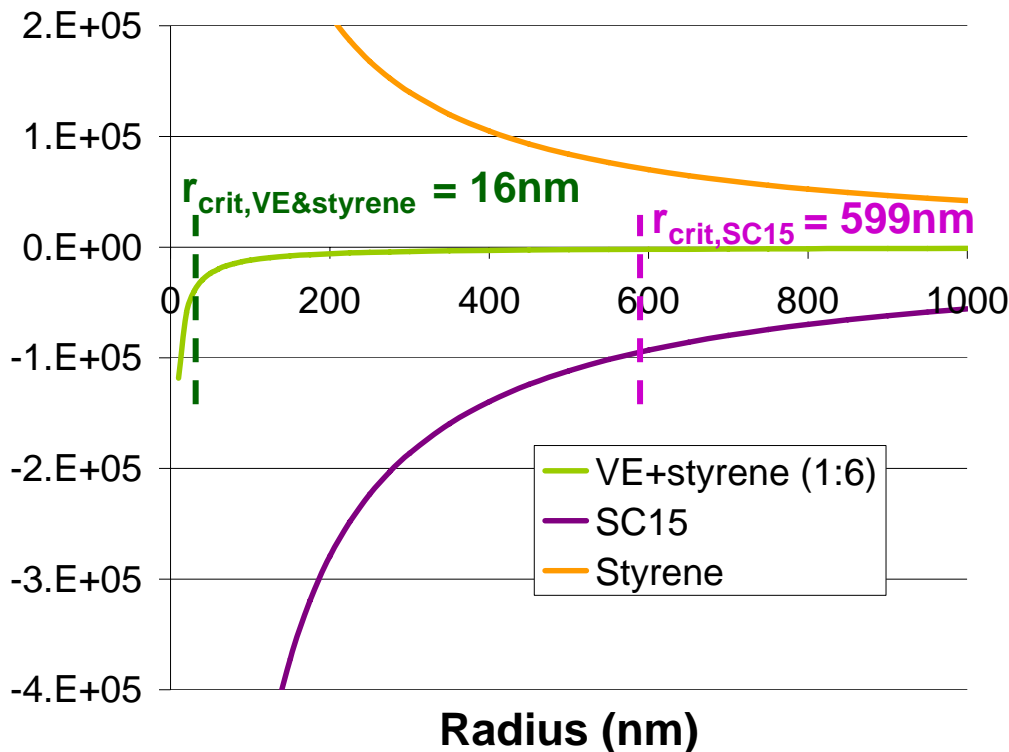
If  $\exists P_{vacuum}$ : Non-Wetting

$\Leftrightarrow P_{cap} > P_{vacuum}$  or  $r < r_{crit}$

$r_{crit,SC15}$  Critical radius for SC15

$r_{crit,VE}$  Critical radius for VE+Styrene

$\Rightarrow$  **Motivation for nanoporous materials**





# Critical Time to go Through the Membrane

Darcy's law

$$\dot{\xi} = \frac{K \Delta P^*}{\eta \xi}$$

Kozeny-Carman

$$K = \frac{\epsilon^3}{k\tau^2 S^{*2}}$$

- The flow front position of the resin into the pore is given by integration of the flow front velocity

– As a result:

$$\xi = \sqrt{\frac{2t}{\eta} \frac{\epsilon^3}{k\tau^2 S^{*2}} \left[ \Delta P + \frac{2\gamma \cos \theta}{r} - P_g \right]}$$

- Time for the resin to flow through the membrane:

– Model

$$t = \frac{h^2}{\frac{2}{5} \frac{\epsilon^3}{\eta S^{*2}} \left[ \Delta P + \frac{2\gamma \cos \theta}{r} - P_g \right]}$$

Vacuum pressure  
applied during VARTM

Capillary effect

Gravity effect

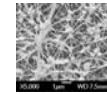


# Case Study of Membrane/Resin Interactions

- 2 Systems

- 1 membrane provided by W. L. Gore and Associates GmbH
- 2 resins: 1 vinyl-ester and 1 epoxy

- Membrane characterization



- **Porosity** : weight and volume measurements  $\Rightarrow \varepsilon_{vol} = 85.7 \pm 0.2 \%$
- **Pore size**: porometry  $\Rightarrow$  mean flow pore diameter  $d = 130.5 \pm 5.7 \text{ nm}$

- Resins' characterization



- **Density** : from manufacturers
- **Viscosity** : viscometer
- **Surface tension** : dynamic contact angle apparatus

- Membrane/Resin characterization



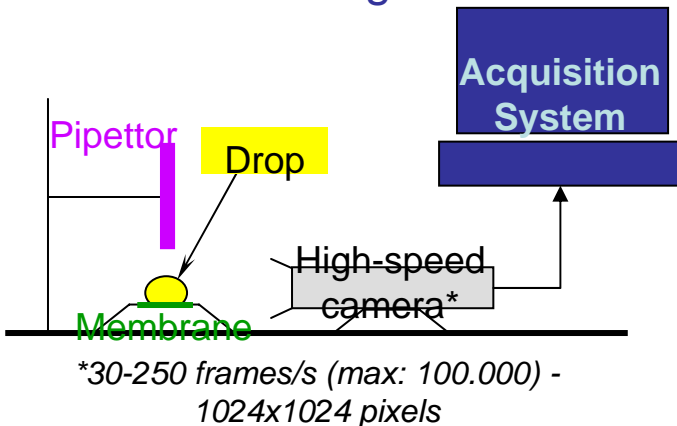
- **Contact angle** : sessile drop/high-speed camera



- Density, viscosity and surface tension of both resin systems:

Fluids	Density (kg/m <sup>3</sup> )	Viscosity (cP)	Surface tension (N/m)
<b>Source</b>	<b>From manufacturer</b>	<b>Measured</b>	<b>Measured</b>
<b>Vinyl-ester resin system</b>	<b>1024</b>	<b>106 ± 3.7</b>	<b>3.39x10<sup>-2</sup> ± 1.8x10<sup>-4</sup></b>
<b>Epoxy resin system</b>	<b>1198</b>	<b>183 ± 4.8</b>	<b>3.7x10<sup>-2</sup> ± 2x10<sup>-4</sup></b>

- Contact Angle Measurements:



SC15 Part A



$\theta = 139^\circ$

VE411-350 +  
 Styrene 1:6



$\theta = 91^\circ$



# Evaluation of Capillary Effect and Gravity

- Evaluation of the different contributions
  - Vacuum pressure:  $\Delta P = 98 \times 10^3 \pm 0.6 \times 10^3$  Pa
  - Capillary pressure:  $P_c = -\frac{2\gamma \cos \theta}{r}$
  - Gravity effect:  $P_g = \rho g \xi$

Fluids	$P_g$ (Pa)	$ P_c $ (Pa)
<b>Epoxy resin system</b>	<b>0.588</b>	<b><math>8.30 \times 10^5</math></b>
<b>Vinyl-ester resin system</b>	<b>0.502</b>	<b><math>1.76 \times 10^4</math></b>

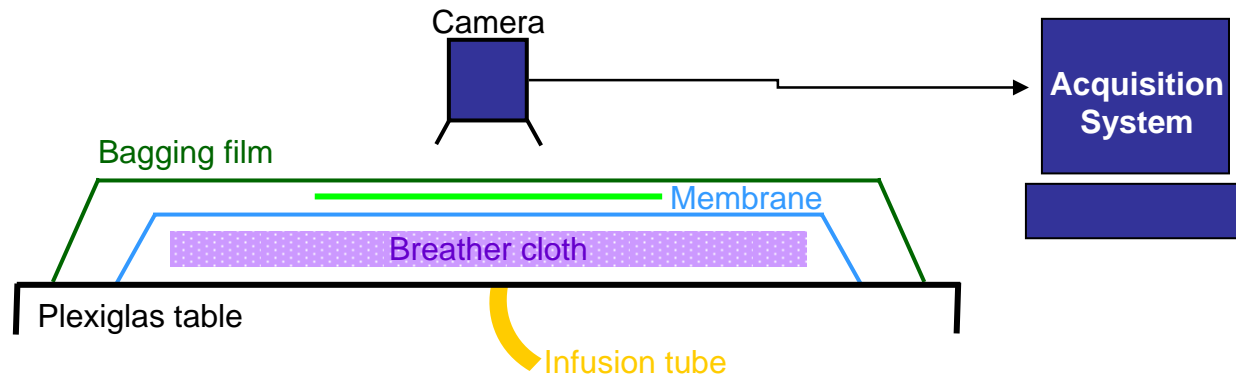
⇒ The gravity term can be neglected

# Experimental time to go through the membrane

- Setup

- Central injection line
- CCD (Charge-Coupled Device) camera to capture the wetting of the membrane

Paper to detect the flow of resin through membrane and create a strong contrast



Resin System	Reminder: gel time	Experimental	Model
<b>VE + Styrene (1:6)</b>	<b>30 minutes</b>	<b>11s ±30% ⇒ INCOMPATIBLE</b>	<b>1.7s (from 0.4s to 4s) ⇒ INCOMPATIBLE</b>
<b>SC15</b>	<b>7 hours</b>	<b>About 10h ⇒ COMPATIBLE</b>	<b>No impregnation ⇒ COMPATIBLE</b>



# Conclusions – Ongoing work

- A model based on:
    - Classical transport through porous medium
    - And surface science
  - was built to address the issue of membrane/resin interactions
- ⇒ The model captures the predominant transport mechanisms but still needs refinement

## Ongoing work

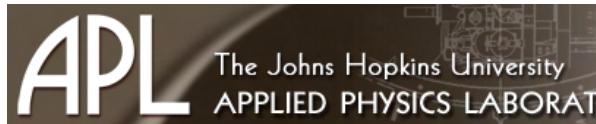
- Model
  - Use the final model of resin transport through membrane to identify the critical parameters of membrane and resin
- Design a membrane
  - Create a design chart, which gives the adequate membrane for a specific resin system
  - Validate the design chart with various membranes
- Optimize membrane for toughened epoxies



# Successful Membrane Research

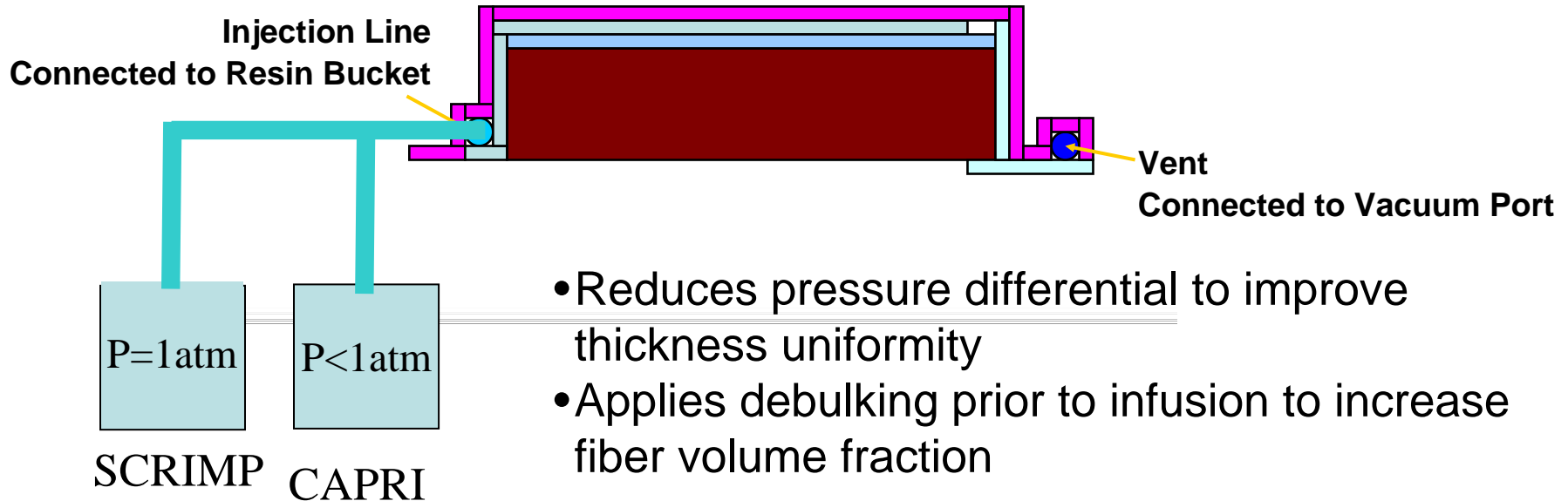


- PhD Student (Solange Amouroux) won numerous awards including
  - R. L. McCullough Scholars Award, May 2006
  - Winner of the Student symposium at the 2006 Long Beach SAMPE Conference and Exhibition
  - 3rd place in the Student symposium at the 2005 Long Beach SAMPE Conference and Exhibition
- Technology highlighted in the JEC and SAMPE journal
- Following companies have shown interest in the membrane research:





# Controlled Atmospheric Resin Infusion Process (CAPRI)

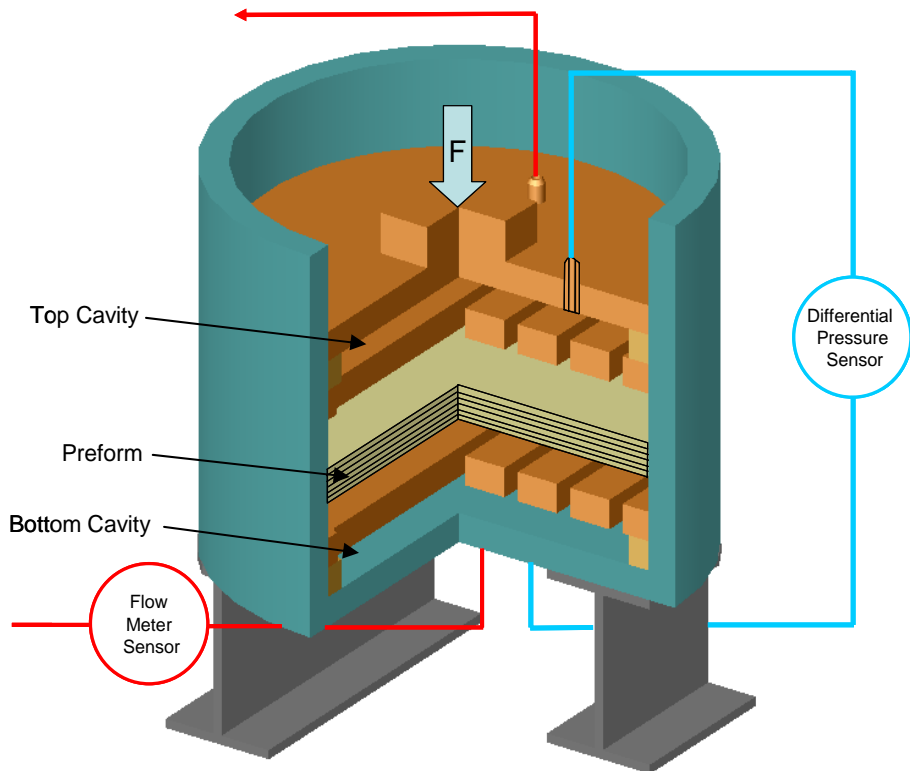


## Approach:

- Develop instrumentation to characterize compaction behavior and permeability as a function of compaction pressure and debulking cycle
- Conduct model experiments to evaluate process and use existing models to predict flow and thickness changes



# Design of the Permeability Measurement Work Cell (PermCell)

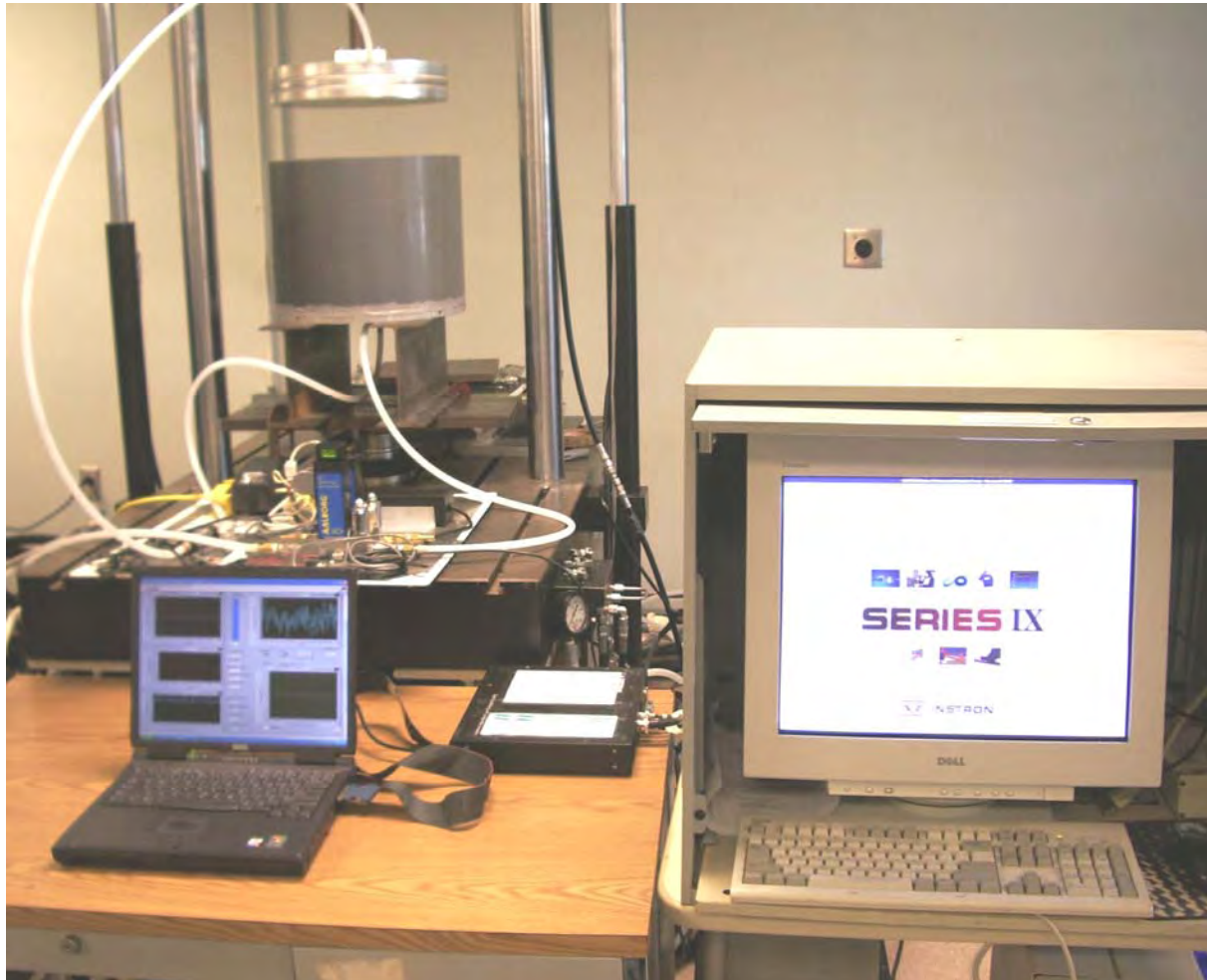


## • Benefits of the PermCell

- Rapid, clean and easy method to obtain permeability
- Permeability can be obtained online as a function of compaction, fiber volume fraction and debulking cycle
- Measurement Cell is capable for use of **gas** well as **liquid flow**
- Dimension (14.5 inch diameter) minimizes errors from boundary effects



# Experimental Set-Up

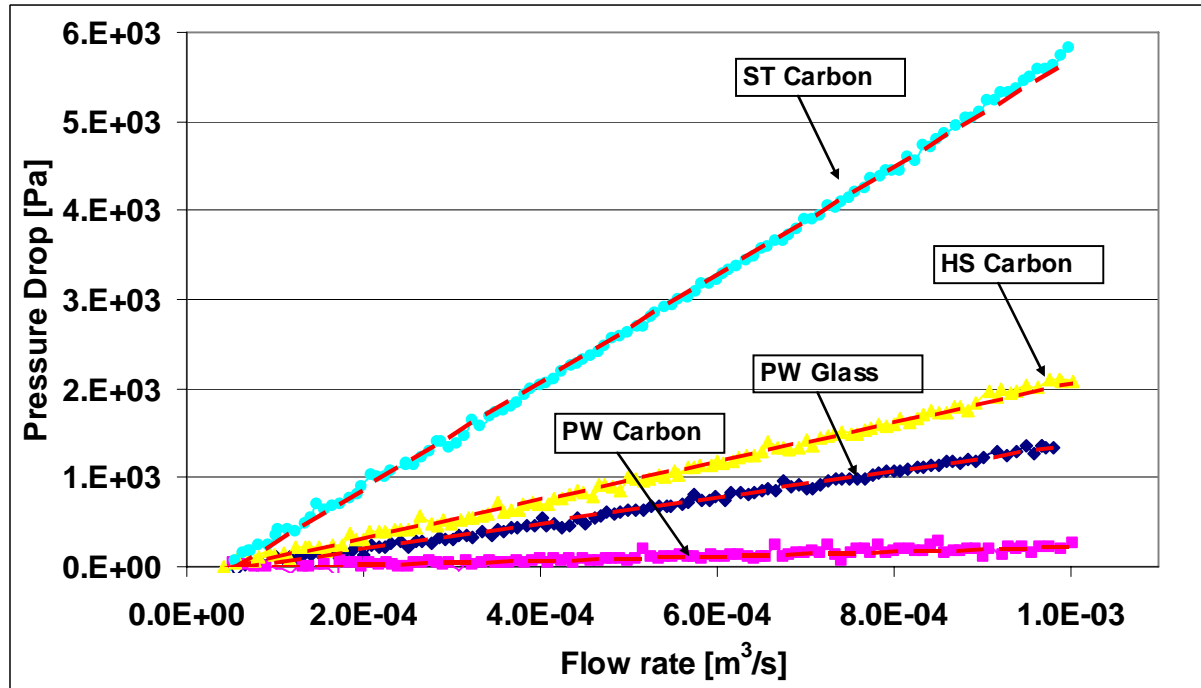




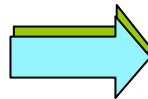


# Validation of Darcy's Law

## Measured Pressure Drop against increasing Flow Rate



$$\Delta P \sim Q$$



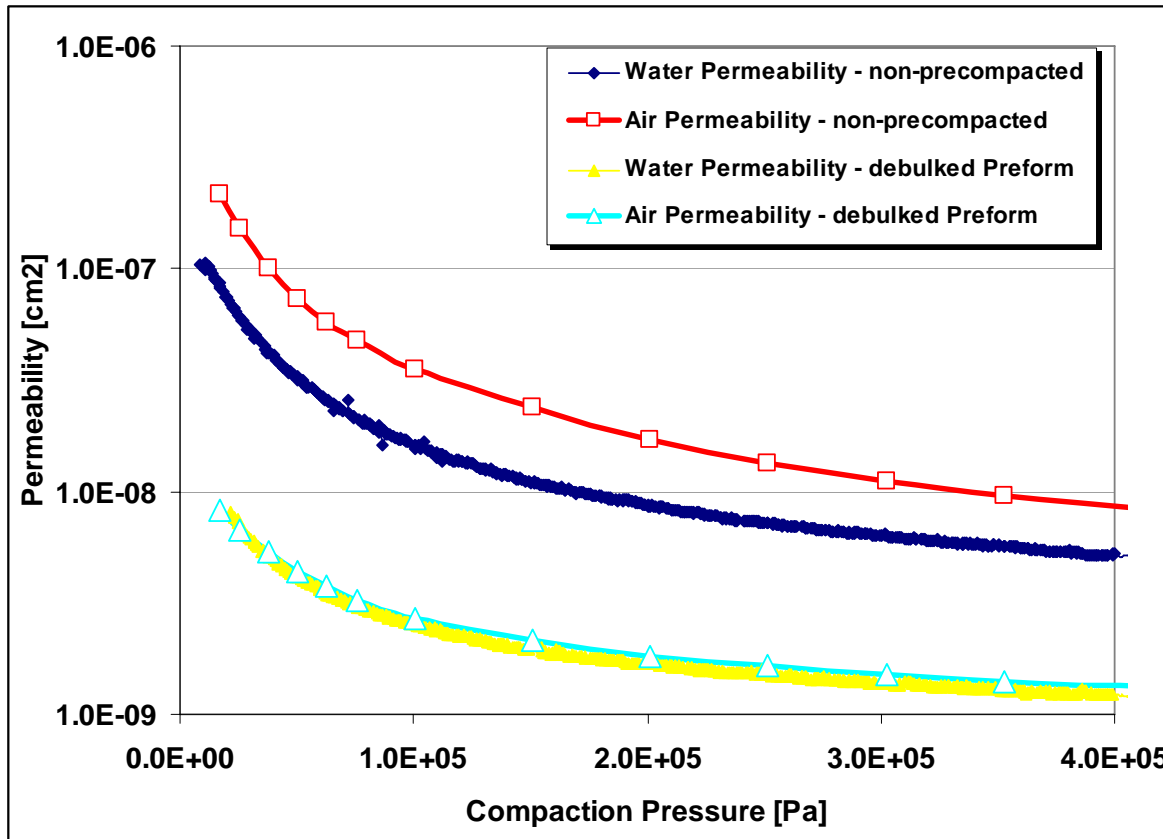
1D-Darcy's Law  
can be applied



$$v = \frac{Q}{A} = -K \frac{\Delta P}{\eta \cdot h}$$



# Water versus Air Permeability

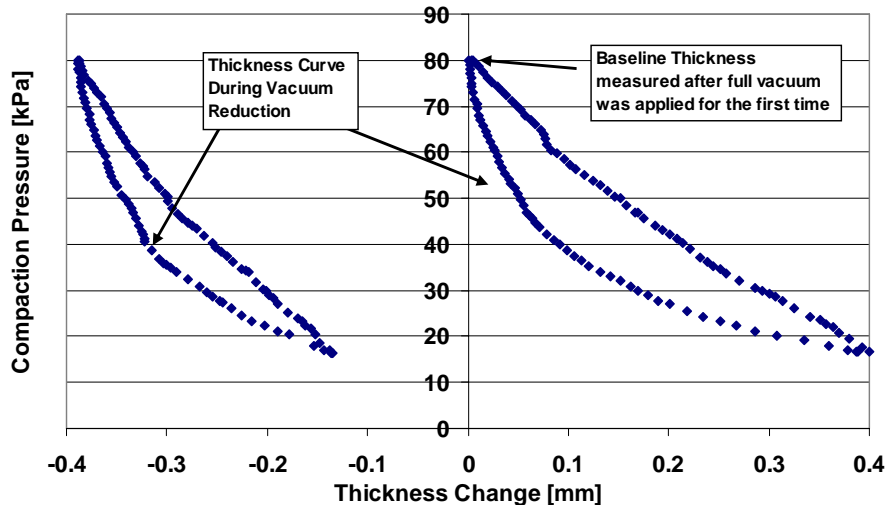


- Water permeability is slightly lower compared to air permeability
  - Lubrication may allow add. sliding during infusion
- After debulking the permeability is reduced and identical

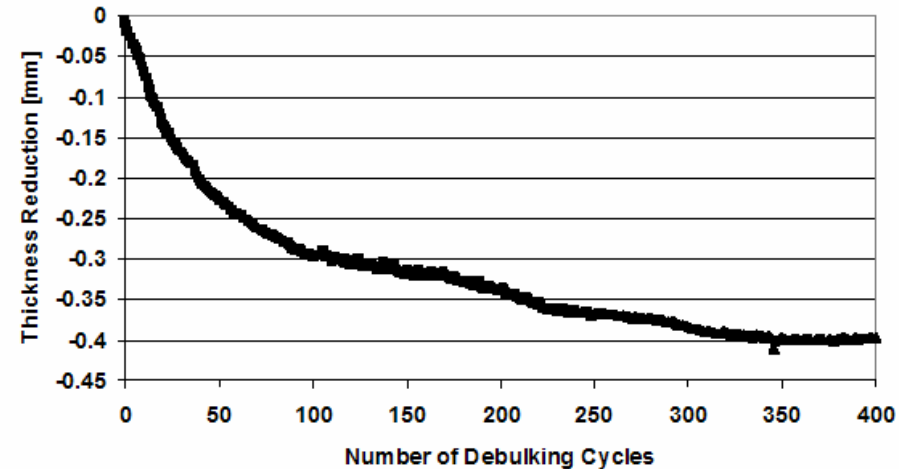


# Effect of Debulking on Thickness and Fiber Volume Fraction

Effect of Debulking on Compaction Behavior  
15 Layers E-Glass



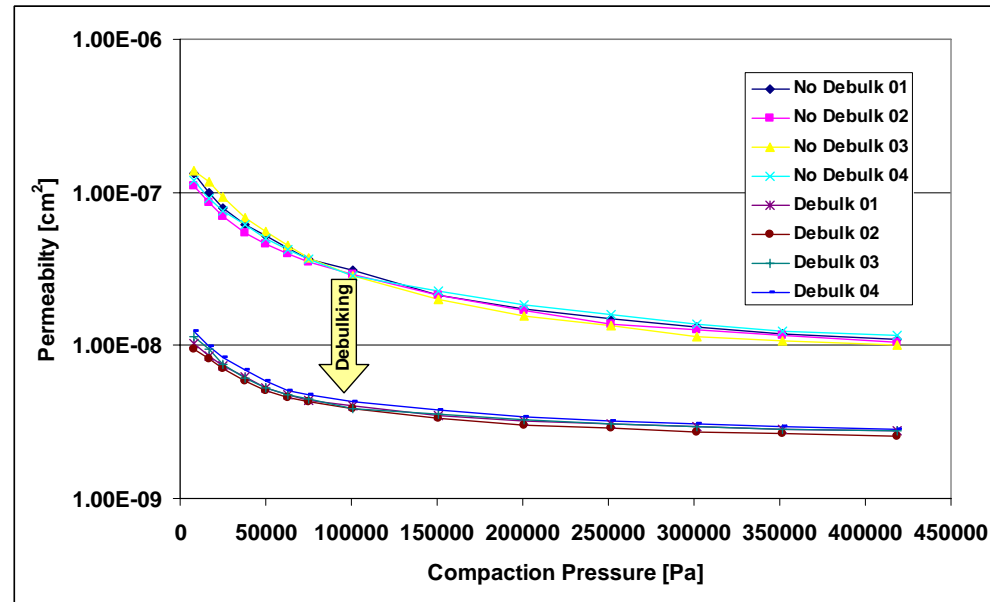
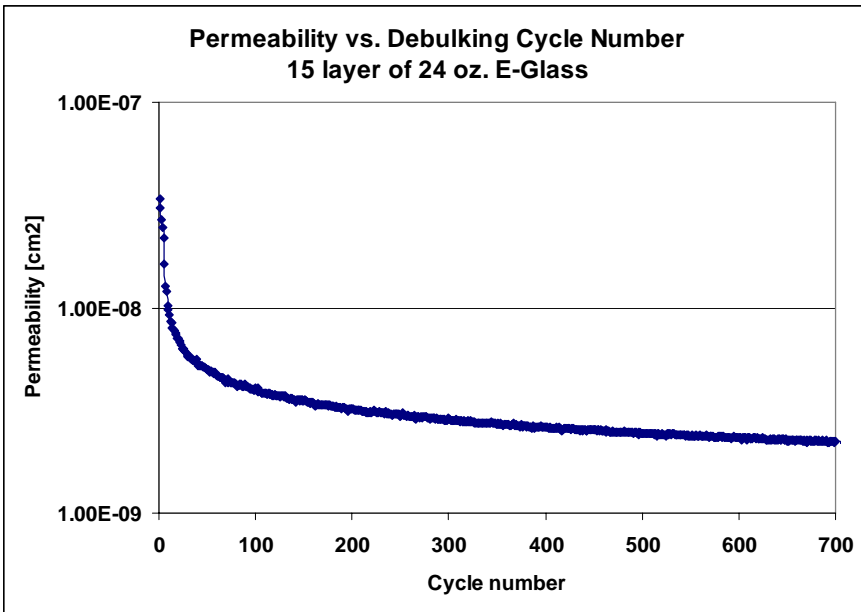
Effect of Debulking on Thickness Reduction  
15 Layers E-Glass



- The thickness and spring-back behavior is greatly reduced during debulking
  - Increases Fv
  - Reduces thickness gradient



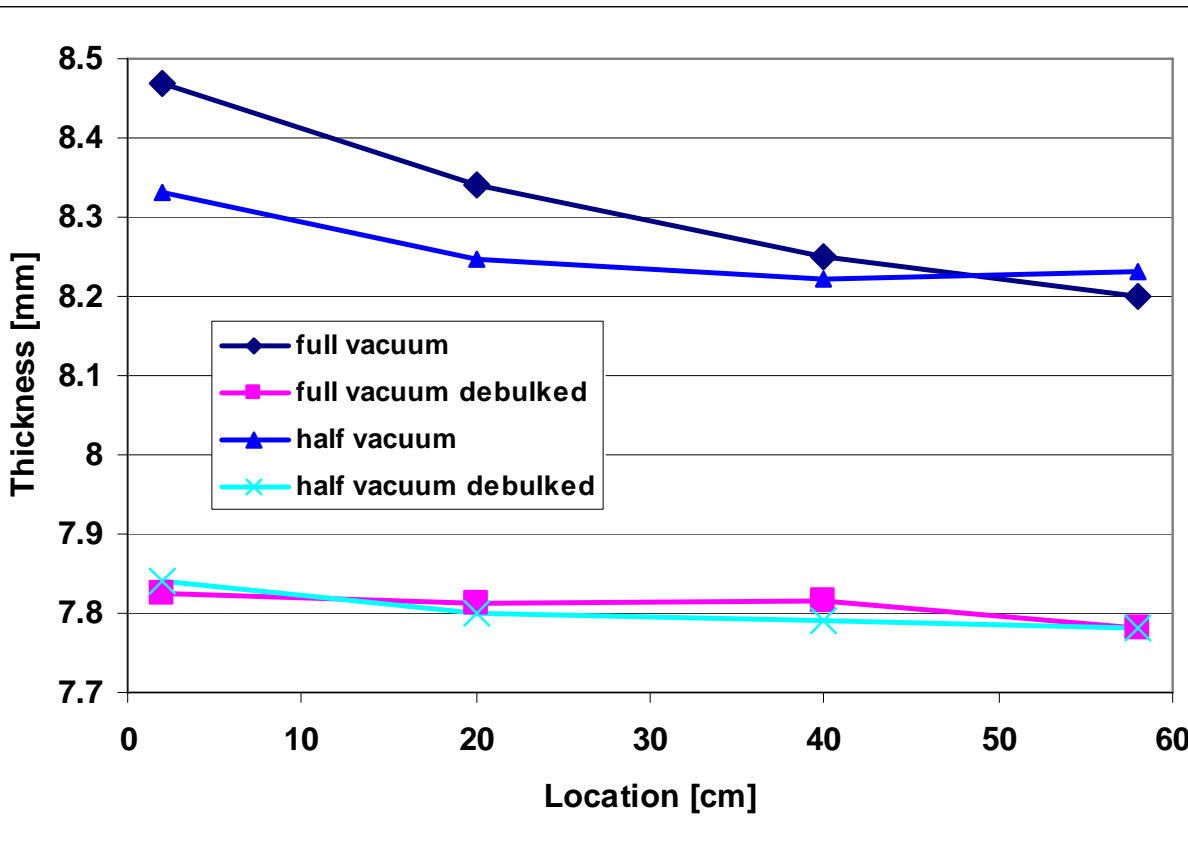
# Effect of Debulking on Permeability



- Debulking also reduces the permeability (up to a factor of 10 times) and thus flow behavior



# Thickness Behavior Comparison between CAPRI and SCRIMP

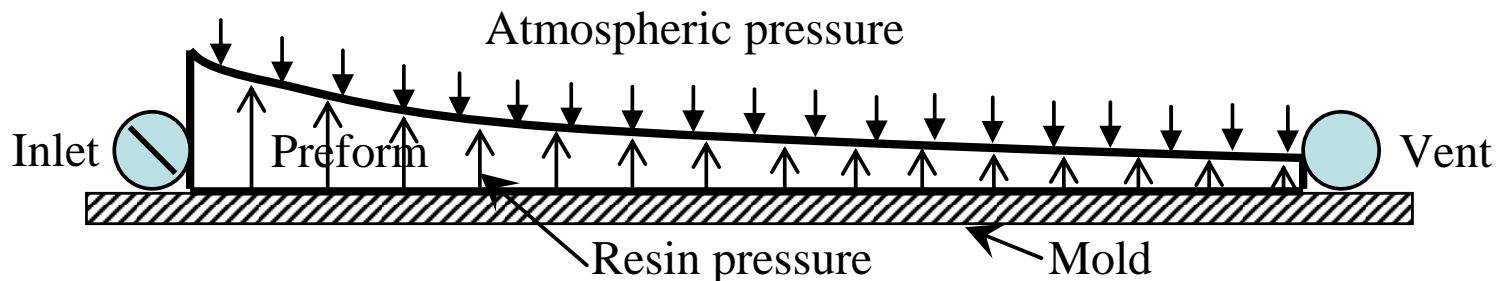


- Debulking can greatly increase final fiber volume fraction
- The thickness gradient is reduced when the CAPRI pressure is applied (insignificant for the debulked case)



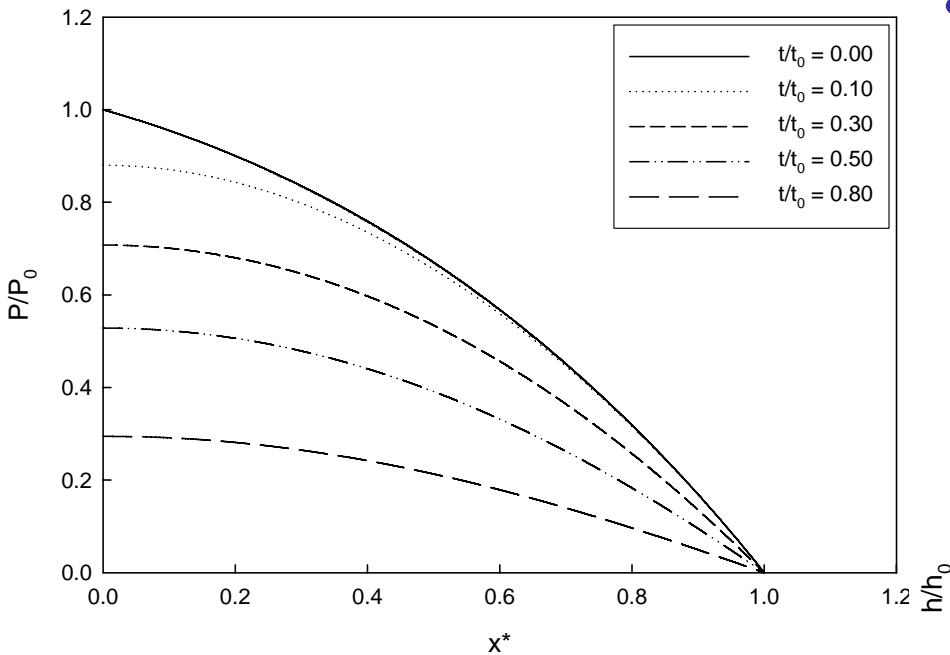
# Model to Predict Thickness Variation

- Considers infusion and resin bleeding behavior
- Requires compaction and permeability behavior of fabric
- Provides anticipated thickness after infusion and during resin bleeding



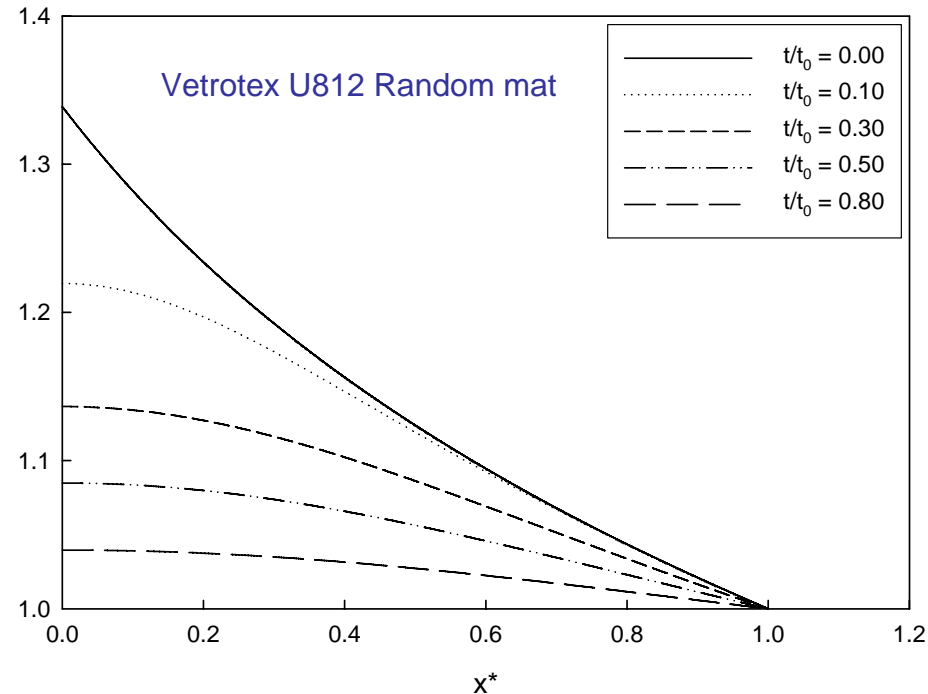


# Thickness Field During Typical SCRIMP Processing



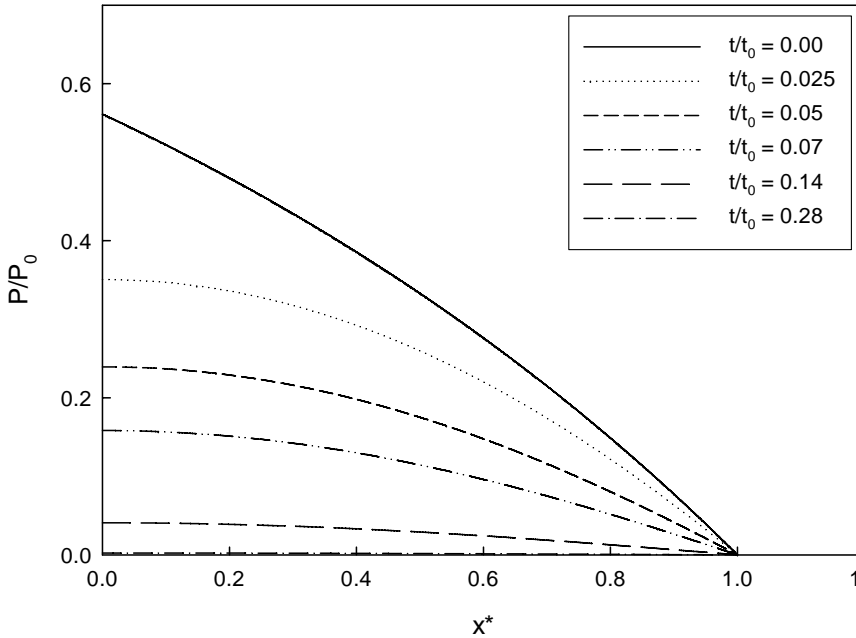
- **Model can be used to predict thickness and pressure variation during and after infusion**

- **It can be used to optimize CAPRI pressure and gel time of resin to minimize thickness gradient**





# Thickness Field During CAPRI Processing

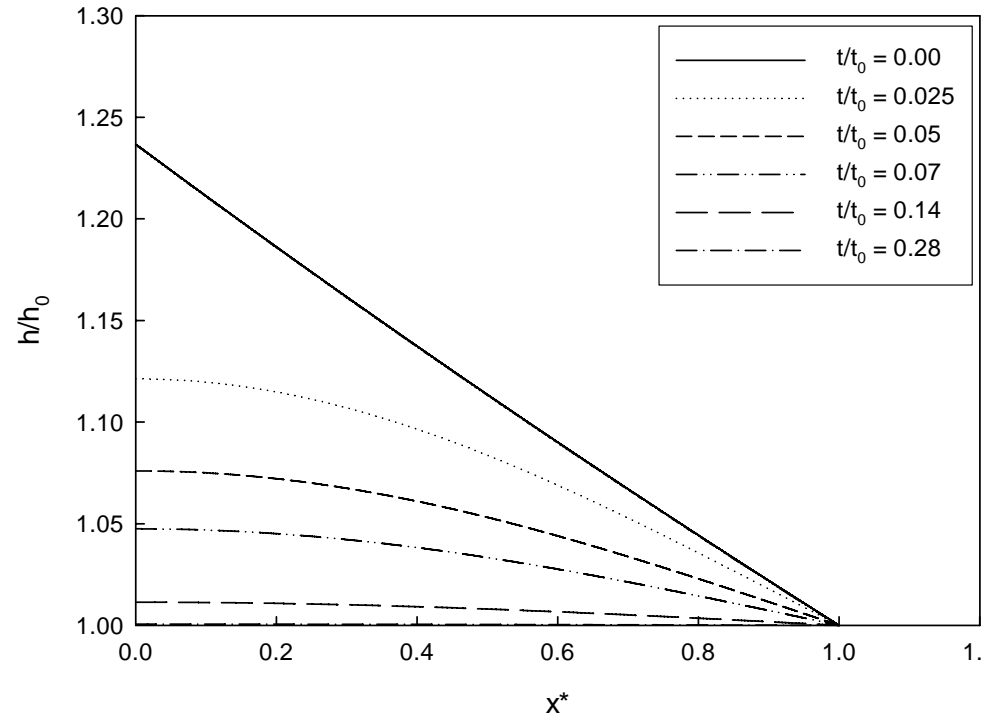


- The infusion bucket was evacuated to 0.5atm (CAPRI pressure)

## CAPRI Process

→ This resulted in 30% reduction in thickness gradient directly after infusion

→ The resin bleeding time reduced by 80% to obtain a 5% thickness gradient

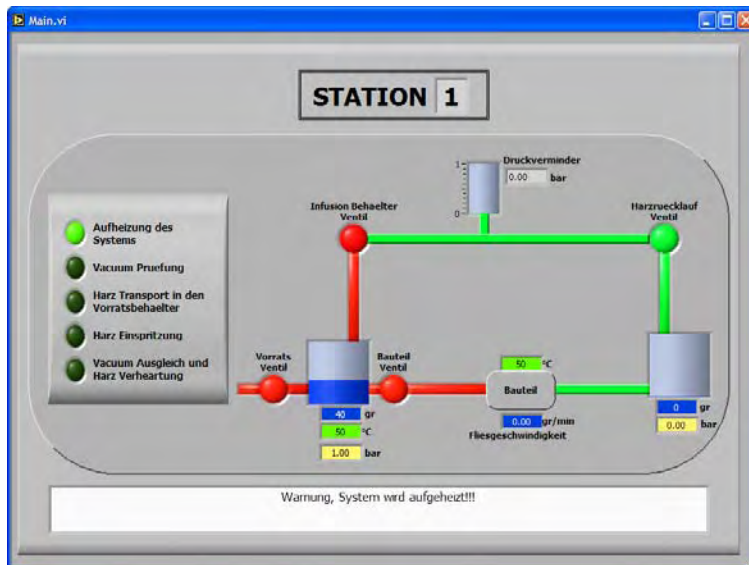
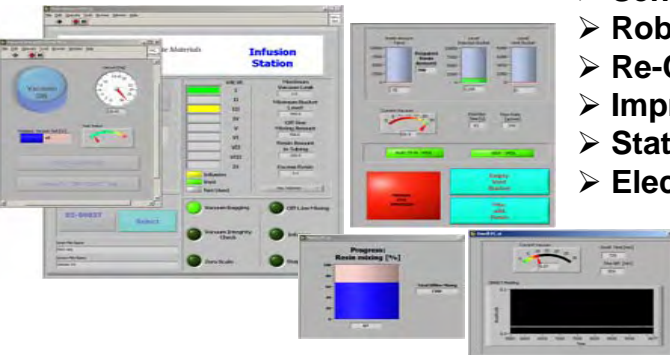






# Aerospace VARTM Requires Elevated Temperature Processing

- Sensor Based Infusion Technology
- Robust System Construction
- Re-Configurable Infusion Schemes
- Improved Resin Mixing System
- Statistical Data Sampling During Infusion & Cure
- Electronic Work Instruction



## ELEVATED PROCESSING REQ

- Heated External Resin Supply
- Heated Tooling
- Adapt IPC capabilities to elevated temperature processing



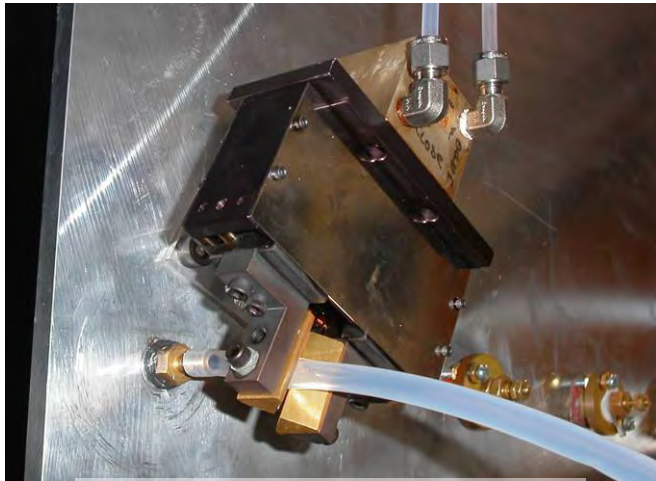
# CONSIDER TWO AEROSPACE TOUGHENED EPOXY SYSTEMS



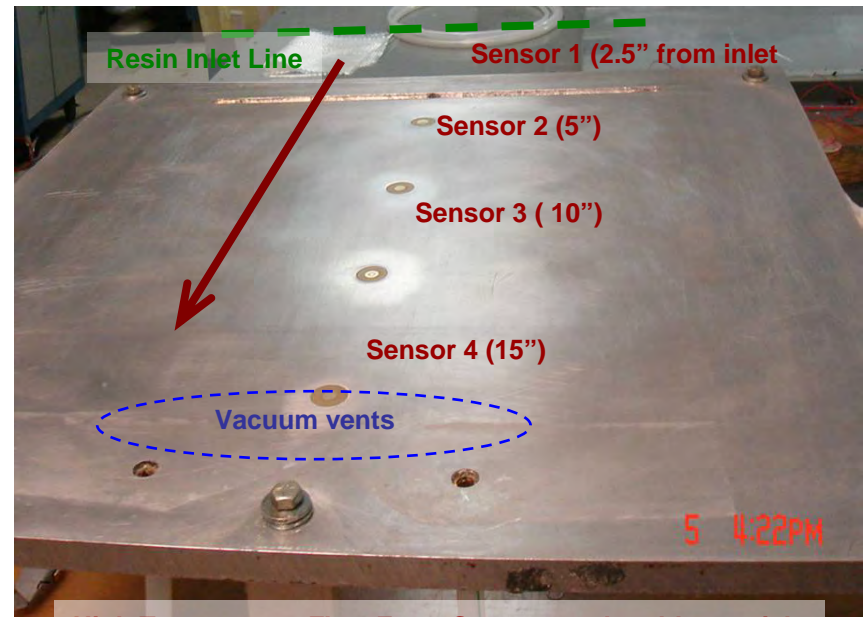
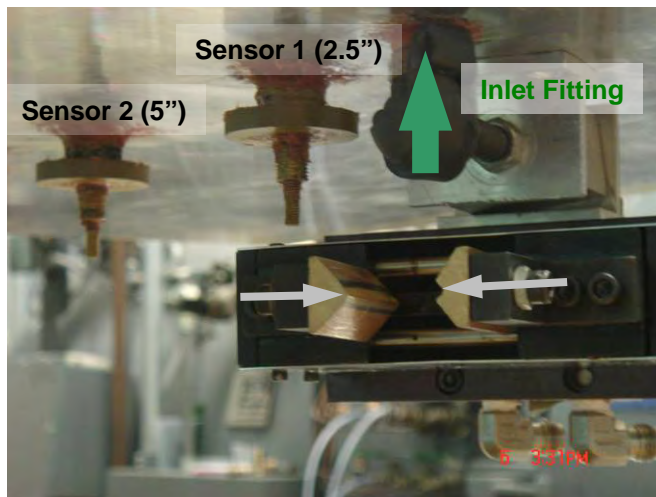
- **Cytec Epoxy Cycom 977-20**
  - Viscosity = 120 cps @ 167°F
  - Ramp with 4°F/min to 355 cure for 3 hours, cool to 140°F @ 5°F/min
  - Cured Resin Density = 1.31g/cm<sup>2</sup>
  - Tg = 212°C
- **Hexcel Epoxy RTM 6**
  - Viscosity = 180 cps @ 177°F / 40 cps @ 248°F
  - Ramp with 5°F/min to 320 °F, cure for 75 minutes
  - Cured Density = 1.14g/cm<sup>2</sup>
  - Tg = 183°C (Hexcel Datasheet)



# Elevated VARTM Requires New Grippers and Sensors



**Pneumatic High Temperature Gripper**

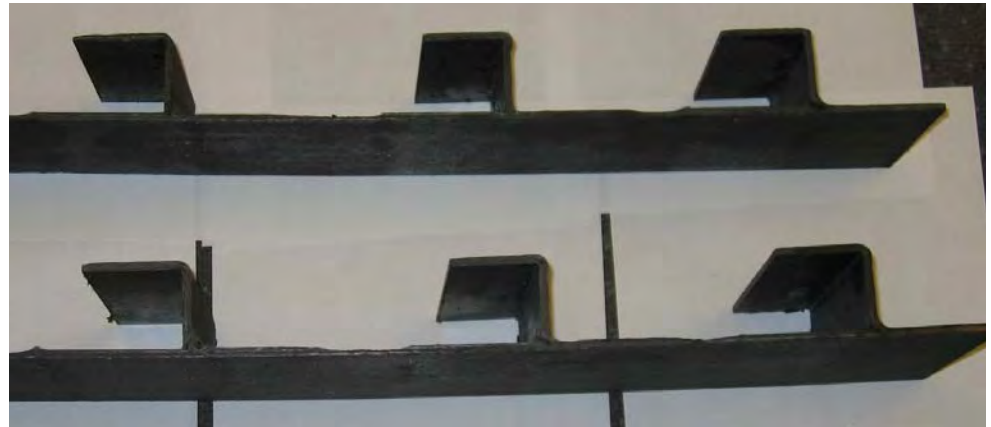


**High Temperature Flow Front Sensors and mold materials**



# Component Fabrication

- Consistent fiber volume fraction of  $> 56\%$  is achievable
  - AS4-GP-6K-5HS 377g/m<sup>2</sup> carbon fabric
  - Higher performance fibers are being considered
- Void Content below 1%
- Unitized structures can be fabricated
  - Stiffened Structures
  - Cored Structures
- Automation has been implemented





# Develop Property Database

- Compare various processes
  - Fiber Volume Fraction
  - Void Content
  - Dimensional Tolerances
- Structural
  - Tension, compression, bending
  - Damage tolerance
    - Open-hole compression
    - Compression after impact



# A Look Forward



- Benefit to Aviation
  - Improved fundamental understanding of VARTM processing to understand benefits and disadvantages of various process variations
  - Reduce part-to-part variations / improve allowables
  - Automated VARTM will allow QA/QC of part production reducing costs and improve quality while maintaining traceability
  - Open-access database of structural properties
- Future needs
  - Work close with VARTM manufacturers to transition technology
  - Improve VARTM to achieve autoclave-level fiber volume fraction
  - Investigate more complex geometries / unitized structures