POROSITY IN COMPOSITE MATERIALS
- PART II -

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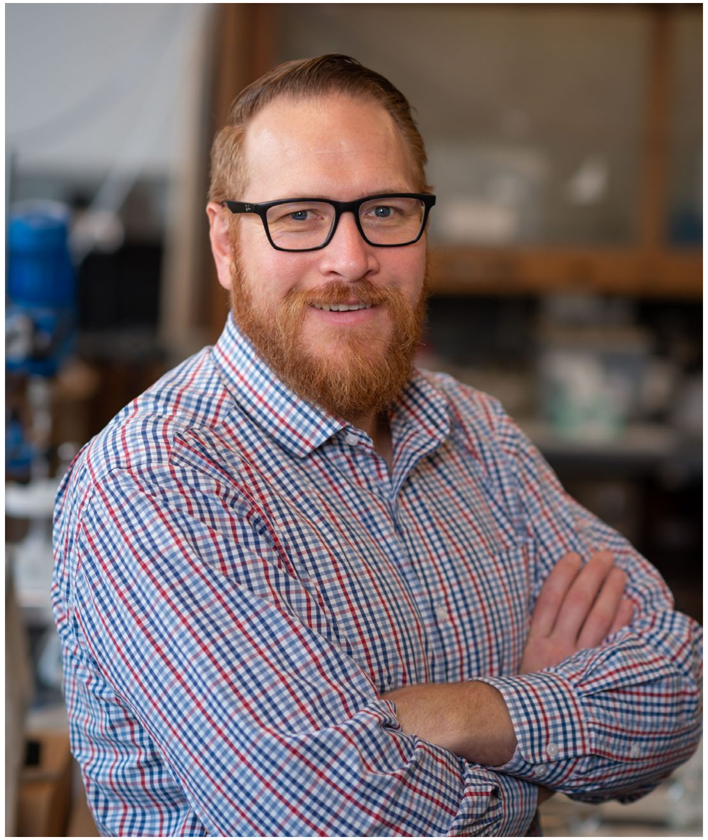
YOUR HOSTS



Casey Keulen, Ph.D, P.Eng.
Assistant Professor of Teaching, University of British Columbia
Director of Advanced Materials Manufacturing MEL Program, UBC
Director of Knowledge in Practice Centre, CKN

- Ph.D. and M.A.Sc. in Composite Materials Engineering
- Over 15 years experience in industry and academia working on polymer matrix composites in aerospace, automotive, marine, energy, recreation and others
- Experience working with over 150 companies from SME to major international corporations
- Expertise in liquid composite moulding and thermal management

YOUR HOSTS

Gavin Tao, M.A.Sc.
Research Engineer, Composites Research Network
Department of Materials Engineering, UBC

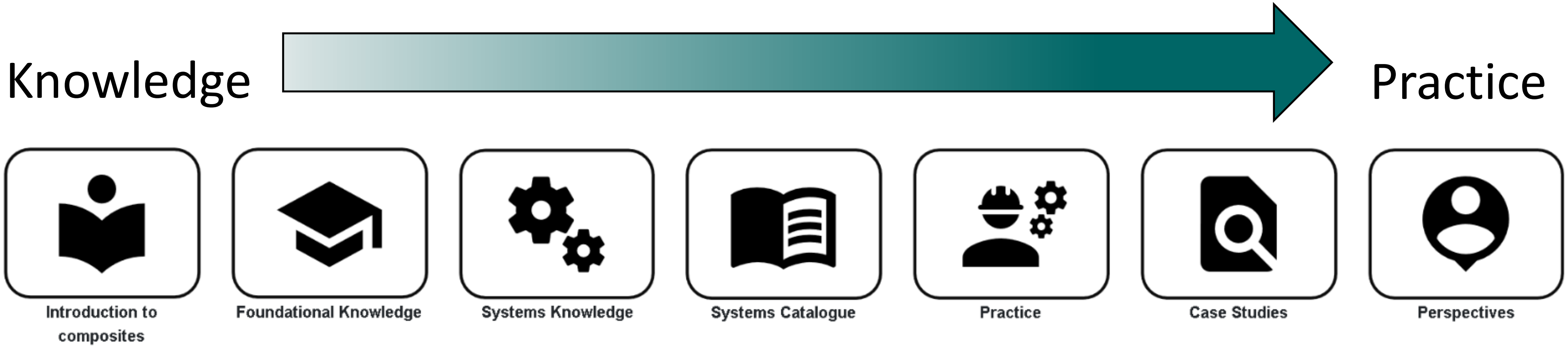
- Expertise in manufacturing, processing of advanced composite materials, process induced deformation, liquid composite moulding and sandwich structures
- Over 7 years experience with various industrial and academic research projects
- Content contributor of the Knowledge in Practice Centre (KPC)

KNOWLEDGE IN PRACTICE CENTRE (KPC)

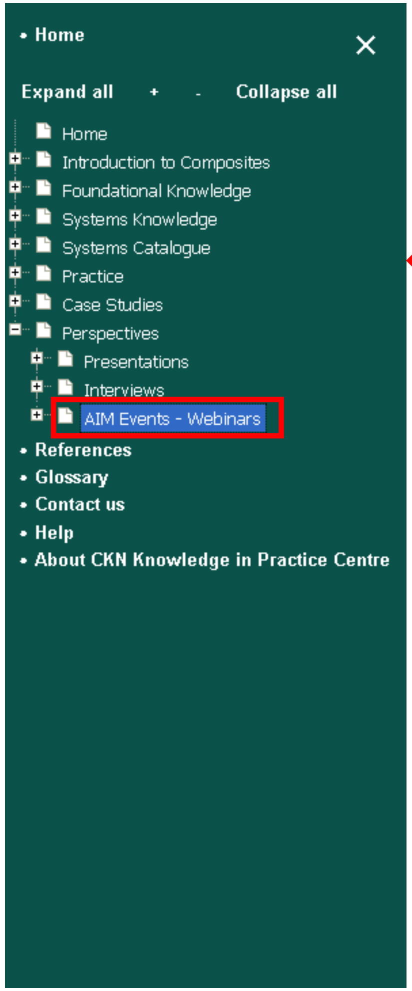
- A freely available online resource for composite materials engineering:

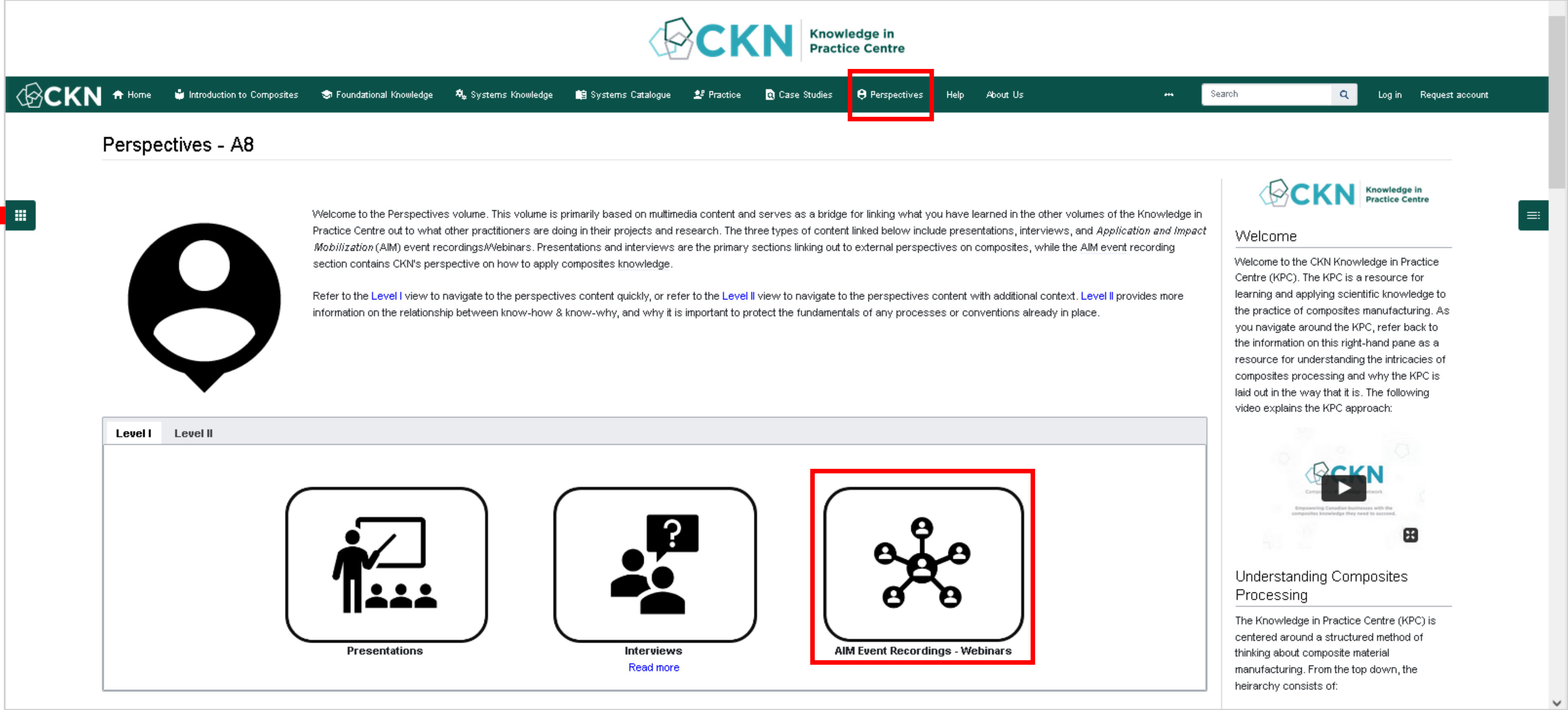
  compositeskn.org/KPC

- Focus on practice, guided by foundational knowledge and a systems-based approach to thinking about composites manufacturing



PAST WEBINAR RECORDINGS AVAILABLE





Today's Webinar will be posted at:
https://compositeskn.org/KPC/A337

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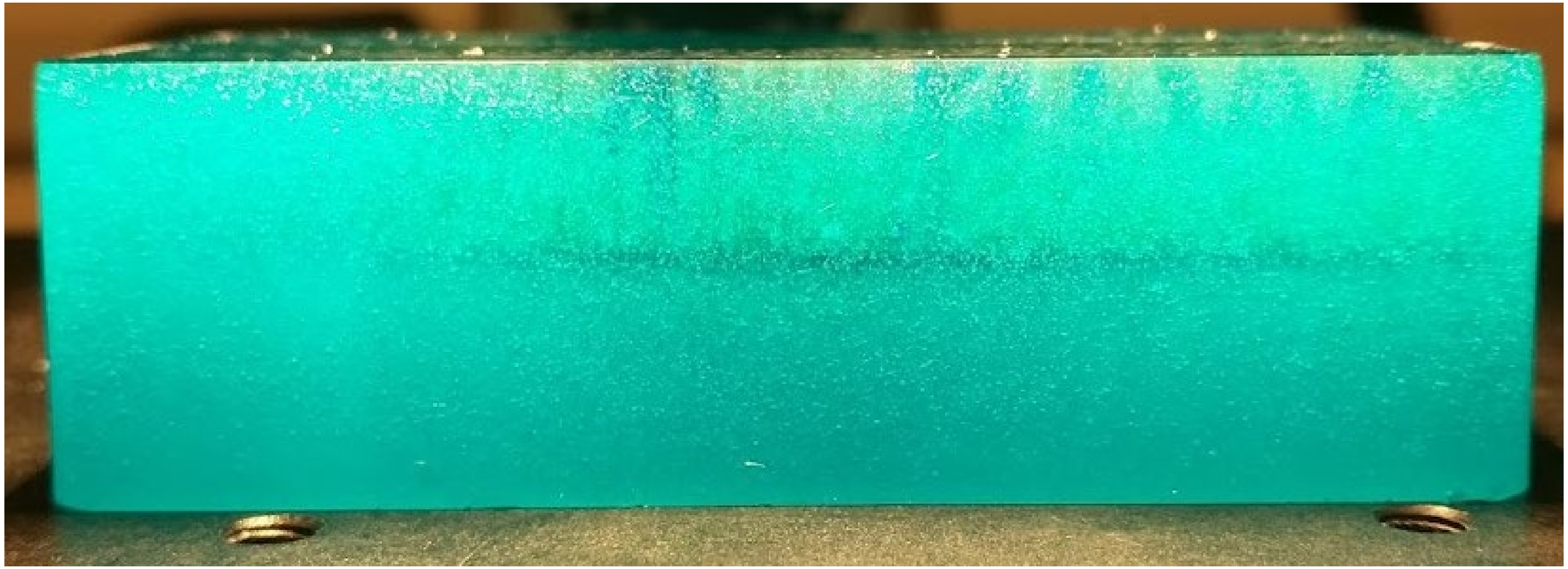
TODAY'S TOPIC:

Porosity in Composite Materials - Part II



OUTLINE - PART I

- Introduction to porosity
- Effect of porosity on performance (mechanical properties)
- Classifying porosity:
  - Morphology: surface vs bulk porosity
- Sources and Sinks
- Manufacturing stages that introduce porosity
- List of sources and sinks
  - Stages in the process that sources occur/affect final porosity level
  - Manufacturing processes that affect the source
  - Sinks for the source
  - Case study/examples
- Wrap up

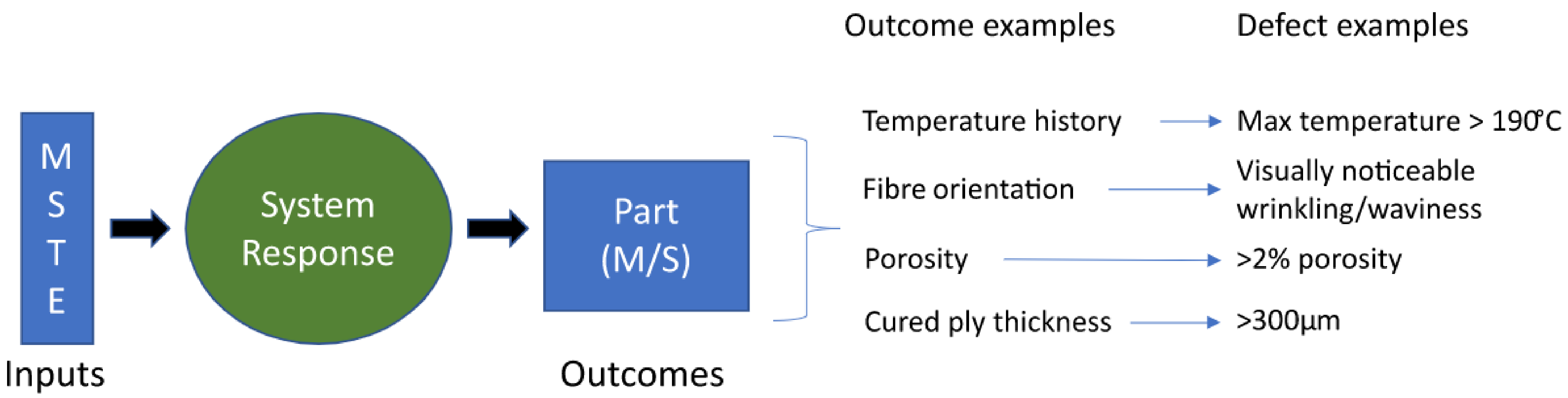
Watch the recording of the previous webinar on page:
A336

OUTLINE - PART II

- List of sources and sinks continued
  - Stages in the process that sources occur/affect final porosity level
  - Manufacturing processes that affect the source
  - Sinks for the source
  - Case study/examples
    - Liquid composite moulding
    - Gas transport in prepreg
- Degassing and debulking
- How does a bubble grow – mechanism
- Assessing and measuring porosity level
- Conclusion

INTRODUCTION

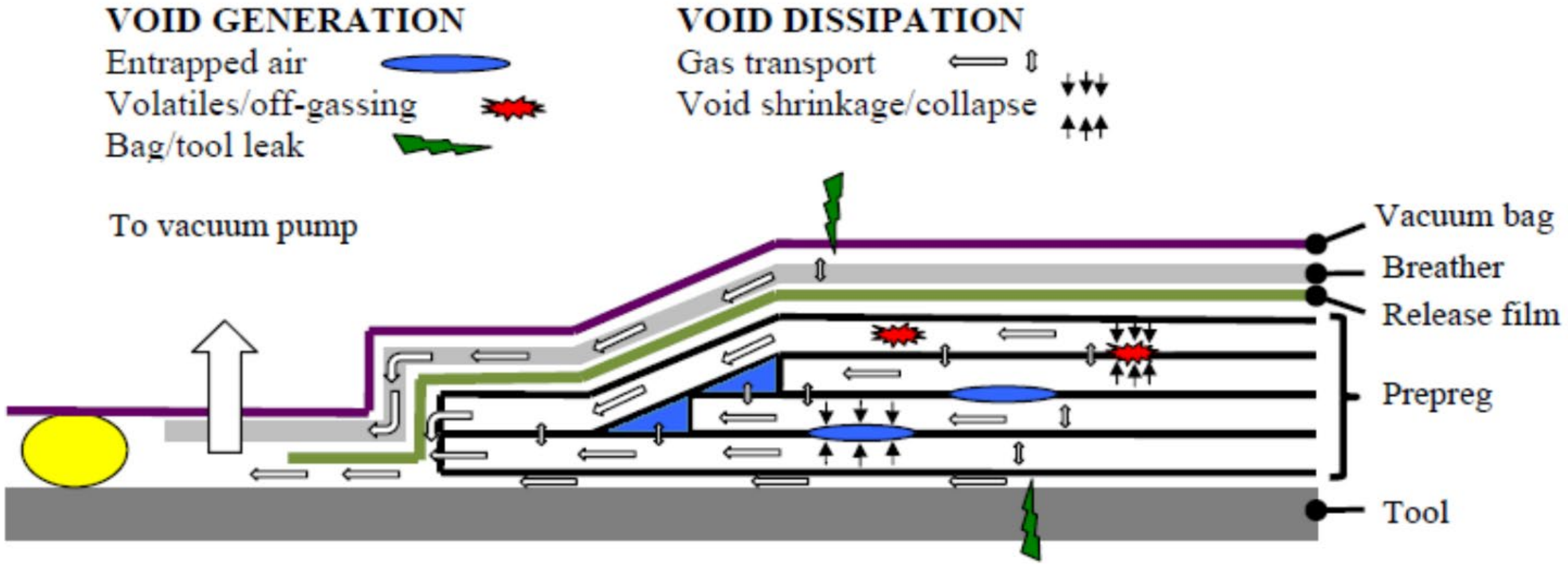
- Porosity definition: the absence of resin in locations where should be occupied by resin. Undesirable in most cases
- Forms: voids, bubbles, pin holes
- One of most common outcomes/defects
- Most porosity in composite parts are induced during the manufacturing process



SOURCES AND SINKS

Porosity sources
(mechanisms that generate voids)

Porosity sinks
(mechanisms that dissipate voids)



Roy, M. (2015). *Porosity in Configured Structures Effect of Ply Drops and Caul Sheets in the Processing of Composite Parts*. The University of British Columbia. https://doi.org/10.14288/1.0220869

LIST OF POROSITY SOURCES AND SINKS

Exhaustive list of porosity sources

- Fiber/resin incompatible
- Entrapped gas in setup and consumables
- Over-bleeding
- Empty space from shape (bridging, ply drop off)
- Tool dimensional error
- Core absorbing resin cause face sheet to starve
- Face sheet dimpling and pull away from the tool
- Moisture absorbed in core
- Not dissolved gases and volatiles in the resin (visible bubbles)
- Resin shrinkage
- Lack of consolidation

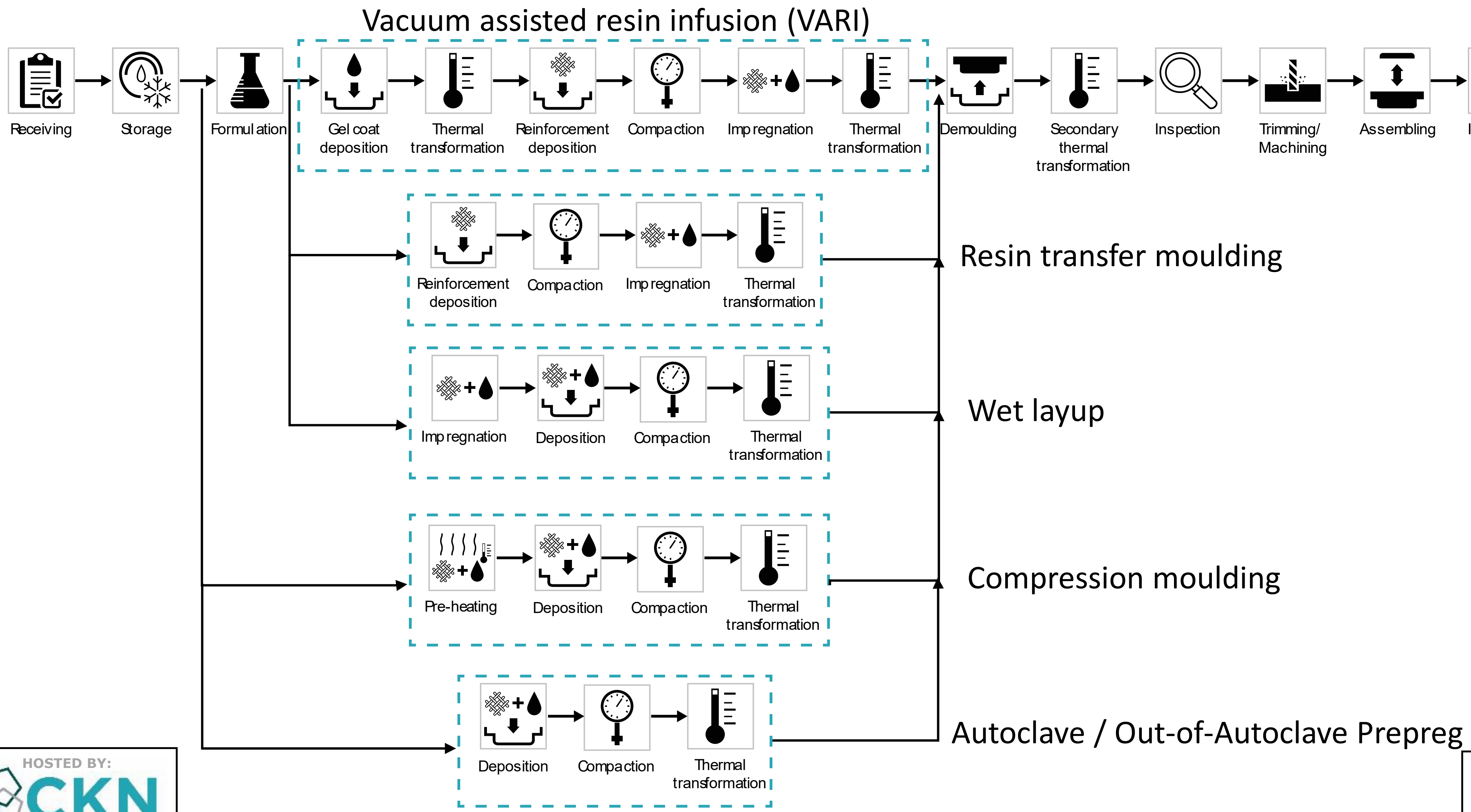
Part I A336

- Tool/bag/vacuum leak
- Outgassing of absorbed volatiles in the fiber
- Outgassing of dissolved gases and volatiles in the resin
- Moisture in the fiber
- Moisture in the resin
- Formation of cure reaction by-products
- Issues with resin flow through fiber bed
- Insufficient gas transport within the laminate

Exhaustive list of porosity sinks

- Resin formulation
- Prepreg design (architecture, degree of impregnation)
- Resin/prepreg storage and handling conditions
- Degassing, vacuum removing air, moisture and volatiles in resin
- Material deposition techniques
- Shape design (avoiding sharp corners, and aggressive ply drop offs)
- Proper amount of bleeding and breathing using consumables
- Debulking, vacuum removing air, moisture and volatiles in the prepreg laminate and resin
- Eliminating vacuum leaks in tool and vacuum bag setup including consumables
- Eliminating gaps between tool and part (fiber bridging, underfill in match-die)
- Pressure (from roller/autoclave/press) to provide consolidation
- Control resin flow speed (pressure differential)
- Control resin flow directions (injection strategies)
- Control thermal history (out time, cure cycle) to reduce moisture driven bubble growth

MANUFACTURING PROCESSES



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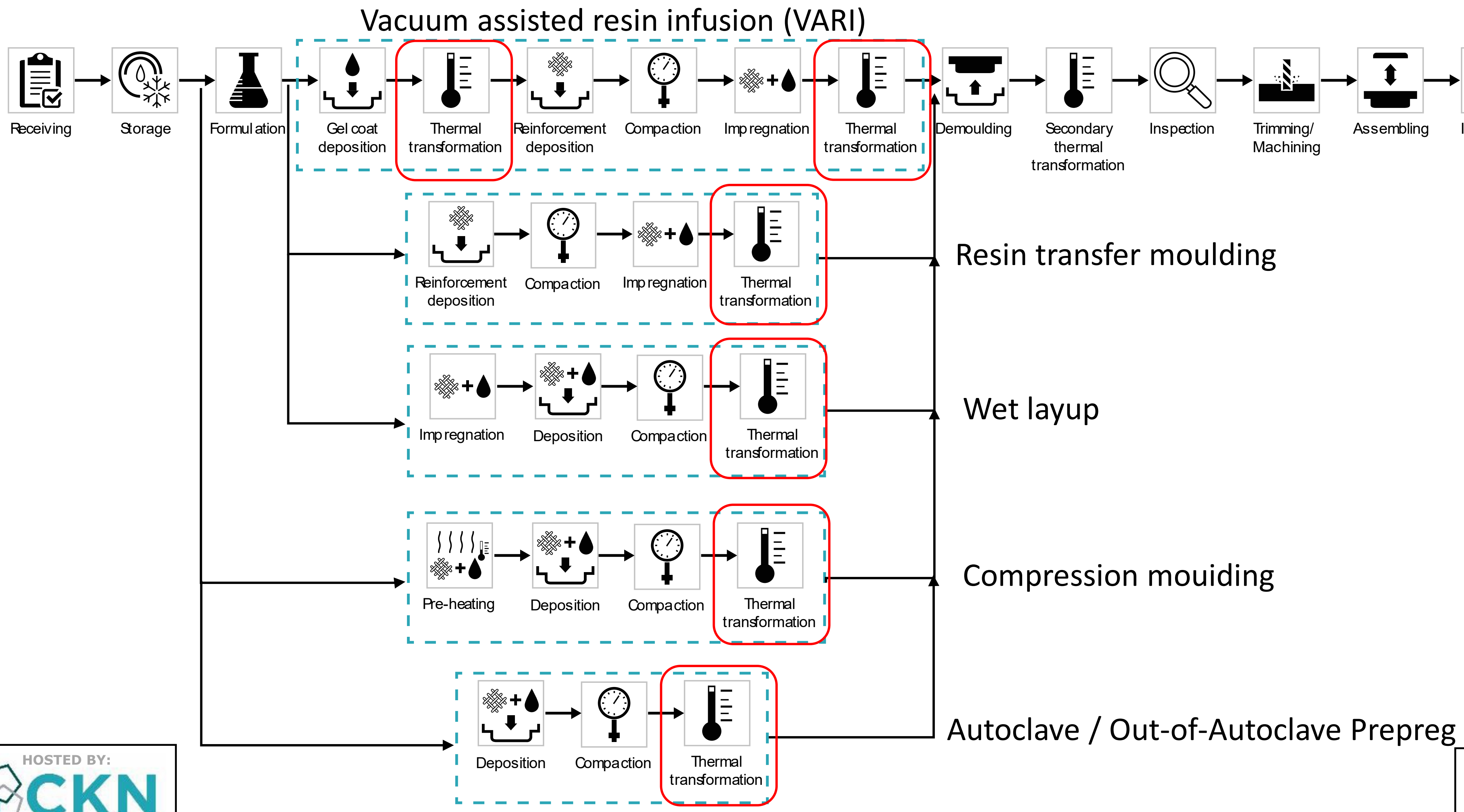

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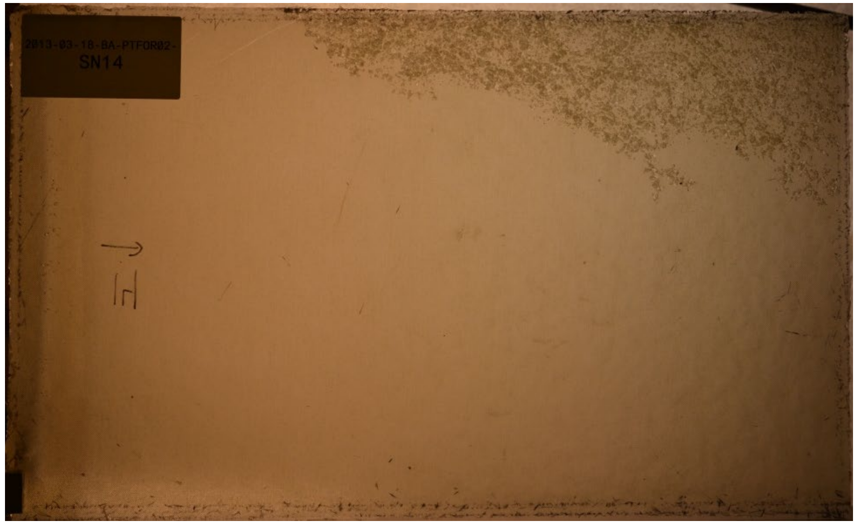
TOOL/BAG/VACUUM LEAK

- Air/gas drawn into the system via a leak
- Common leak sources:
  - Tacky tape – pleats, overlap, inlets/outlets, objects passing through (backing tape, peel ply, reinforcement/fiber)
  - Vacuum bag puncture
  - Consumable leak – inlet tube leak
  - Tool leak – from damage, bad welds, poorly sealed tool surface, tooling material permeability



TOOL/BAG/VACUUM LEAK

What stages in the manufacturing process do Tool/bag/vacuum leak occur/affect final porosity level?

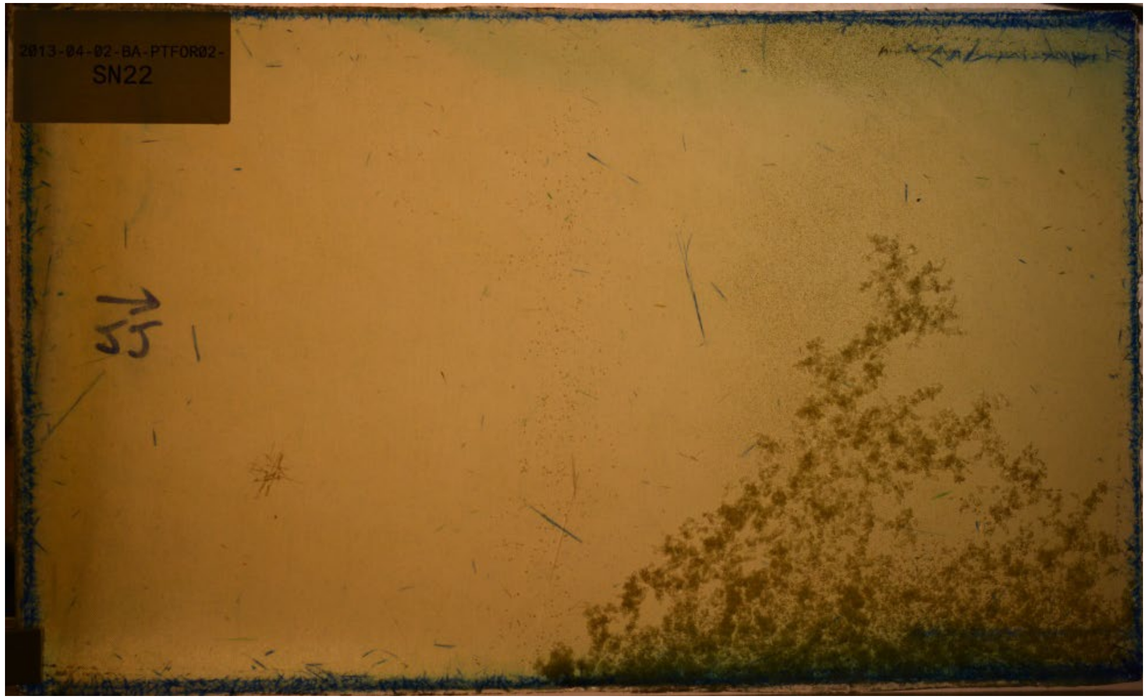
- Material deposition
- Thermal transformation

What manufacturing process does tool/bag/vacuum leak affect porosity?

- Processes that hold the material under vacuum during cure

Sinks for tool/bag/vacuum leak

- Material deposition techniques
- Eliminating vacuum leaks in tool and vacuum bag setup including consumables
- ***Leak test!!!***



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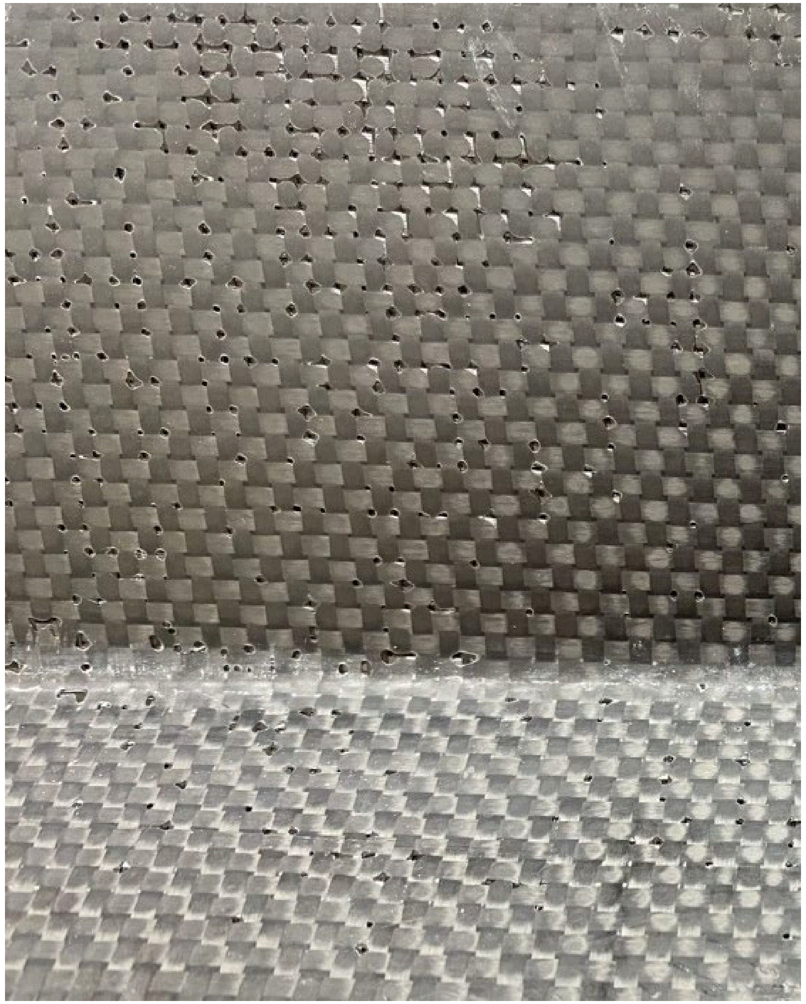

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OUTGASSING

- Outgassing: a release of gas before resin gelation and vitrification
- Which gases? [1]
  - Air
  - Water
  - Low molecular weight volatiles
  - Curing by-products
- Where do the gases come from?
  - Dissolved gases and volatiles in the resin
  - Absorbed volatiles in the fiber
  - Moisture in the fiber
  - Moisture in the resin
  - Formation of cure reaction by-products

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1. J.Kay, L. Fahrang, K. Hsiao, & G. Fernlund. (2015). Effect of process conditions on porosity in out-of-autoclave prepreg laminates. *18th International Conference on Composite Materials*

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MOISTURE

- Under vacuum, absorbed moisture can evaporate and form porosity before/during cure
- Moisture source can be fiber/resin, complete extraction of moisture is not practical

What stages in the manufacturing process does moisture affect porosity?

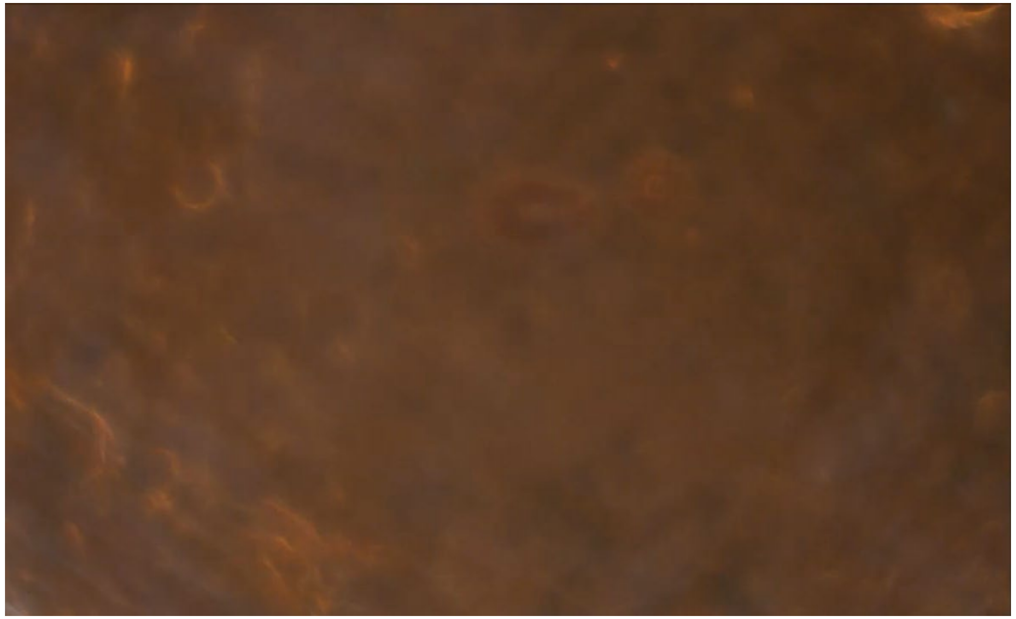
- Storage
- Formulation (mixing)
- Material deposition
- Thermal transformation

What manufacturing process does moisture affect?

- All processes

Sinks for porosity due to moisture :

- Degassing, vacuum removing air, moisture and volatiles in resin
- Debulking, vacuum removing air, moisture and volatiles in the prepreg laminate and resin
- Thermal history control (out time, cure cycle) to reduce moisture driven bubble growth



Mohseni, M. (2020). *Experimental Study and Analytical Approaches to Avoid Matrix Defects during Composites Manufacturing*. University of British Columbia.

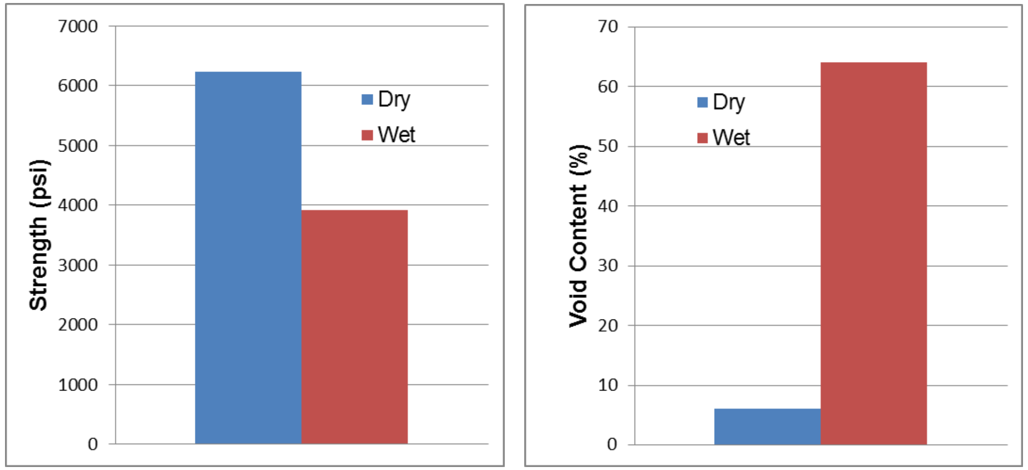
STORAGE AND HANDLING

- Prepregs are adversely effected by moisture
- Prepregs require cold transport and storage
- Many prepregs require moisture control during transportation and storage
- Moisture can condense on the exterior surface of raw materials and absorb into the resin
- This is particularly important when opening packages immediately after removing them from the freezer

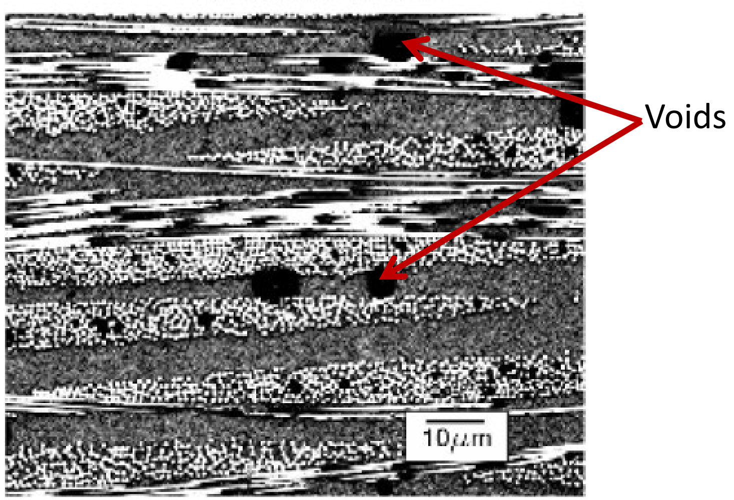
https://www.pxfuel.com/en/search?q=moist+glass
FAA CMfgT Course, Convergent

STORAGE AND HANDLING

- Excess moisture within prepreg can significantly reduce the final properties of the composite part
- Moisture in the layup can turn to vapor during heating, which can lead to excess porosity in the final part
- Even small amounts of water can lead to large porosity



Effect of pre-bond moisture on the strength of composite lap joints



Source: FAA CMfgT Course, Convergent

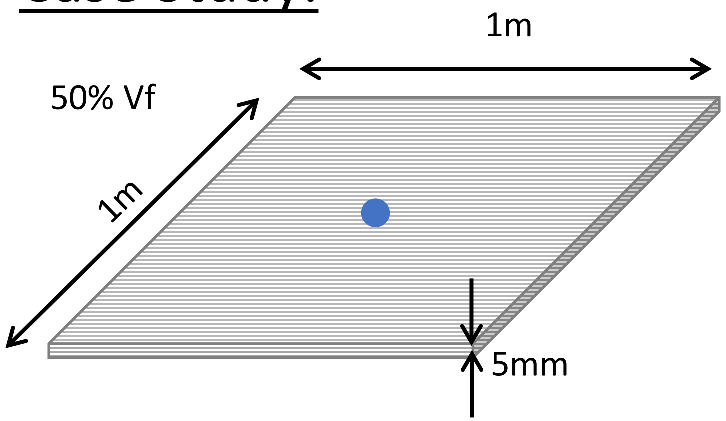
MOISTURE DISSOLVED IN RESIN

As an example, for polyester resin:

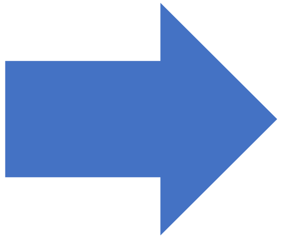
	Moisture
Saturation Concentration	≈ 0.5 to 1 wt% [1,2]
Equivalent Volume in Porosity	≈ 800 vol%

[1] Dhakal et al, 2007
[2] Chin et al, 1998

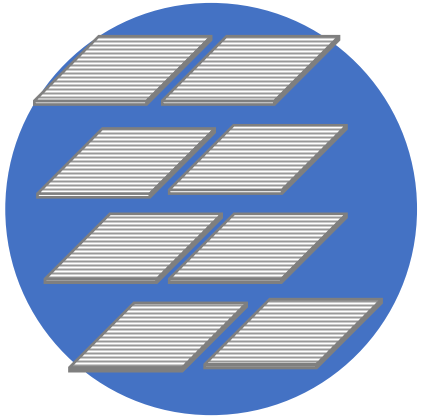
Case Study:



Laminate Volume: 5E-3 m^3



Translates to…

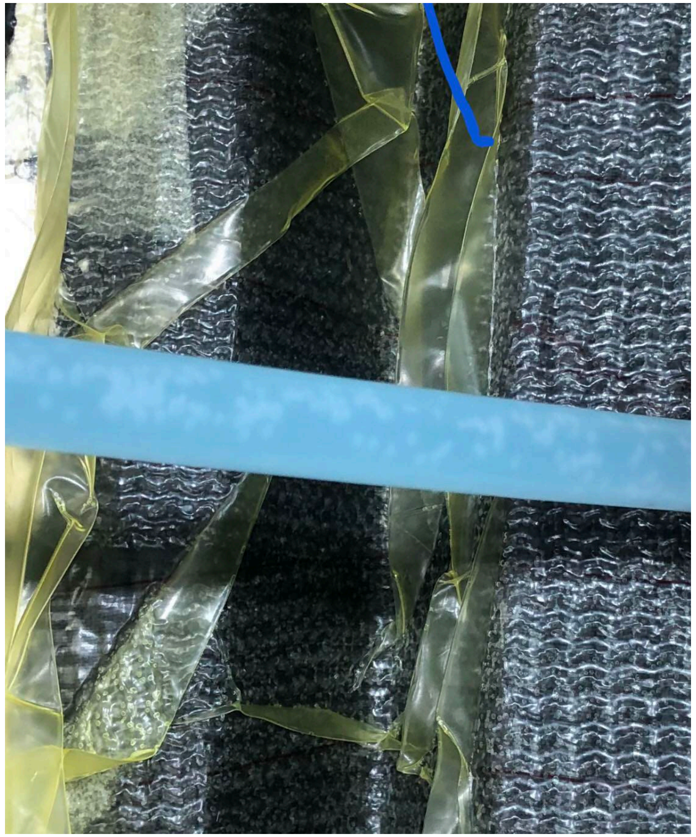


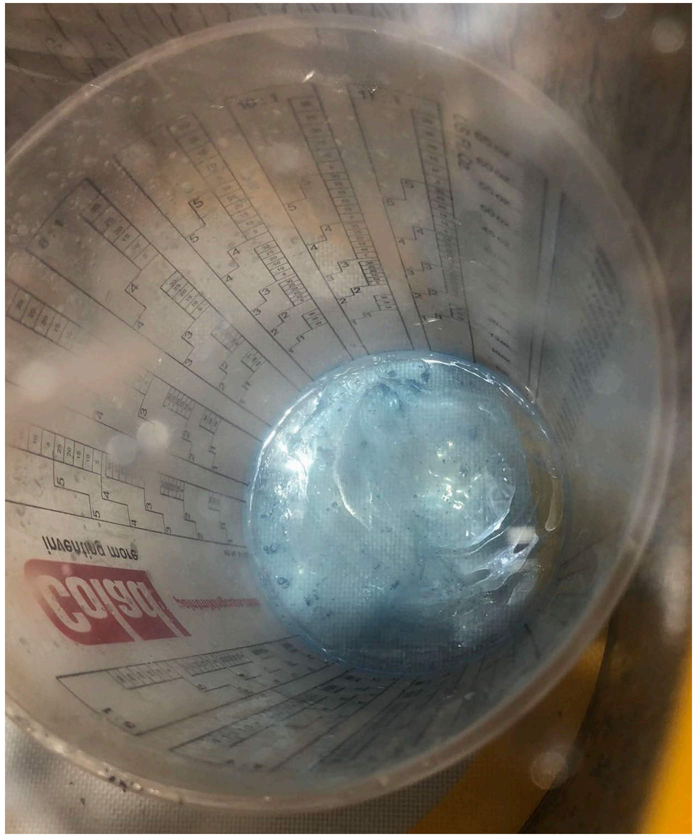
Water Vapour Volume: 4E-2 m^3
(8X Laminate Volume)

TAKE AWAY:

Dissolved gas can lead to high levels of porosity as a result of outgassing under vacuum.

OUTGASSING





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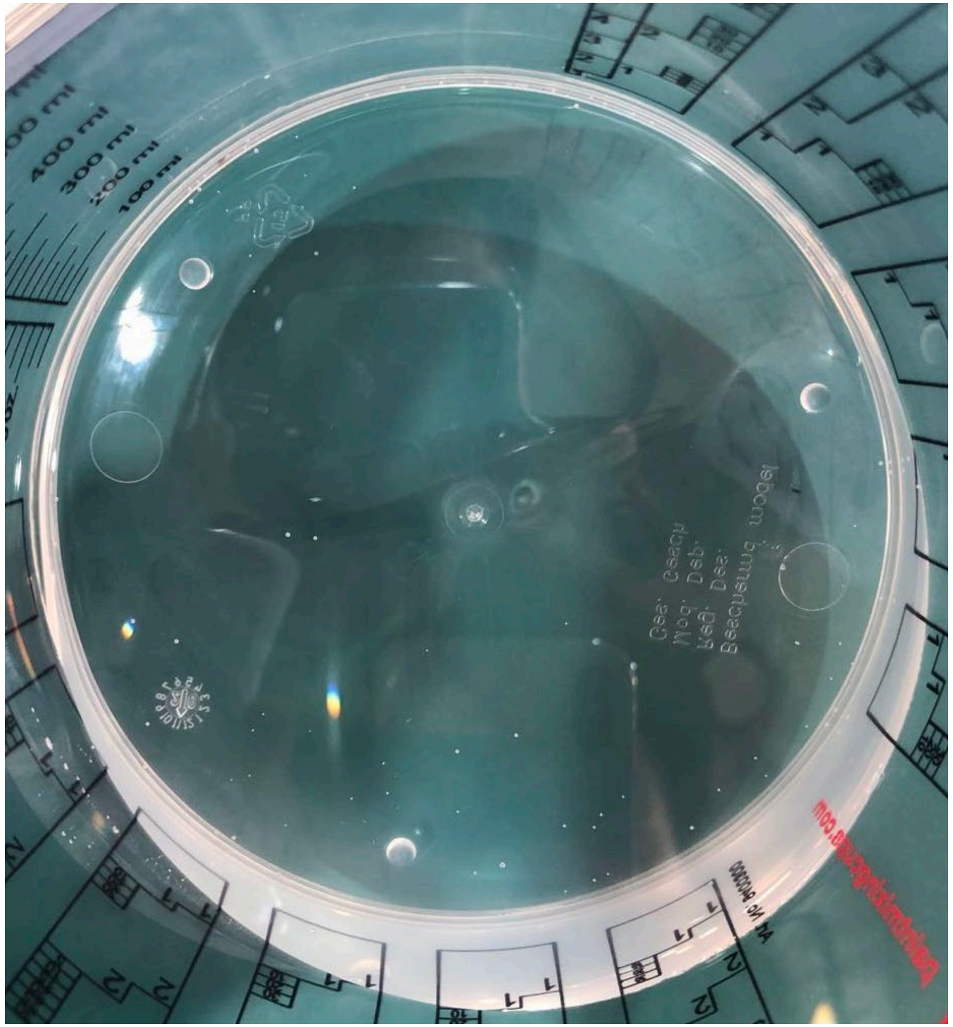
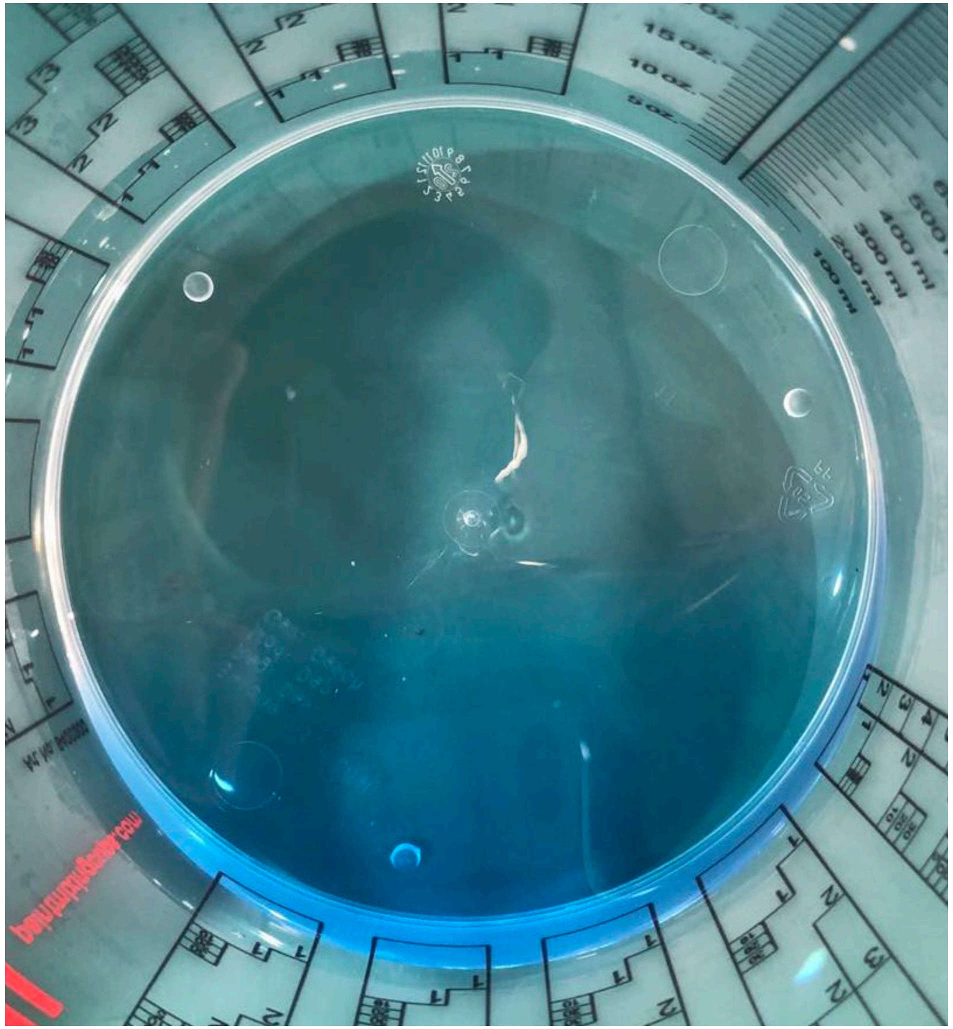
OUTGASSING





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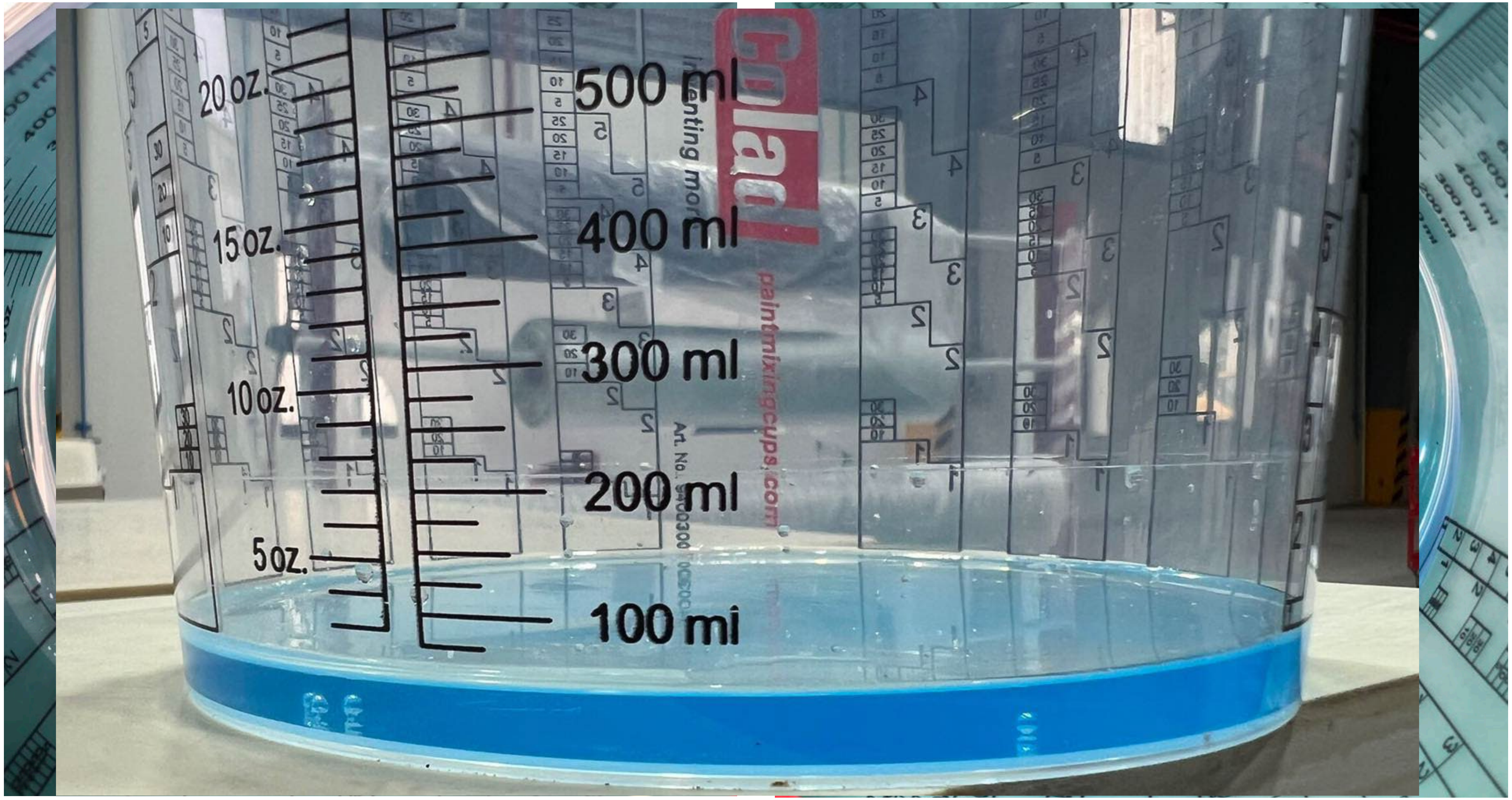
OUTGASSING



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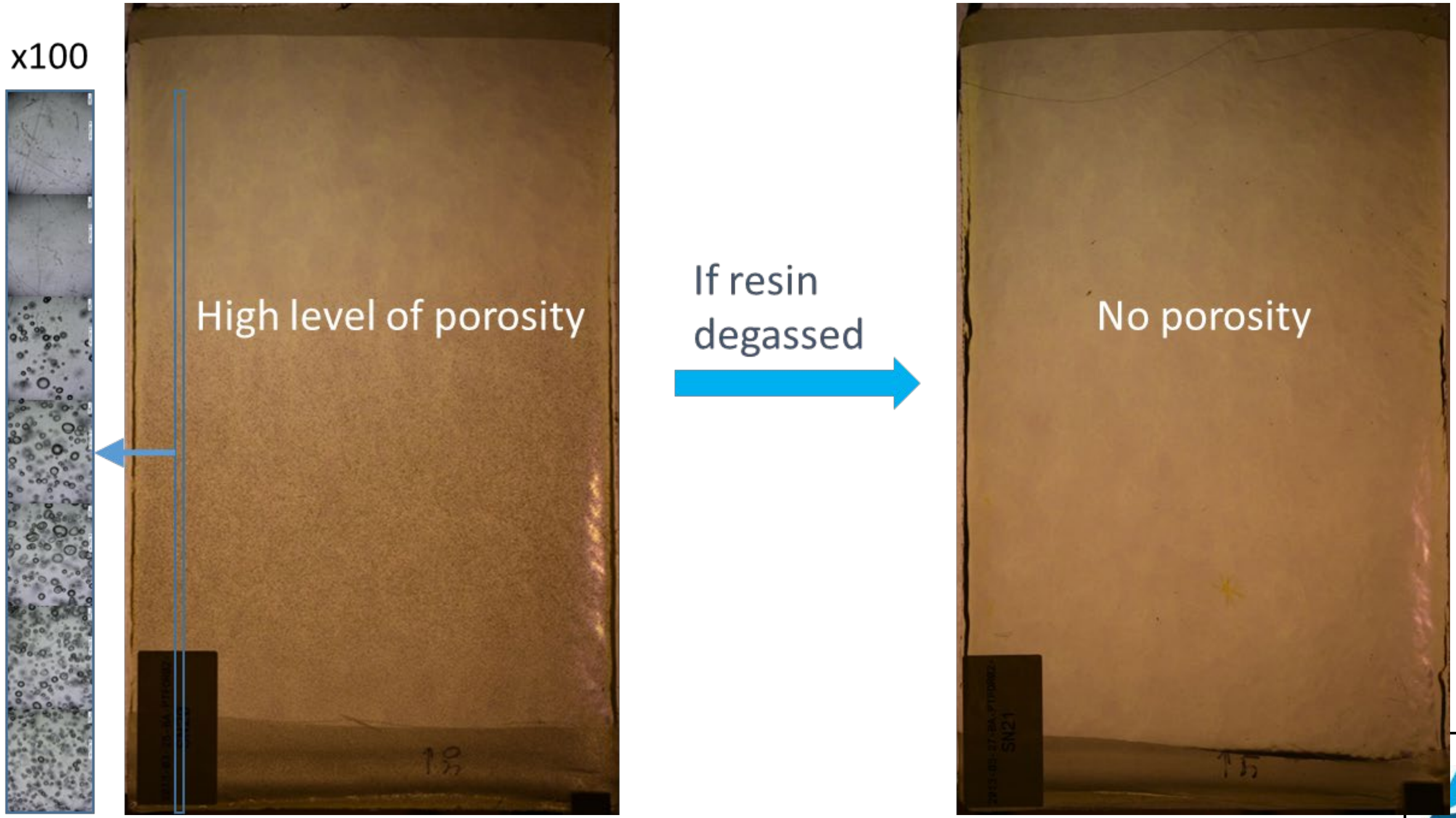
DEGASSING

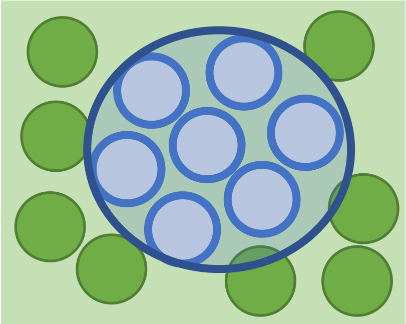
- Degassing - holding liquid resin under vacuum to remove moisture and volatiles is a common practice for liquid composite moulding
- Parameters that affect degassing rate:
  - Nucleation – porous media, plastic container
  - Temperature
  - Hydrostatic pressure
  - Agitation



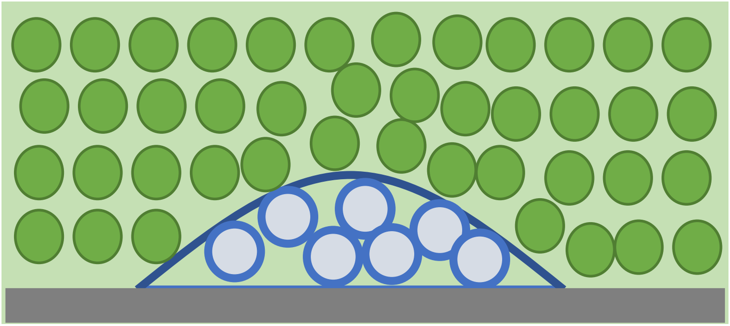
DEGASSING - EFFECT OF NUCLEATION MATERIALS

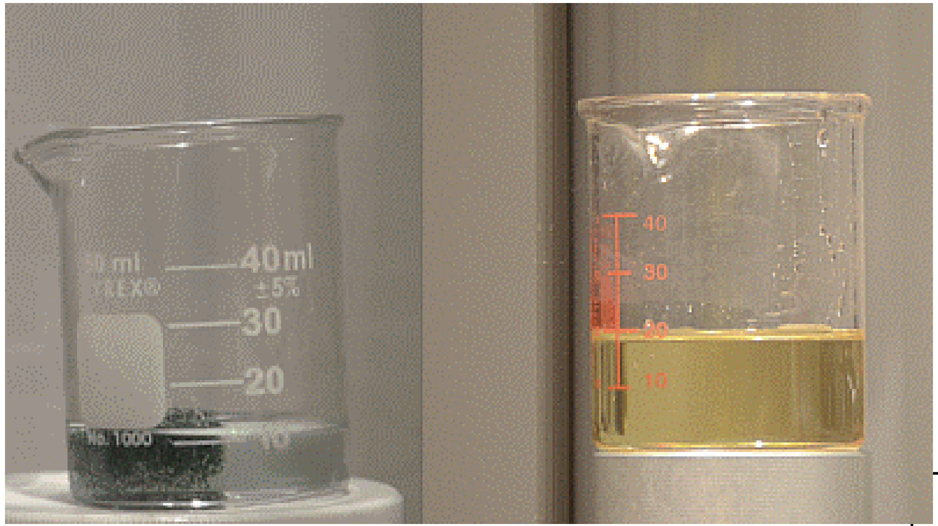
Two pathways to bubble nucleation:

Homogeneous Nucleation **VS** ***Heterogeneous Nucleation***



$$\gamma_{Hom} \gg \gamma_{Het}$$





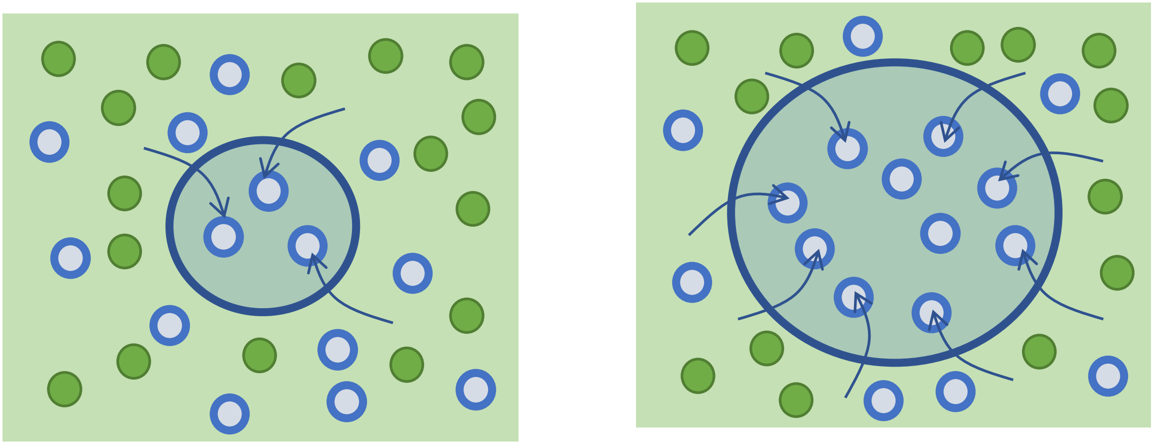
TAKE AWAY:

Nucleation materials can be used to speed up bubble nucleation

DEGASSING - EFFECT OF TEMPERATURE AND HYDROSTATIC PRESSURE

- **Increasing temperature** T ↑

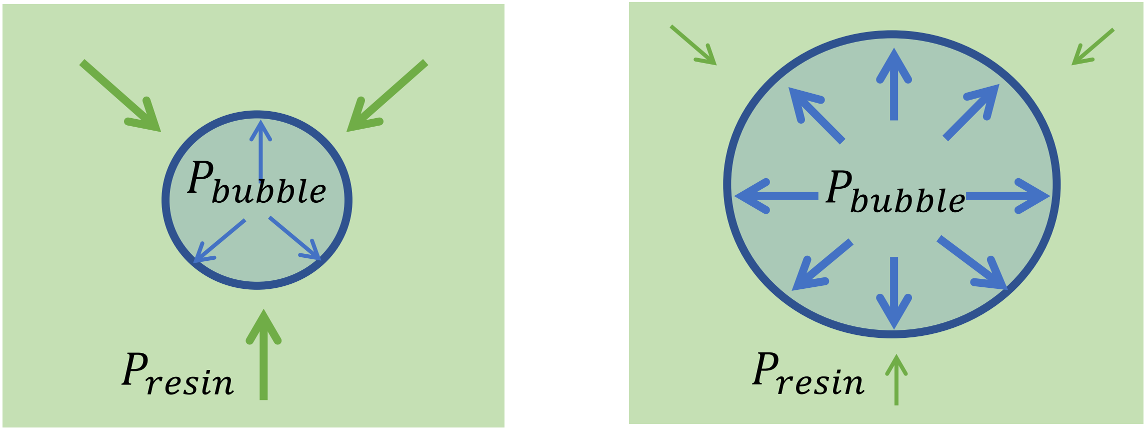
Rate of bubble growth increases with temperature (increases diffusion rate)



- **Decreasing hydrostatic resin pressure** P ↓

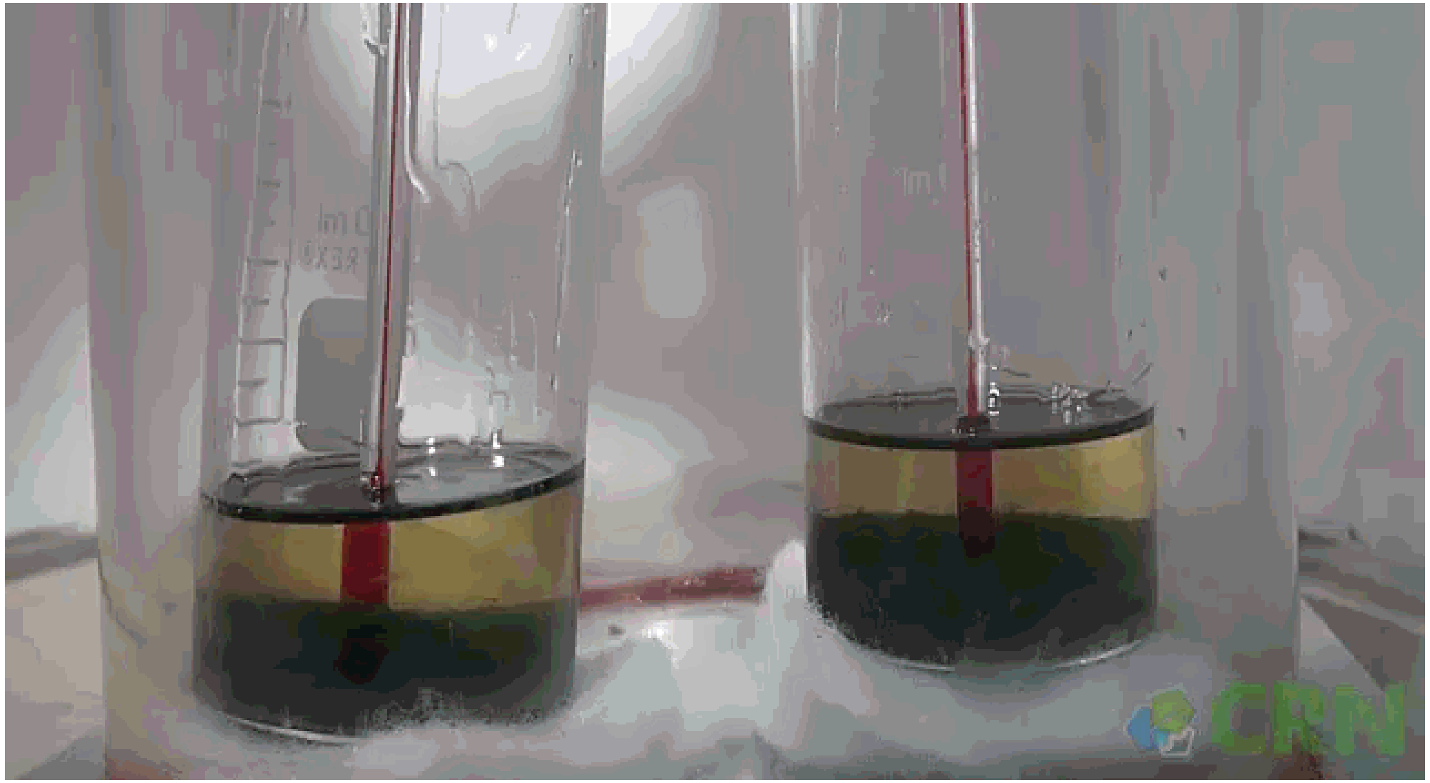
Bubble grows when

$$P_{bubble} > P_{resin}$$



DEGASSING - EFFECT OF TEMPERATURE

Preheated (34°C) Not heated (22°C)

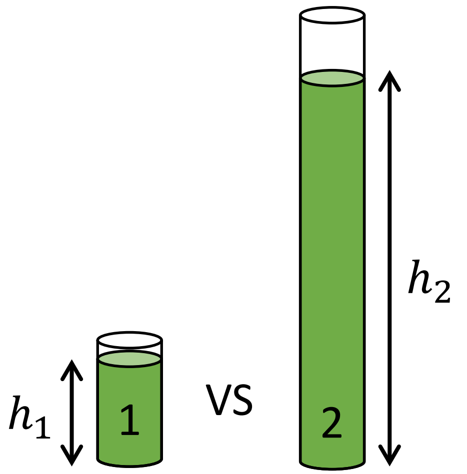


TAKE AWAY:

Raising temperature increases degassing kinetics by speeding up the growth rate of bubbles and bubble evacuation

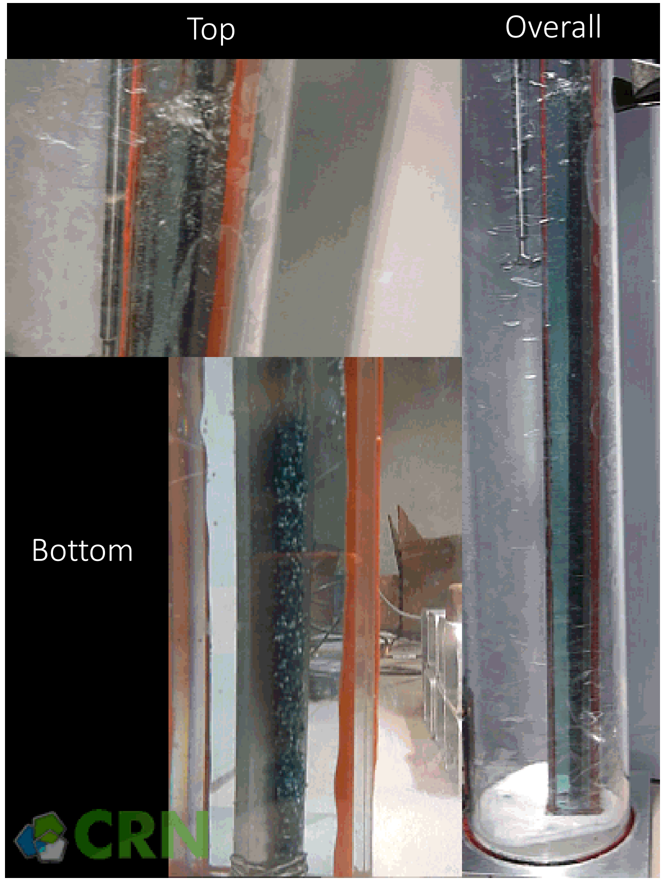
DEGASSING - EFFECT OF HYDROSTATIC PRESSURE

In some cases, resin hydrostatic pressure (P_{resin}) is great enough to slow down degassing:



$$P_{resin,1} < P_{resin,2}$$

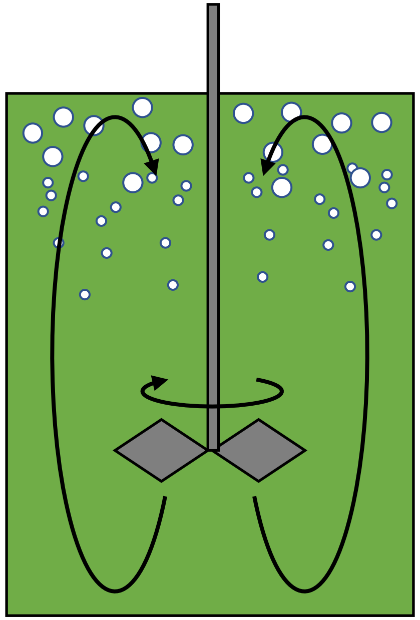
Beginning



End

DEGASSING - EFFECT OF AGITATION

By using a mixer to stir the resin during degassing, un-degassed resin from the bottom can be brought to the top (less hydrostatic pressure)

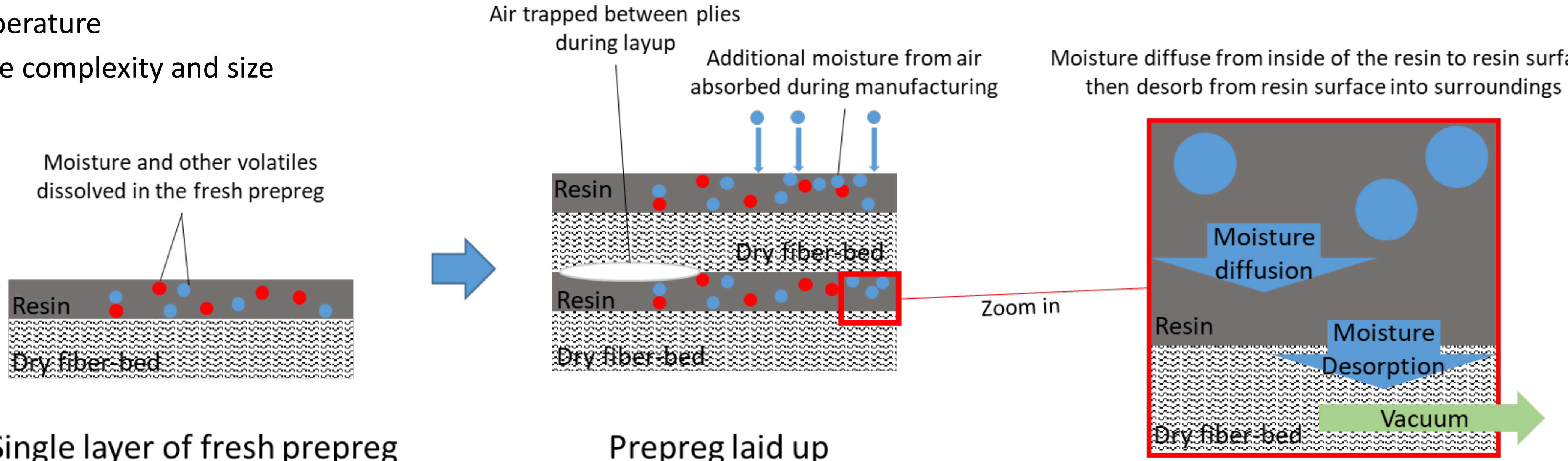


TAKE AWAY:

Hydrostatic pressure effects can be overcome by using a mixer to stir up un-degassed resin

DEBULKING

- Debulking - putting a pre-cured layup under vacuum for a period of time to remove entrapped air and moisture between the plies and conform/consolidate the plies to the tool
- Common practice for composite parts made from prepreg
- Typically de-bulked every 3-5 layers for 5-15 minutes depending on the shape complexity
- Parameters that affect debulking:
  - Vacuum level
  - Temperature
  - Shape complexity and size
  - Time



Single layer of fresh prepreg

Prepreg laid up

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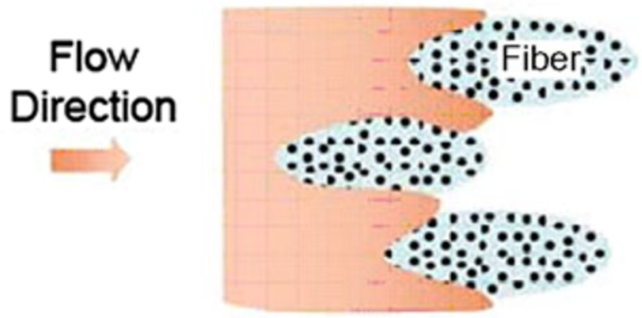
WHAT HAPPENS WHEN RESIN FLOWS THROUGH THE FIBER BED?

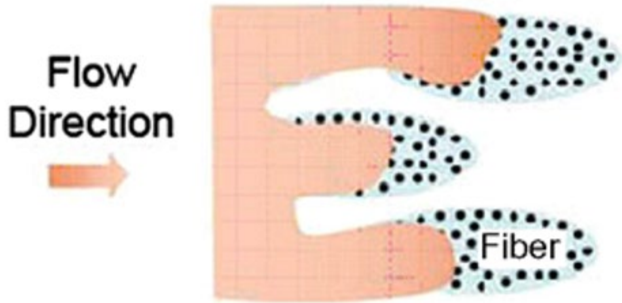
As the resin flows through the fiber bed, there are two types of flows:

- The flow through the gaps between the fiber tows
- The flow within the fiber tows (between individual fibres)

Macroscopic flow

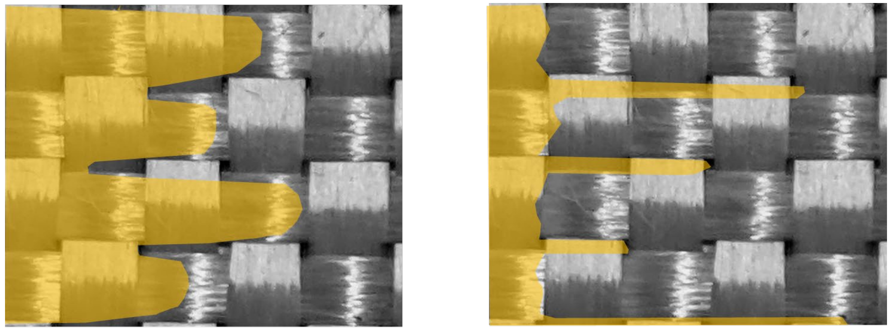


Microscopic flow

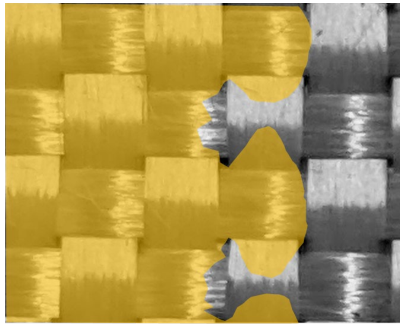


Leclerc, Jean Sébastien; Ruiz, Edu (2008). "Porosity reduction using optimized flow velocity in Resin Transfer Molding". **39** (12). Elsevier Ltd. doi:10.1016/j.compositesa.2008.09.008. ISSN 1359-835X.

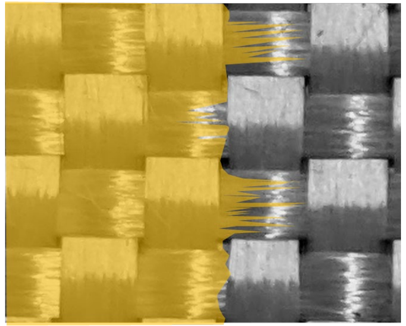
Fingering



Cross flowing



Wicking



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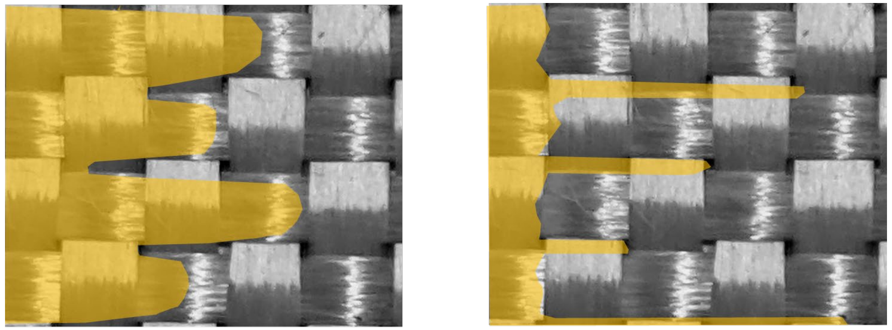
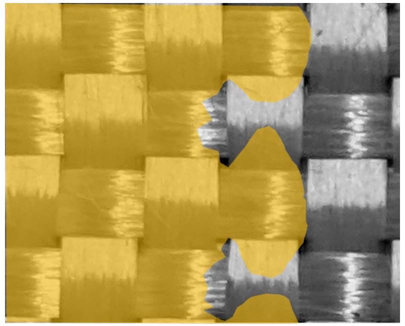
POROSITY WHEN RESIN FLOWS THROUGH THE FIBER BED

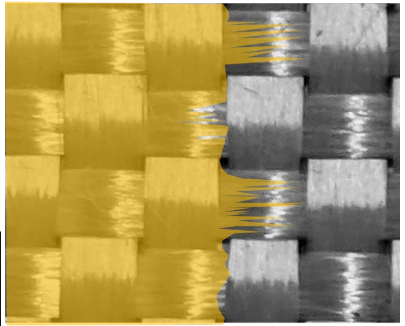
Fingering



Cross flowing



Wicking



- Fingering/cross flowing/wicking or the combination, trap gas and forms porosity
- Gas can be trapped in between tows or within tows
- The trapped gas (bubbles) could potentially be removed after the mould is filled

POROSITY WHEN RESIN IS FLOWING THROUGH THE FIBER BED

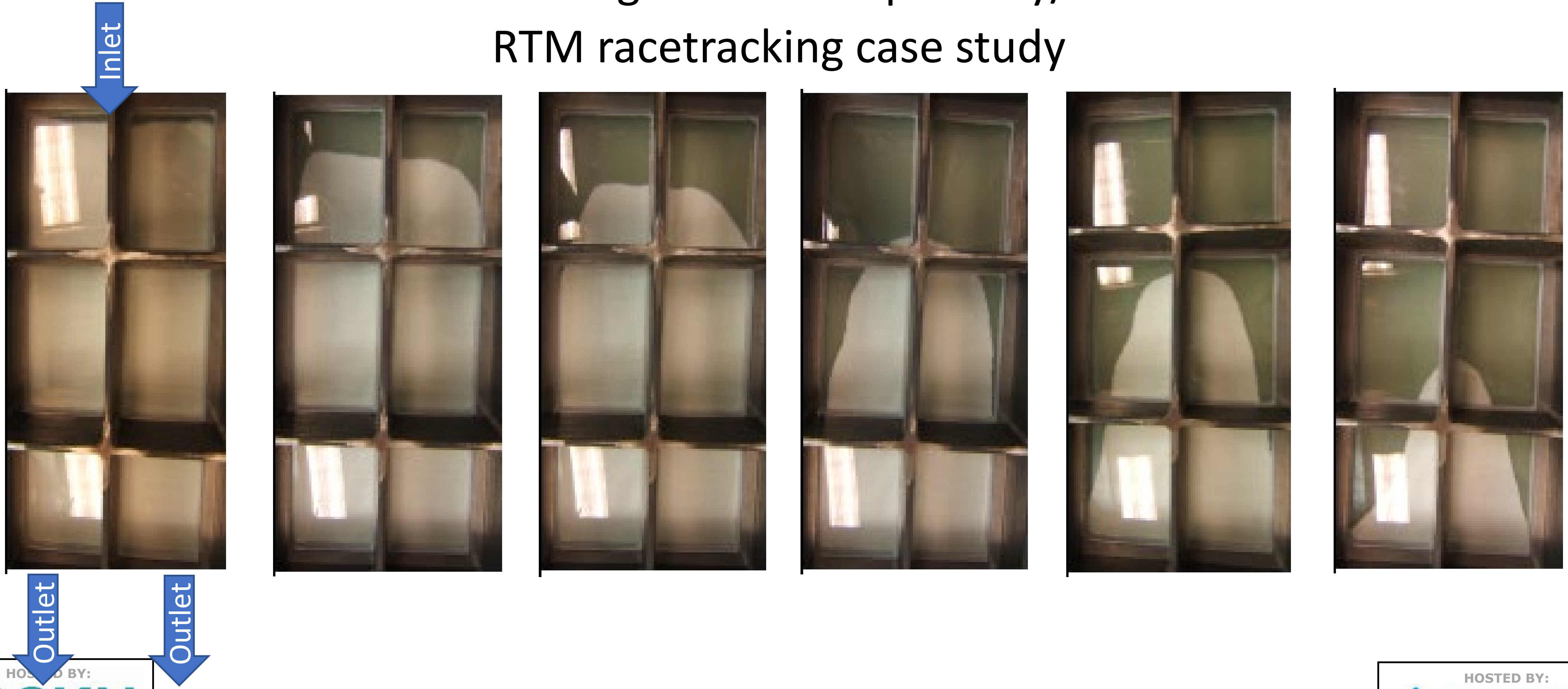
- Fingering/cross flowing/wicking as the resin flows through the fiber bed depends on the following parameters:
  - Fiber architecture – gaps, stitches, fiber count per tow, symmetry, tow direction, crimps, binder particles
  - Flow speeds of the two types of flows, which are dependent on:
    - Resin viscosity
    - Pressure differential
    - Permeabilities – into the tow and overall porous fiber bed
    - Capillary pressure
  - Viscous forces (drag), which depends on:
    - Sizing
    - Surface tension
  - Consumables (peel ply, flow mesh), release agent
  - Part geometry

Note on flow speeds:
- **NOT** a single number. Each flow has its own speed
- Speeds change as the resin flows from the inlet to outlet
- Speeds can also be controlled by adjusting the flow rate at the inlet
- Speeds vary constantly at different locations within a laminate

ISSUES WITH RESIN FLOW THROUGH FIBER BED

Racetracking can lead to porosity/voids

RTM racetracking case study



LIST OF POROSITY SOURCES AND SINKS

Exhaustive list of porosity sources

- Fiber/resin incompatible
- Entrapped gas in setup and consumables
- Over-bleeding
- Empty space from shape (bridging, ply drop off)
- Tool dimensional error
- Core absorbing resin cause face sheet to starve
- Face sheet dimpling and pull away from the tool
- Moisture absorbed in core
- Not dissolved gases and volatiles in the resin (visible bubbles)
- Resin shrinkage
- Lack of consolidation

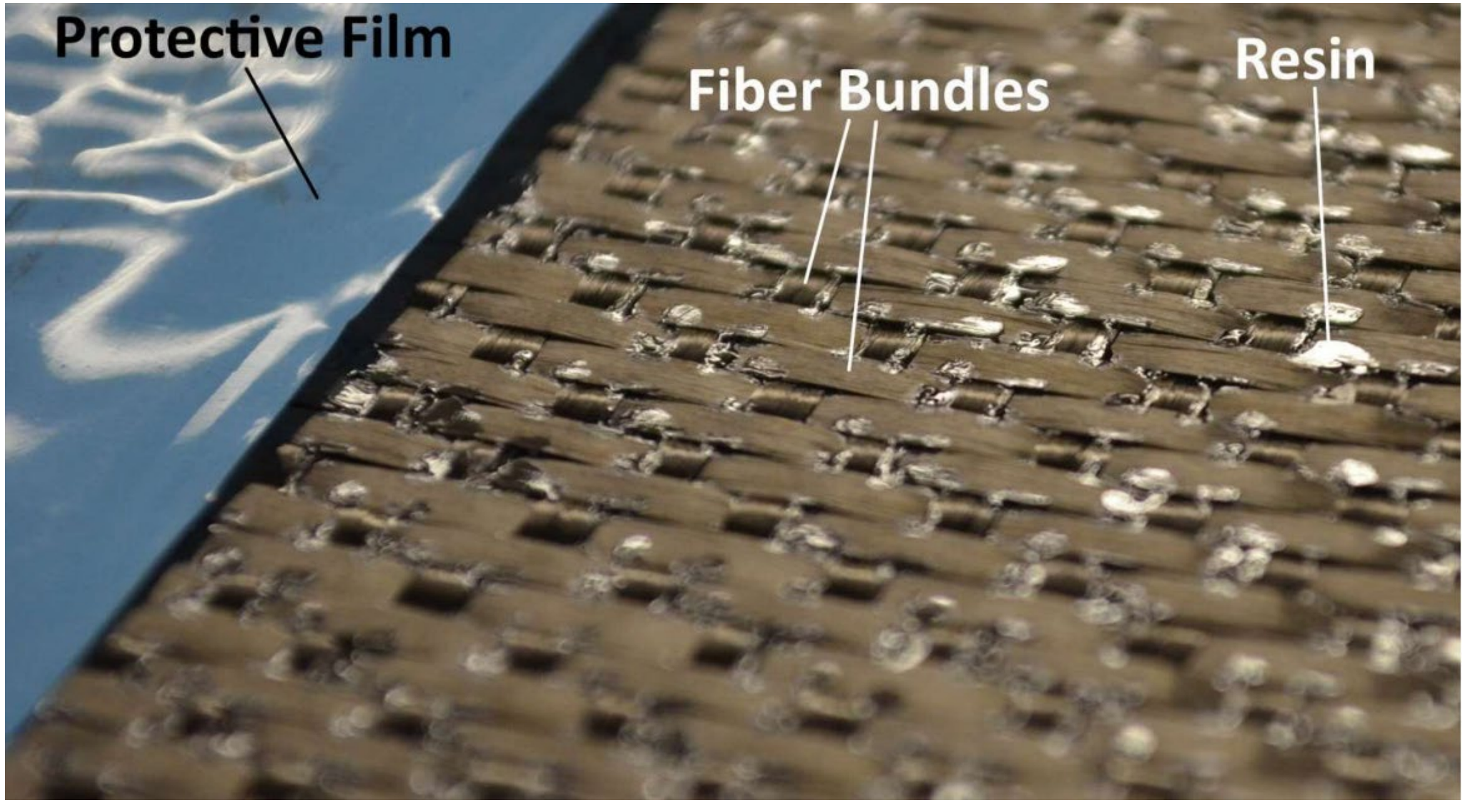
- Tool/bag/vacuum leak
- Outgassing of absorbed volatiles in the fiber
- Outgassing of dissolved gases and volatiles in the resin
- Moisture in the fiber
- Moisture in the resin
- Formation of cure reaction by-products
- Issues with resin flowing through fiber bed
- Insufficient gas transport within the laminate

Exhaustive list of porosity sinks

- Resin formulation
- Prepreg design (architecture, degree of impregnation)
- Resin/prepreg storage and handling conditions
- Degassing, vacuum removing air, moisture and volatiles in resin
- Material deposition techniques
- Shape design (avoiding sharp corners, and aggressive ply drop offs)
- Proper amount of bleeding and breathing using consumables
- Debulking, vacuum removing air, moisture and volatiles in the prepreg laminate and resin
- Eliminating vacuum leaks in tool and vacuum bag setup including consumables
- Eliminating gaps between tool and part (fiber bridging, underfill in match-die)
- Pressure (from roller/autoclave/press) to provide consolidation
- Control resin flow speed (pressure differential)
- Control resin flow directions (injection strategies)
- Control thermal history (out time, cure cycle) to reduce moisture driven bubble growth

"BREATHING" IN PREPREGS

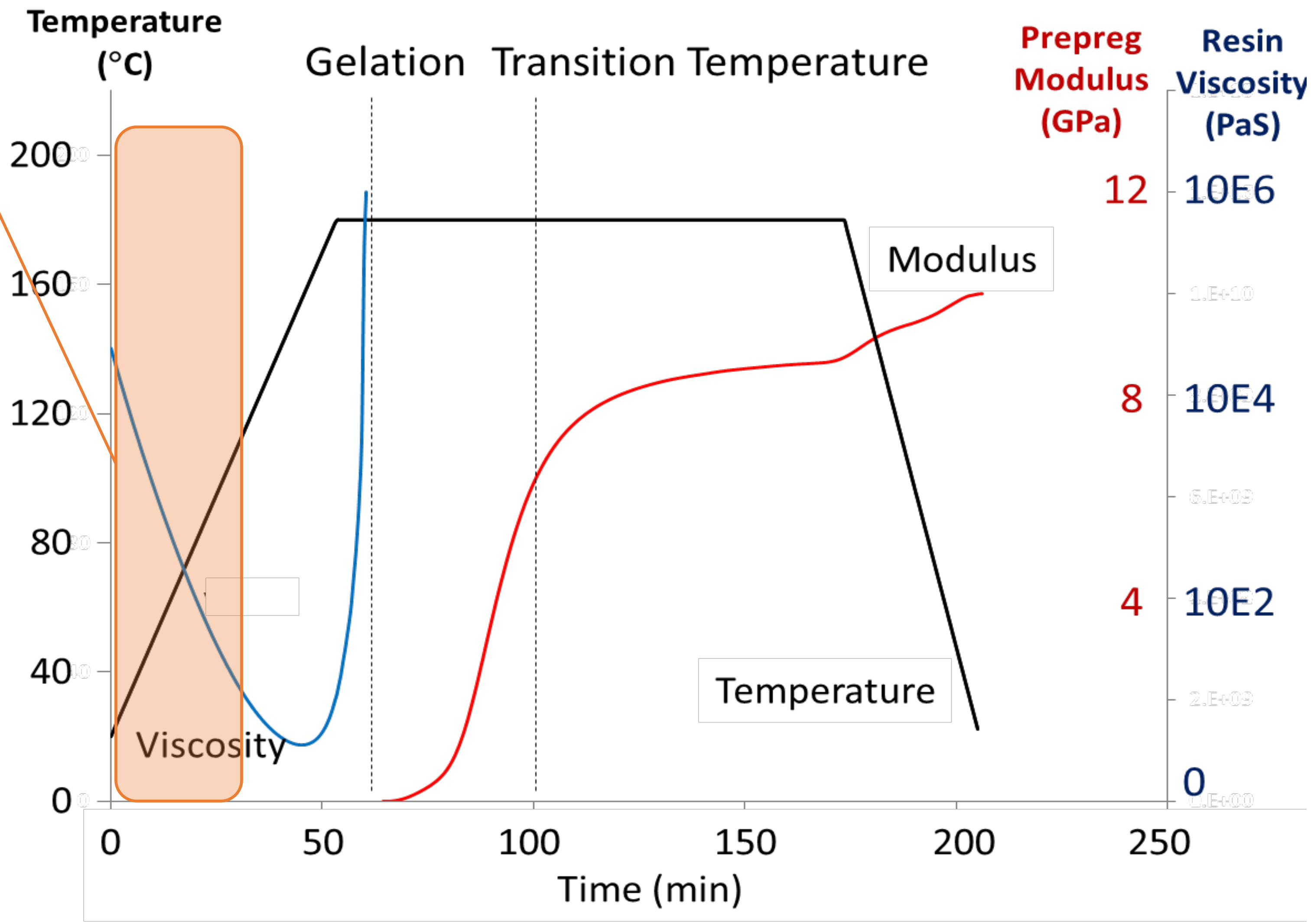
- Recent generations of prepregs are partially impregnated – leaving voids in-between tows (intra-tow voids) that act as inter-connected in-plane breathing network for gas extraction. A.K.A Engineered vacuum channels (EVaCs)
- During vacuum compaction/debulking, gases can evacuate via the breathing network
- Breathing can be in-plane or through the thickness
- In-plane breathing rely on edge breather such as dry glass fiber, cork or other breathing dams



RESIN INFILTRATION AND GAS TRANSPORT

During cure as temperature increase, resin viscosity drops, resin infiltrates the network and fill the voids. EVaCs close

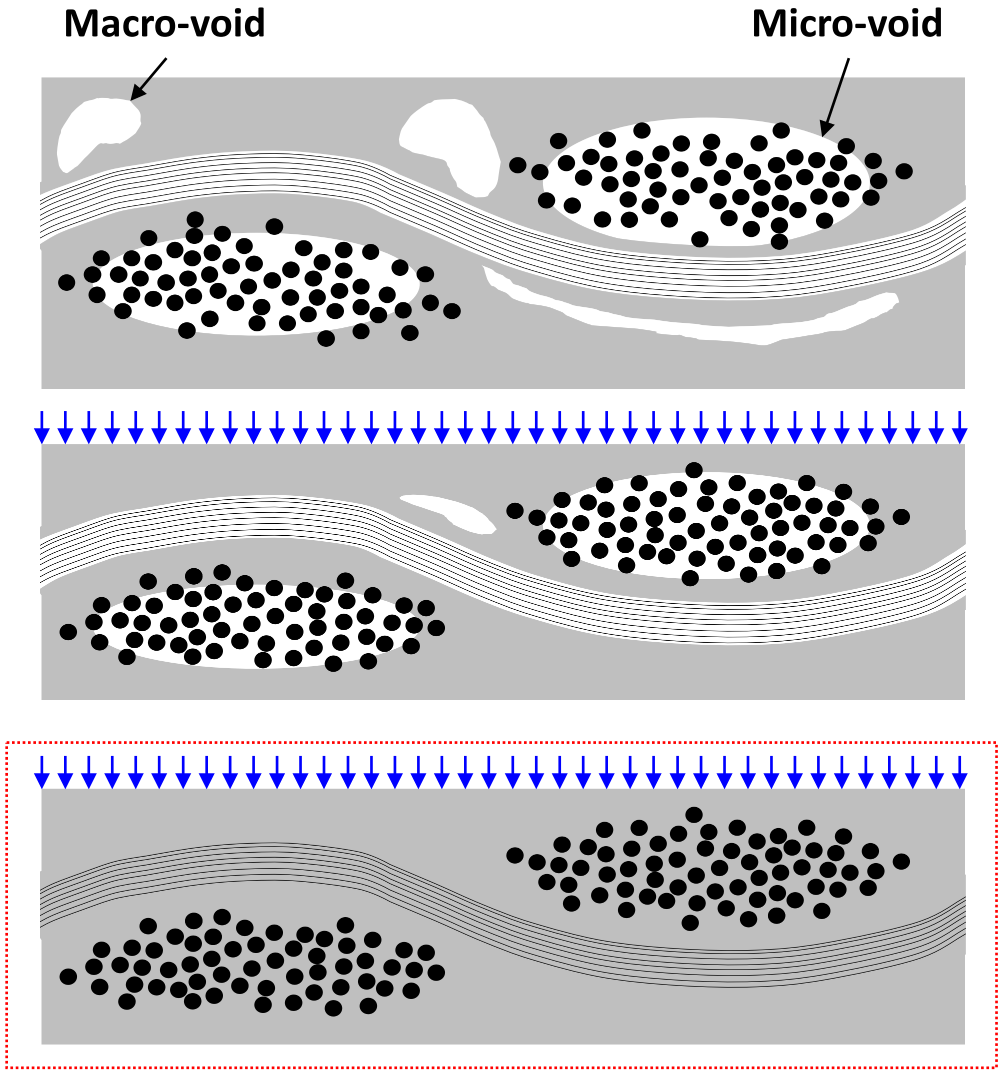
- Once EVaCs are closed, entrapped gas is difficult to remove
- As temperature continues to increase, vapor pressure of the gases (moisture, volatile etc.) increases, forming porosity. Resin pressure needs to be maintained higher than the vapor pressure to suppress the porosity



RESIN INFILTRATION

Modified from: Jeremy Wells 2014, CRN Advisory Board Meeting

RESIN FLOW – IMPREGNATION MECHANISMS



Layup
Heat: No
Vacuum: No

Layup and bagging

Vacuum hold
Vacuum: Yes
Heat: No

Air evacuation through dry tows

Heated processing
Vacuum: Yes
Heat: Yes

Air evacuation and tow impregnation

Courtesy of Dr. Pascal Hubert, McGill

RESIN INFILTRATION AND GAS TRANSPORT

What stages in the manufacturing process do resin infiltration and gas transport affect final porosity level?

- Material deposition/debulking
- Thermal transformation

What manufacturing process does resin infiltration and gas transport affect?

- Processes with pre-preg and out-of-autoclave prepregs

Sinks for non-breathable resin infiltration and gas transport

- Debulking, vacuum removing air, moisture and volatiles in the prepreg laminate and resin
- Proper amount of breathing using appropriate consumables
- Pressure (from roller/autoclave/press) to provide consolidation
- Control thermal history (out time, cure cycle) to reduce moisture driven bubble growth

Jeremy Wells 2014

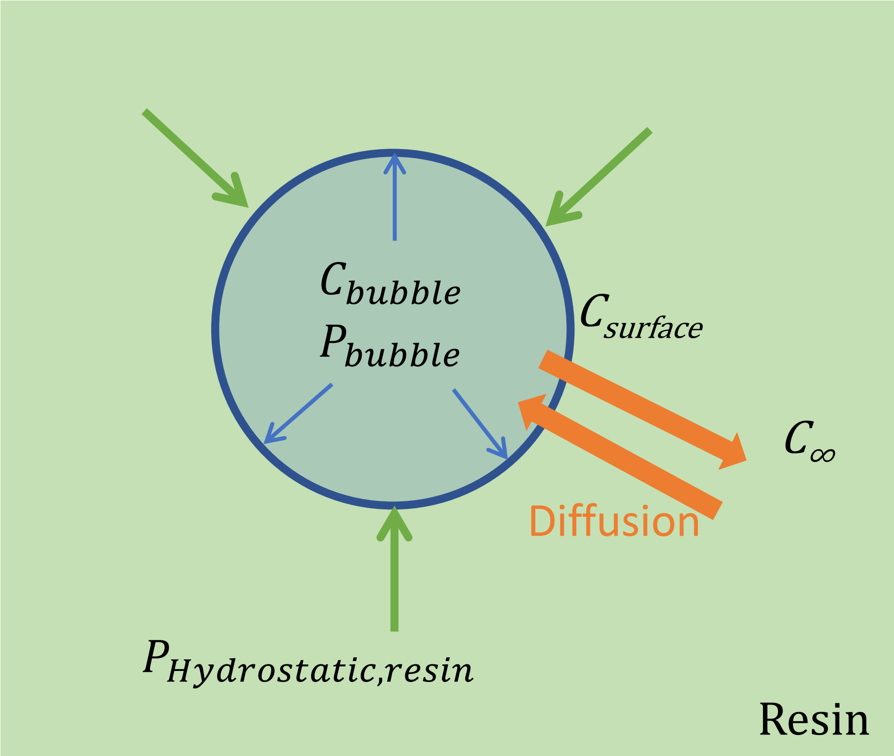
PARAMETERS OF CONCERN FOR GAS TRANSPORT [1,2]

- Cure cycle – time, temperature, pressure
- Vacuum – timing, location, magnitude
- Fibre bed properties – material, architecture, in-plane and through thickness permeability
- Matrix properties – material, viscosity, surface tension, cure kinetics
- Surface characteristics between fiber and matrix
- Volatiles – content, concentration, diffusivity, solubility

1. Farhang, L., & Fernlund, G. (n.d.). *Void Morphology, Void Evolution and Gas Transport in Out-of-Autoclave Prepregs*.
2. Wood, J. R., & Bader, M. G. (n.d.). *Void control for polymer-matrix composites (1): theoretical and experimental methods for determining the growth and collapse of gas bubbles*.

HOW DOES A BUBBLE GROW? - DIFFUSION CONTROLLED BUBBLE KINETICS

- Mechanisms:
  1. Gas inside the bubble can diffuse into the surrounding resin and get dissolved
  2. Dissolved gas in the surrounding resin can diffuse into the bubble
  3. Bubble can grow when temperature increase
  4. Bubble can grow when pressure decrease (vacuum)
- Parameters:
  - Concentration (gradient)– driving force for the diffusion
    - Concentration in resin
    - Concentration at resin bubble interface
    - Concentration within the bubble
  - Diffusion coefficient
  - Surface tension
- Knobs that can be controlled:
  - Time, temperature, pressure



ASSESSING/MEASURING POROSITY

Method	Measuring principle	Accuracy	Repeatability	Time	Cost	Limitation
Ultrasonic testing	Reflection of ultrasonic waves	± 1% (± 0.5% if material is well known)	Medium	Fast	High	Porosity content, shape, orientation. Sample shape
Acid digestion	Gravimetry	± 1% (± 0.5% if material is well known)	N/A (destructive)	Slow	Medium	Size and material properties
Microscopy	Image analysis	± 1%	N/A (destructive)	Fast to slow	Low to High	Size, porosity distribution
Computer tomography	Absorption of X-ray	± 0.5%	High	Medium	High	Size

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Recreated from: Kastner, J., Plank, B., Salaberger, D., & Sekelja, J. (2010). Defect and Porosity Determination of Fibre Reinforced Polymers by X-ray Computed Tomography. NDT in Aerospace 2010 - We.1.A.2, (February 2015), 1–12.

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Thank you for joining us!

Keep an eye out for upcoming AIM events:

An Introduction to Composites Sustainability
Hosted by Dr. Adam Smith
July 26, 2023
https://compositeskn.org/KPC/A338

And don't forget to visit the KPC for more information:

https://compositeskn.org/KPC

Today's Webinar will be posted at:
https://compositeskn.org/KPC/A337

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