



Effect of Composition Variation on Isothermal Rapid-Curing Resins for Aerospace Applications

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Welcome Slide

- Hailey Moore
- NASA Langley Research Center
- Materials Research Engineer
- Research Interests:
 - High-performance resins for aerospace applications
 - Evaluation of high temperature materials for aerospace applications
 - Solid-state batteries for extreme environments
- Master's Degree in Chemistry: University of New Hampshire
- Bachelor's Degree in Materials Science and Engineering: Virginia Tech





Outline

- Introduction
- Results
 - Isothermal processing temperature
 - Effect of catalyst concentration
 - Effect of epoxy additives
 - Effect of hardener additives
- Composite fabrication
- Concluding remarks



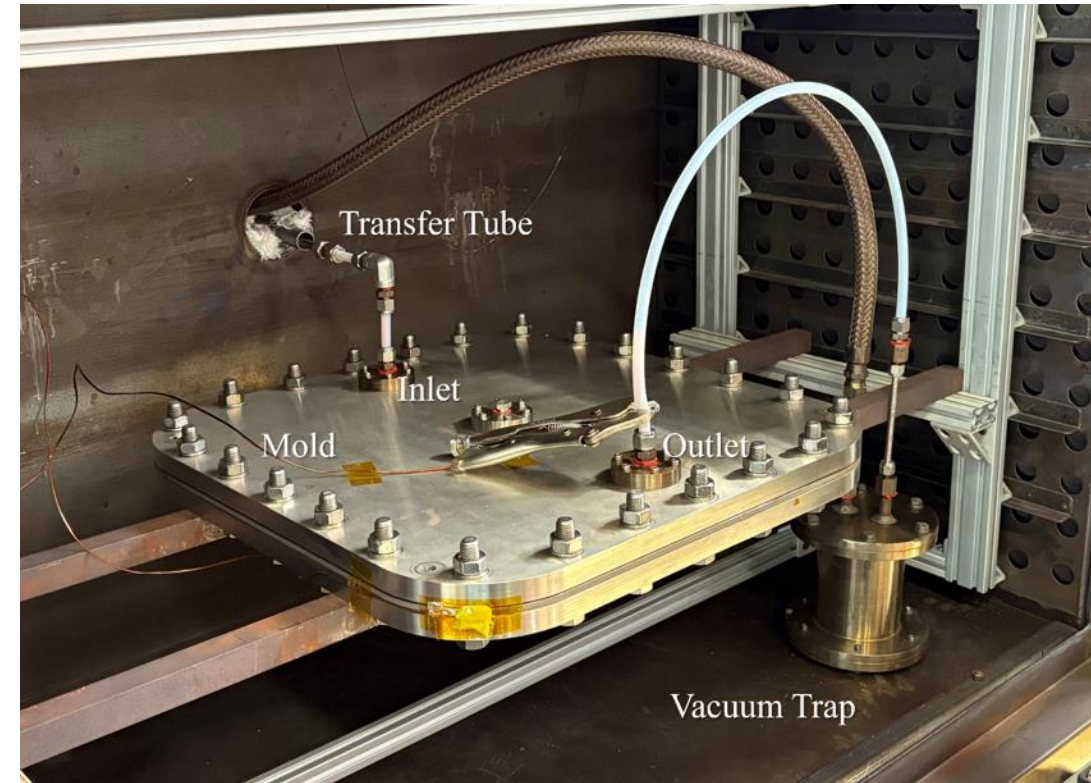
Introduction

- Projected demand in 2040 requires an increased rate of production of single-aisle composite aircraft by 4-6X over current state of the art
- Hi-Rate Composite Aircraft Manufacturing (HiCAM) Project
 - Evaluate technologies that enable high-rate production of aircraft to meet projected demand
 - Reduce production costs
 - Maintain high-performance composite properties



Introduction

- Resin Transfer Molding (RTM) is a potential enabling technology
 - Minimize part assembly time
 - Facilitate out-of-autoclave processing techniques
 - Produce high-quality parts reproducibly



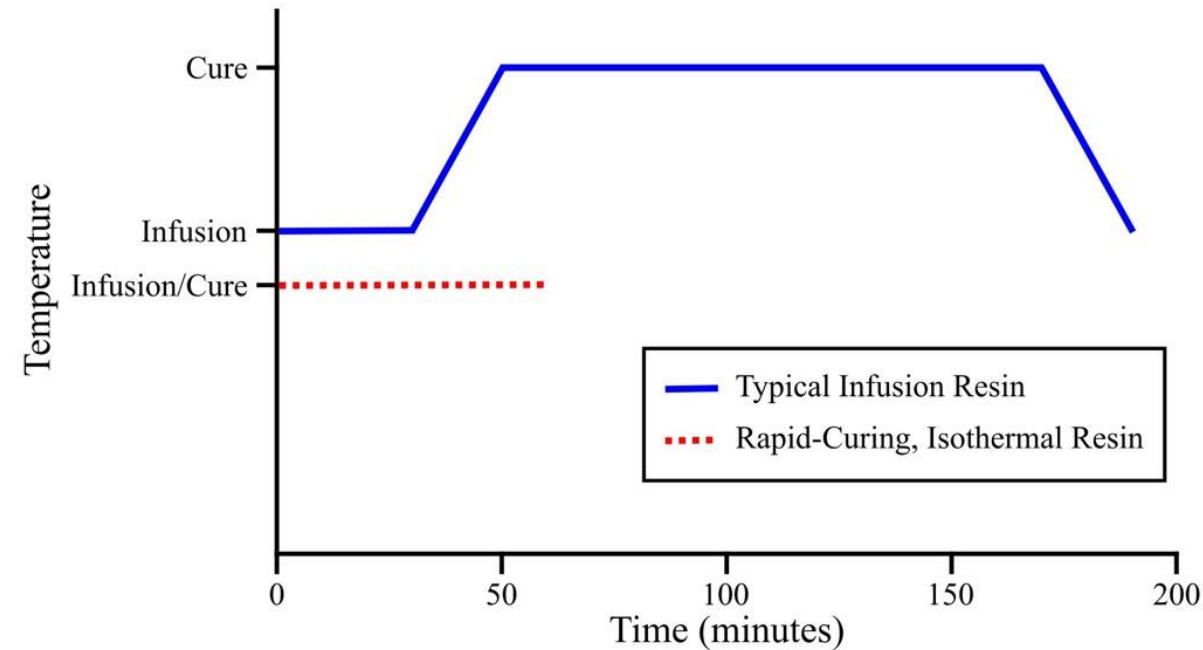


Isothermal Rapid-Curing Resins

Isothermal rapid-curing resins infuse and cure at the same temperature

Requirements

- Total infusion/cure time: **1 hour**
- Infusion window: **30 minutes**
 - Complex viscosity ≤ 1000 cps
- Part removal at the isothermal temperature
- Free-standing post-cure
- High-performance aerospace grade composite mechanical properties
 - Baseline: Hexcel HexFlow[®] 1078

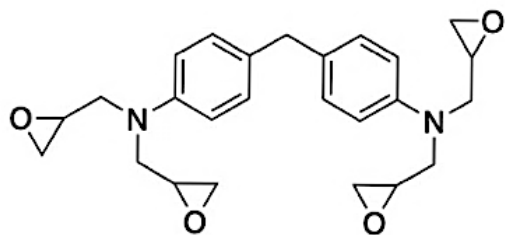




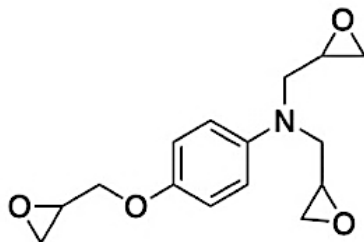
Resin System

Core Isothermal Rapid-Cure Resin System

Core Epoxies

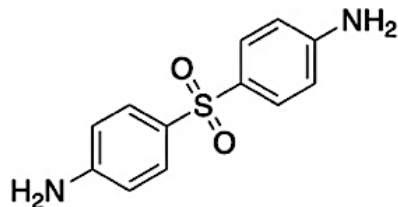


TGMDA

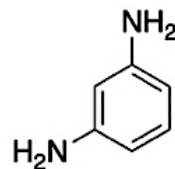


DGOA

Core Amines

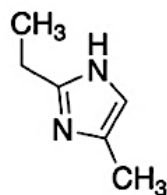


4,4'-DDS

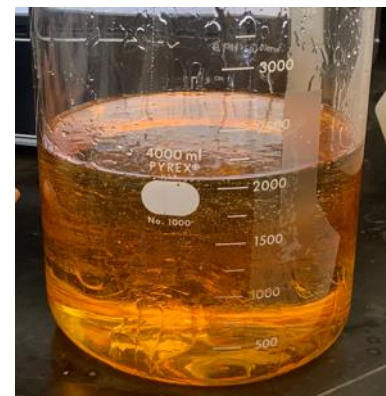


mPDA

Catalyst



2Et4MeIm



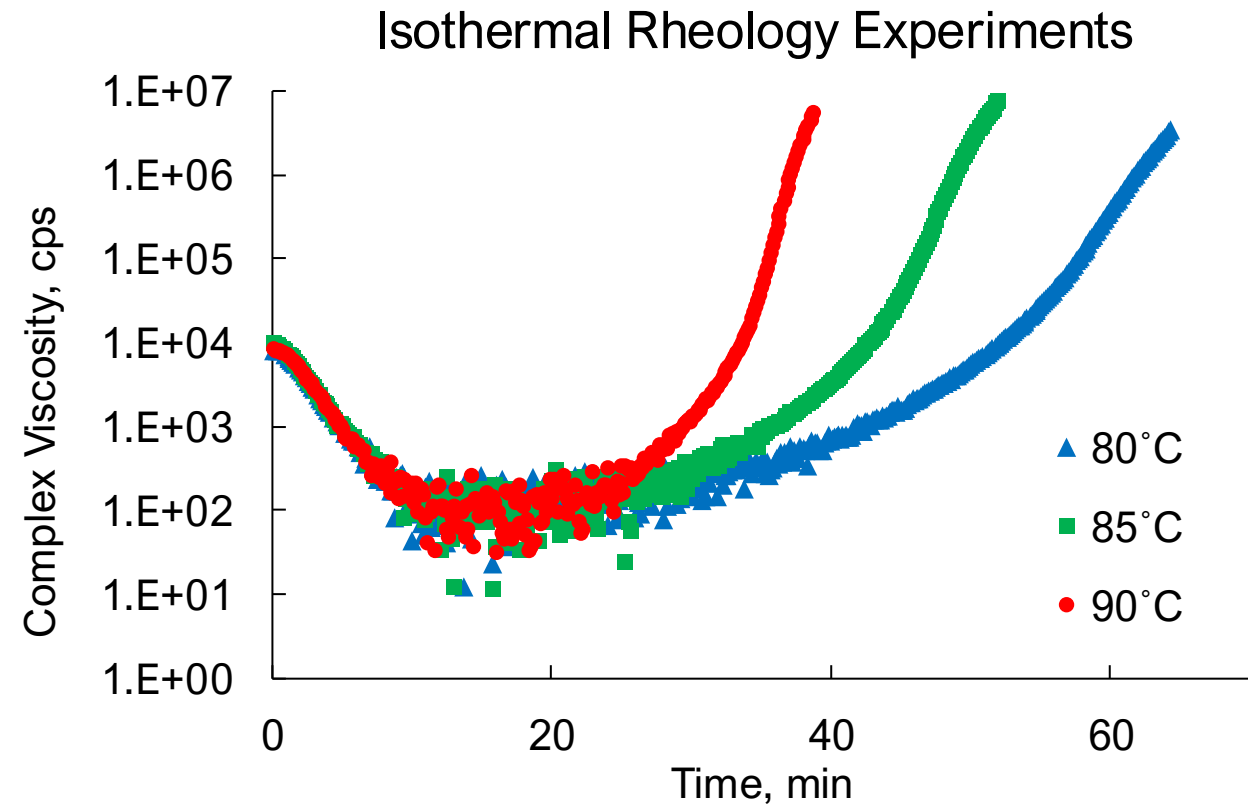
Preparation

1. Melt hardener composition under a blanket of nitrogen at $\sim 150^{\circ}\text{C}$
2. Add epoxy composition to cooled hardener mixture and mix manually at room temperature
3. Degas while stirring under vacuum at room temperature



Isothermal Processing Temperature

1. Conduct isothermal rheology experiments at three temperatures – 80°C, 85°C, and 90°C



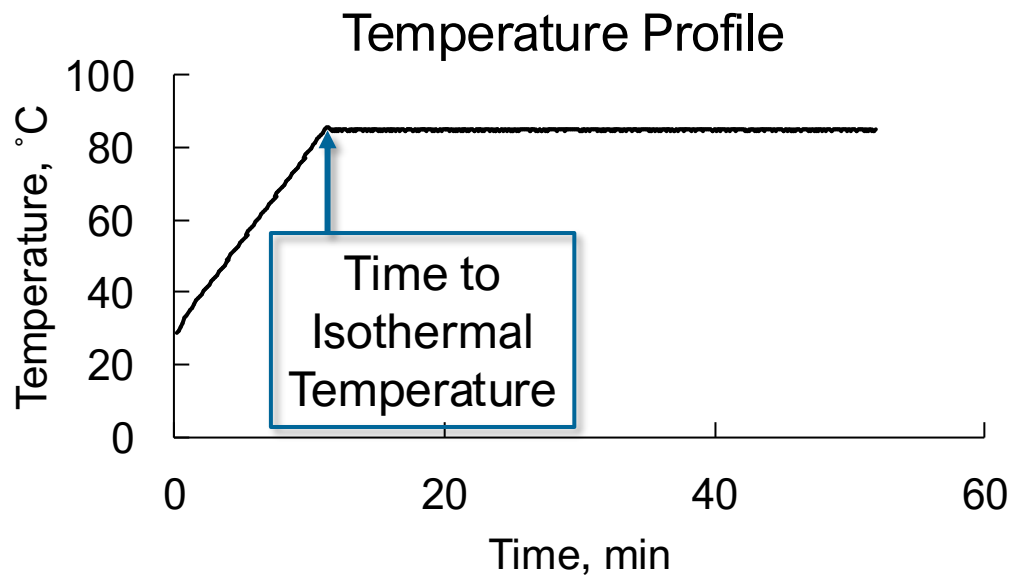
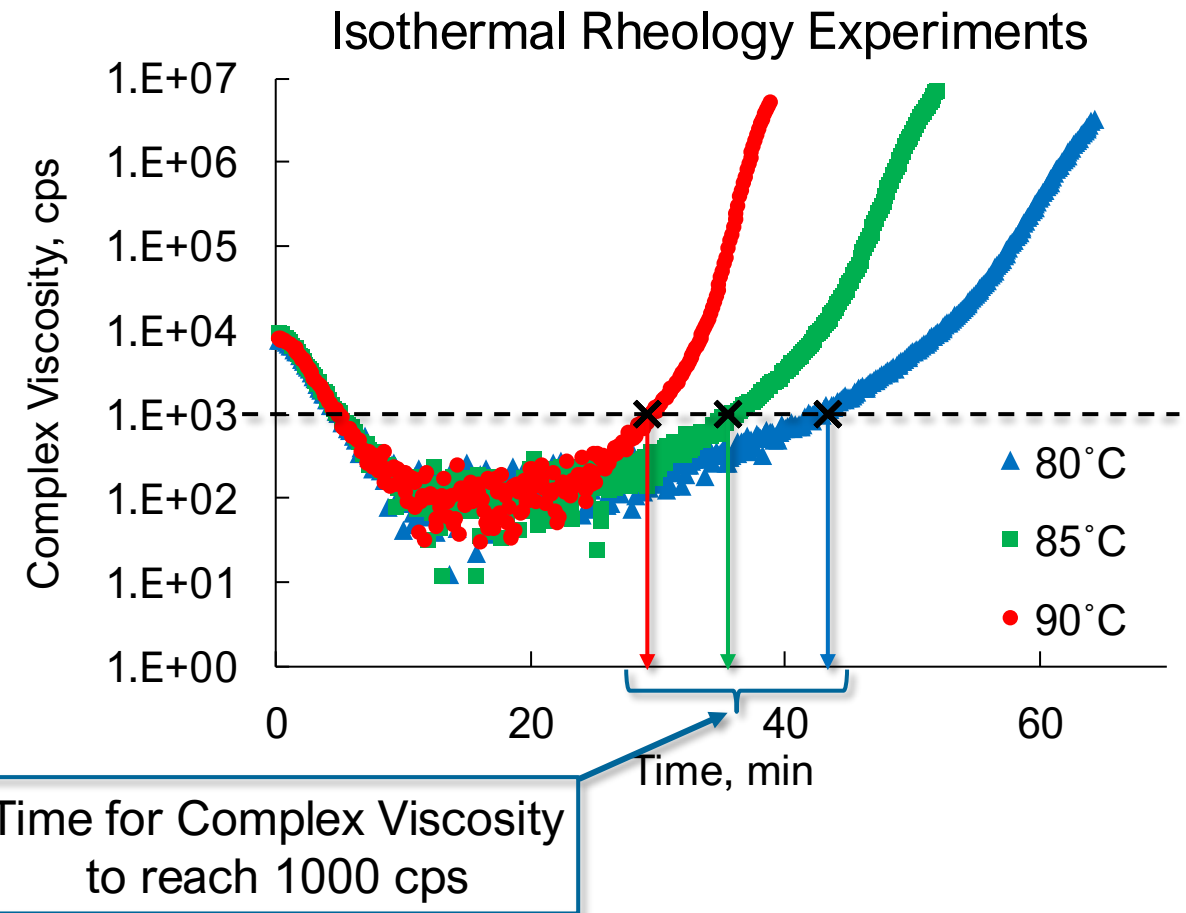


Isothermal Processing Temperature

1. Conduct isothermal rheology experiments at three temperatures – 80°C, 85°C, and 90°C
2. **Determine the infusion window for each temperature**

Infusion Window

= (Time for Complex Viscosity to reach 1000 cps)
– (Time to Isothermal Temperature)



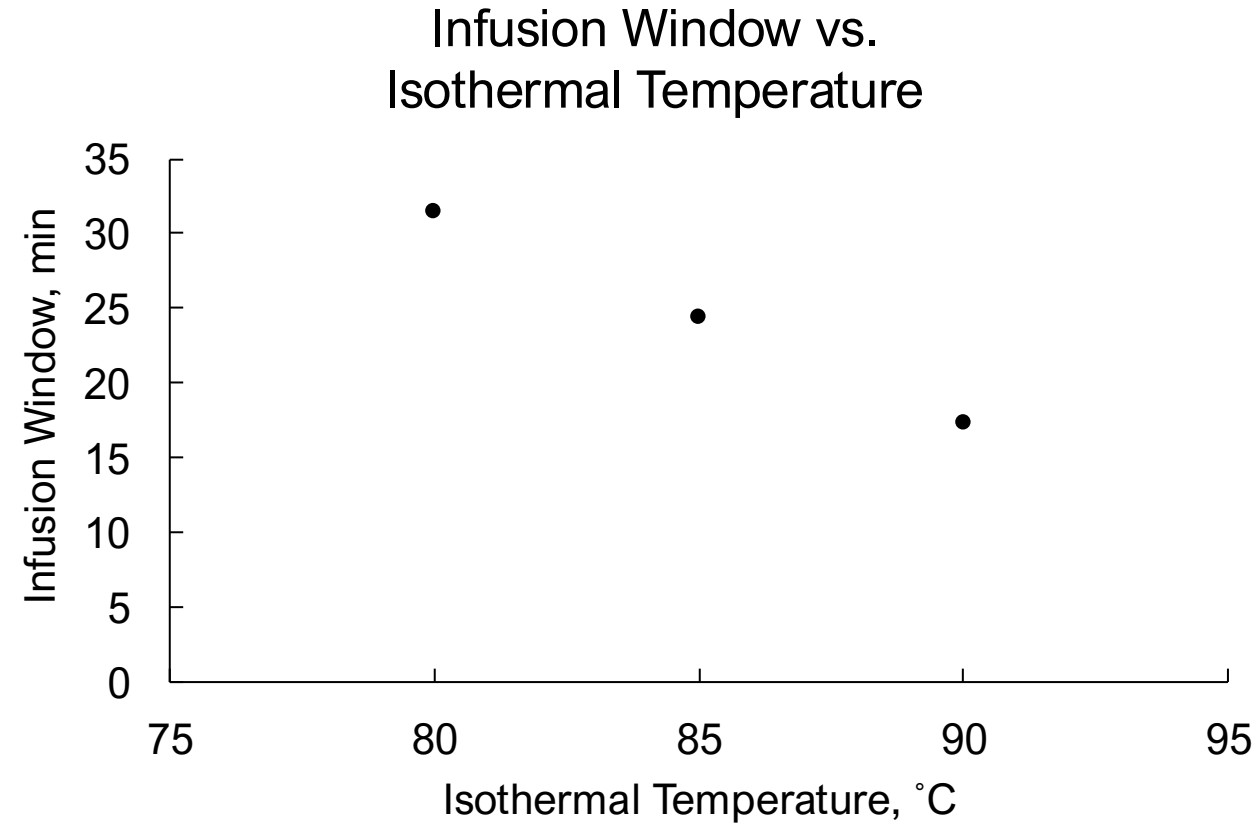


Isothermal Processing Temperature

1. Conduct isothermal rheology experiments at three temperatures – 80°C, 85°C, and 90°C
2. Determine the infusion window for each temperature

$$\begin{aligned} & \text{Infusion Window} \\ &= (\text{Time for Complex Viscosity to reach } 1000 \text{ cps}) \\ &\quad - (\text{Time to Isothermal Temperature}) \end{aligned}$$

3. Plot infusion window vs. isothermal temperature





Isothermal Processing Temperature

1. Conduct isothermal rheology experiments at three temperatures
– 80°C, 85°C, and 90°C
2. Determine the infusion window for each temperature

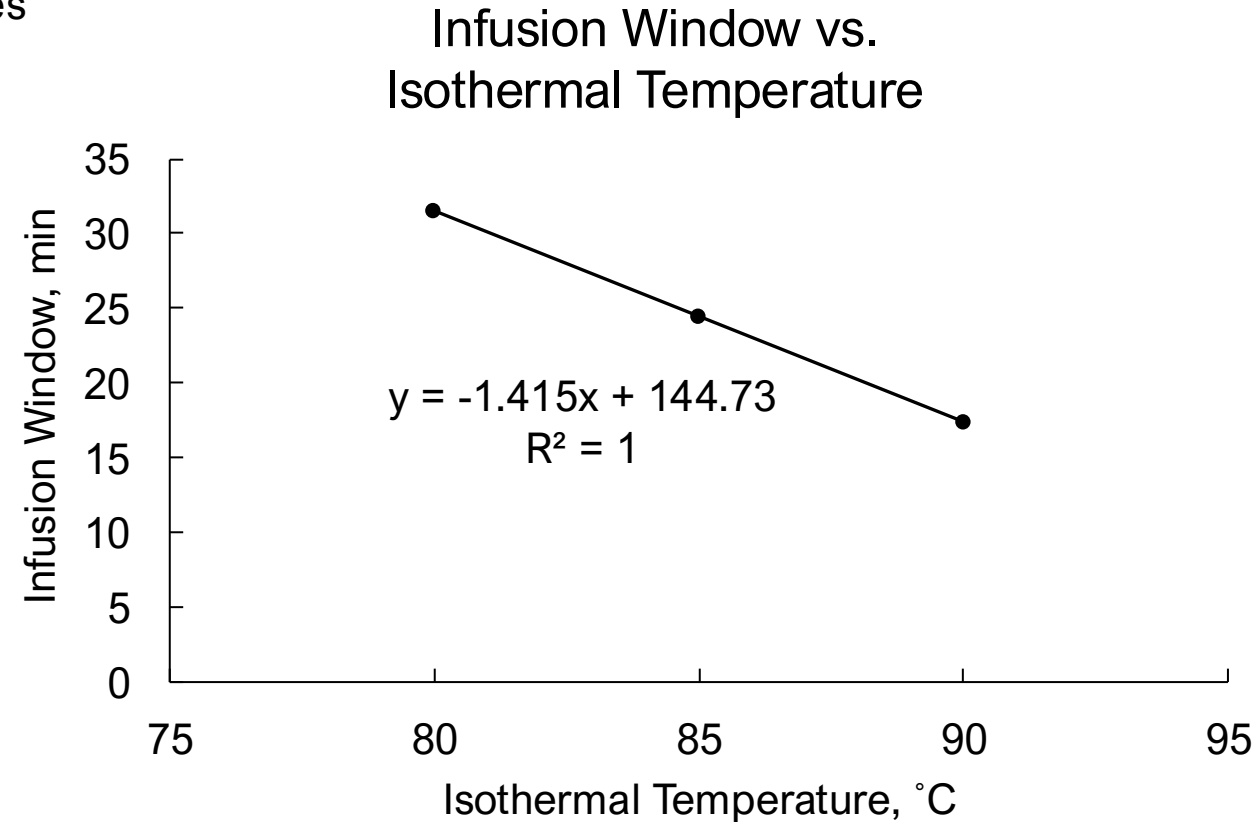
$$\begin{aligned} \text{Infusion Window} \\ = & (\text{Time for Complex Viscosity to reach } 1000 \text{ cps}) \\ & - (\text{Time to Isothermal Temperature}) \end{aligned}$$

3. Plot infusion window vs. isothermal temperature
4. **Use the least-squares method to determine a linear fit line:**

$$y = mx + b$$

$$y = \text{Infusion Window} = 30 \text{ minutes}$$

$$x = \text{Isothermal Processing Temperature}$$





Isothermal Processing Temperature

1. Conduct isothermal rheology experiments at three temperatures – 80°C, 85°C, and 90°C
2. Determine the infusion window for each temperature
3. Plot infusion window vs. isothermal temperature
4. Use the least-squares method to determine a linear fit line:

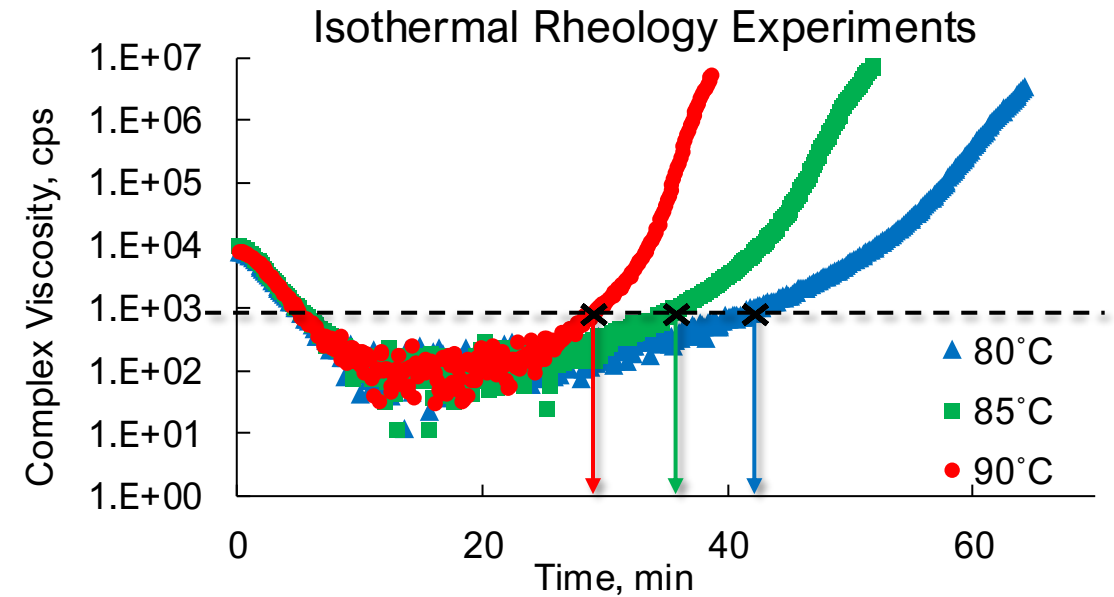
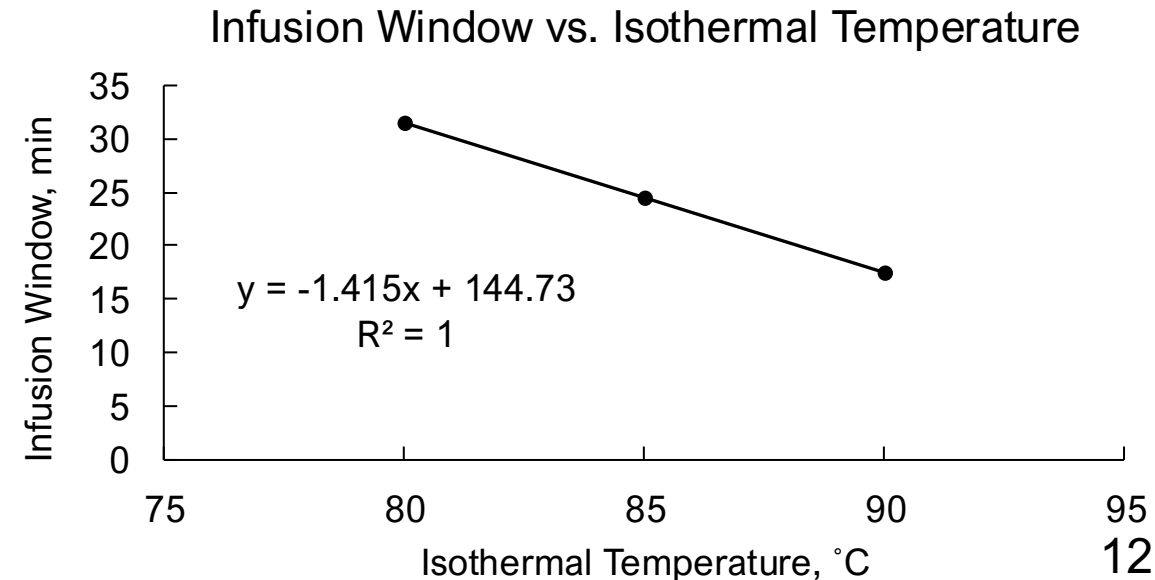
$$y = mx + b$$

y = Infusion Window, 30 minutes

x = Isothermal Processing Temperature

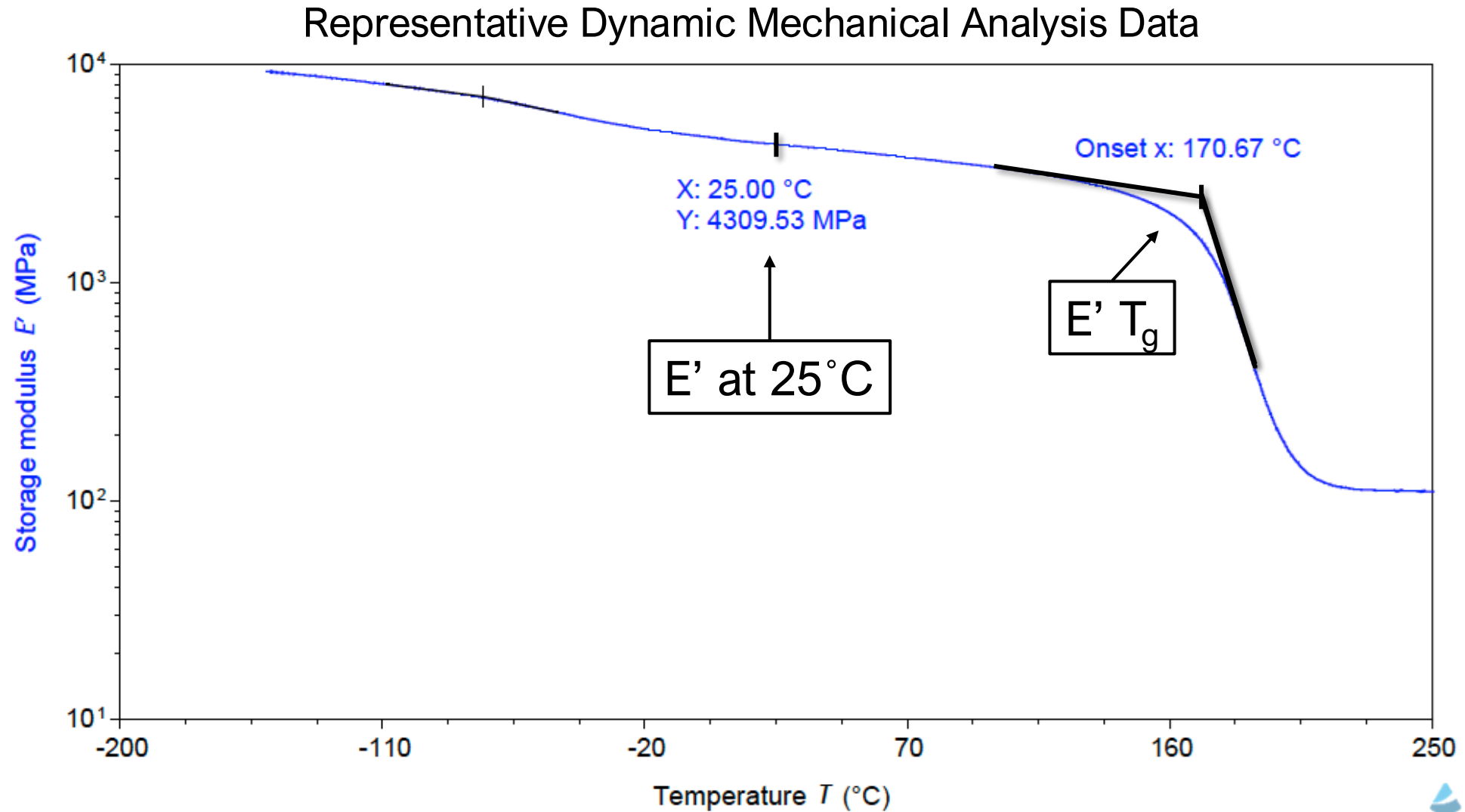
5. Calculate the isothermal processing temperature by solving for x when $y = 30$ minutes

$$x = \frac{y - b}{m}$$



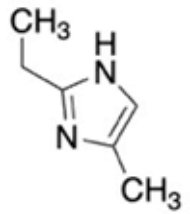


Thermomechanical Properties





Effect of Catalyst Concentration



2Et4MeIm

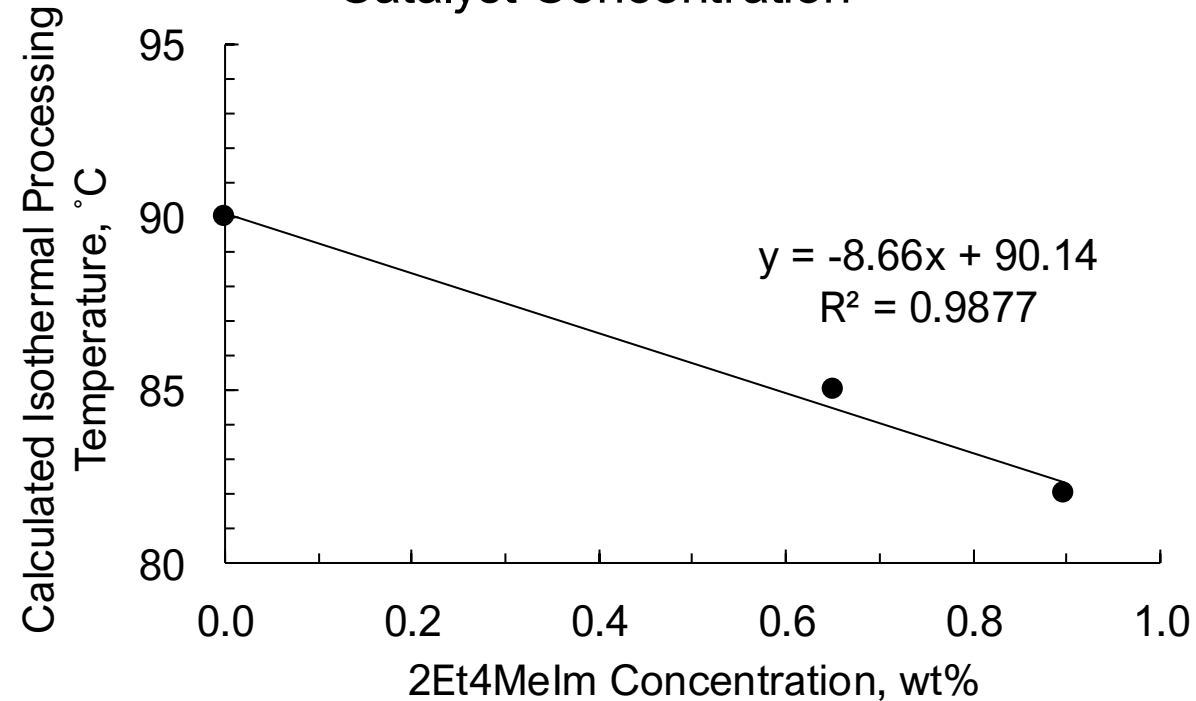
Core Resin Composition

- Epoxy: TGMDA, DGOA
- Hardener: 4,4'-DDS, mPDA

2Et4MeIm Loading: 0 – 0.90 wt%

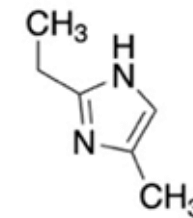
Increasing catalyst loading
decreases the calculated
isothermal processing
temperature

Calculated Cure Temperature vs.
Catalyst Concentration





Effect of Catalyst Concentration



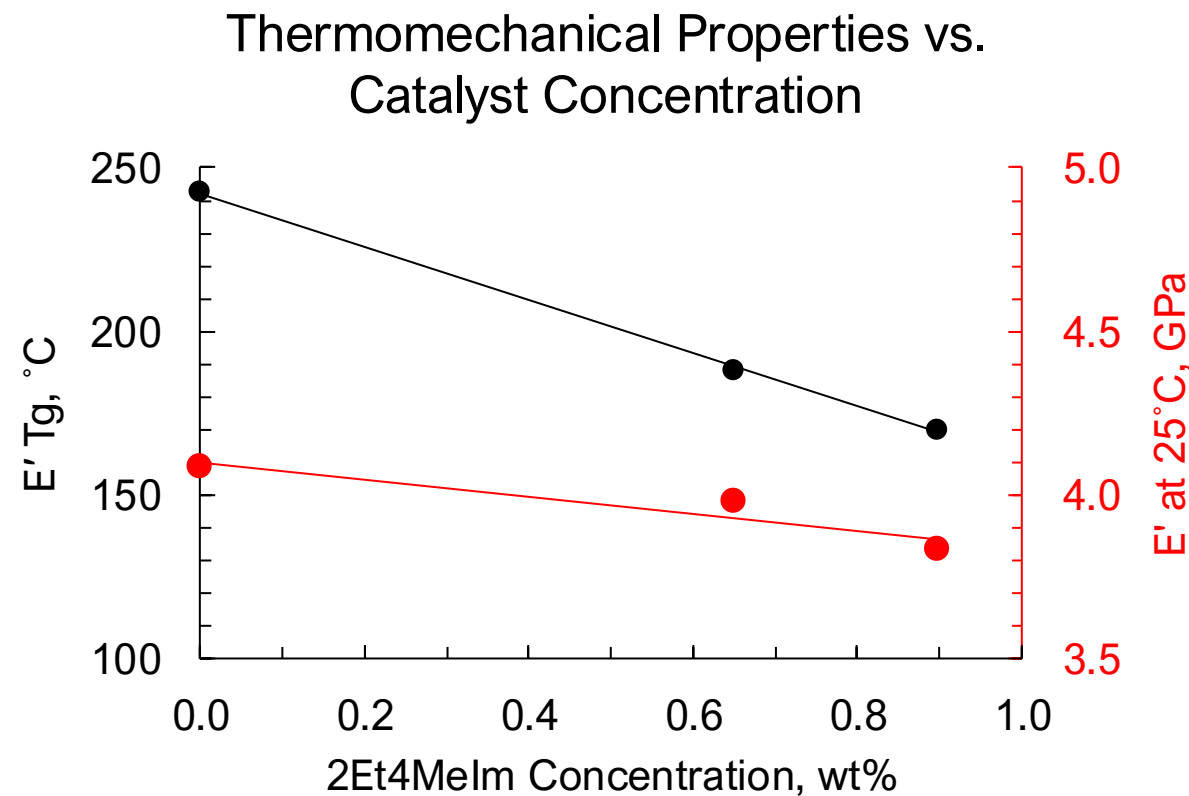
2Et4MeIm

Core Resin Composition

- Epoxy: TGMDA, DGOA
- Hardener: 4,4'-DDS, mPDA

2Et4MeIm Loading: 0 – 0.90 wt%

- Significant effect on E' T_g
 - 0.9 wt%: 30% reduction
- Insignificant effect on E' at 25°C
 - 0.9 wt%: 6% reduction



Increasing catalyst loading reduces the thermomechanical properties

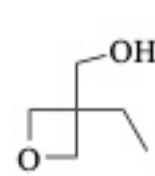


Effect of Epoxy Additives

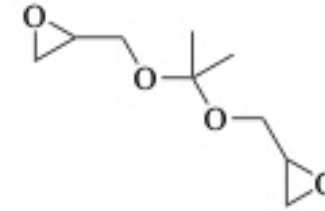
Core Resin Composition

- Epoxy: TGMDA, DGOA
- Hardener: 4,4'-DDS, mPDA
- Catalyst: 2Et4Melm, 1.0 wt%
- Additive Loading: ≤ 5 wt%

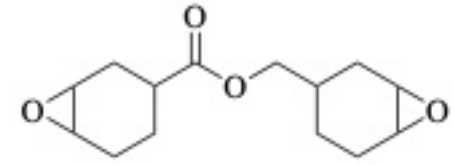
Aliphatic epoxy additives
reduce isothermal processing
temperatures



EOM

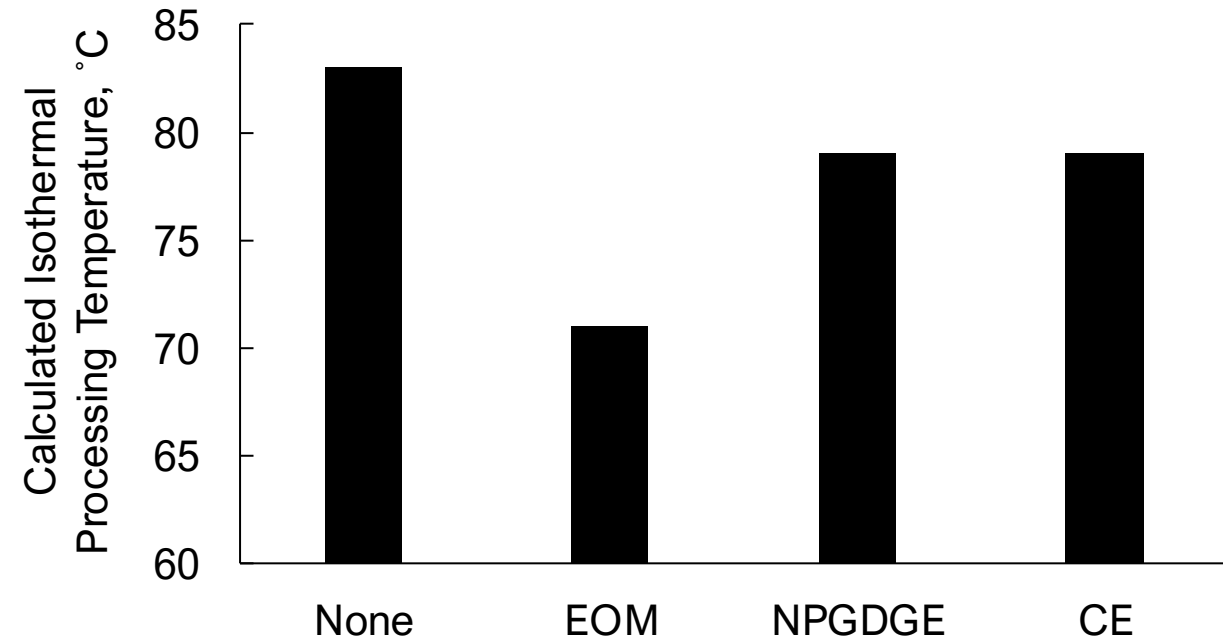


NPGDGE



CE

Effect of Epoxy Additives





Effect of Epoxy Additives

Core Resin Composition

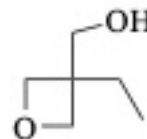
- Epoxy: TGMDA, DGOA
- Hardener: 4,4'-DDS, mPDA
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Storage Modulus

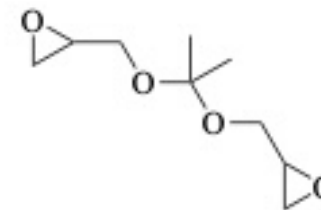
- Increased for all additives by $<10\%$

Glass Transition Temperature

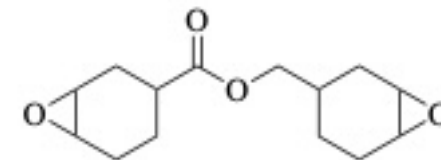
- Decreased for NPGDGE and EOM: 10 – 30%
- CE: Insignificant effect



EOM

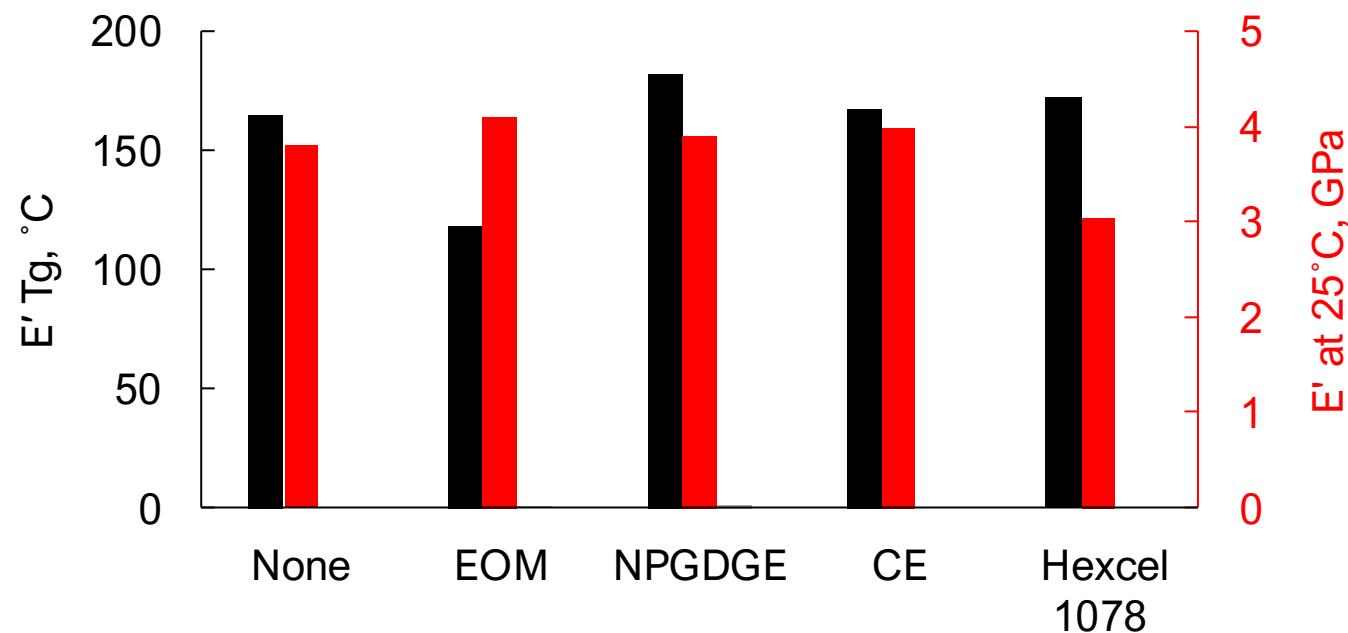


NPGDGE



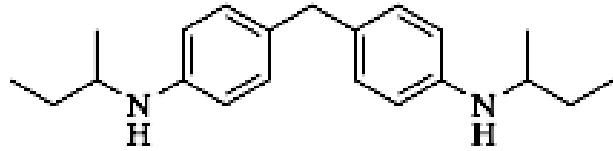
CE

Effect of Epoxy Additives

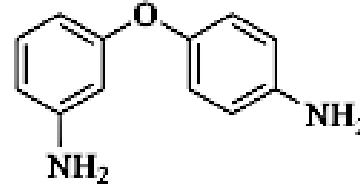




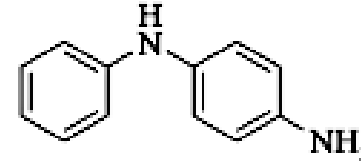
Effect of Hardener Additives



E-420



3,4'-ODA



PPD



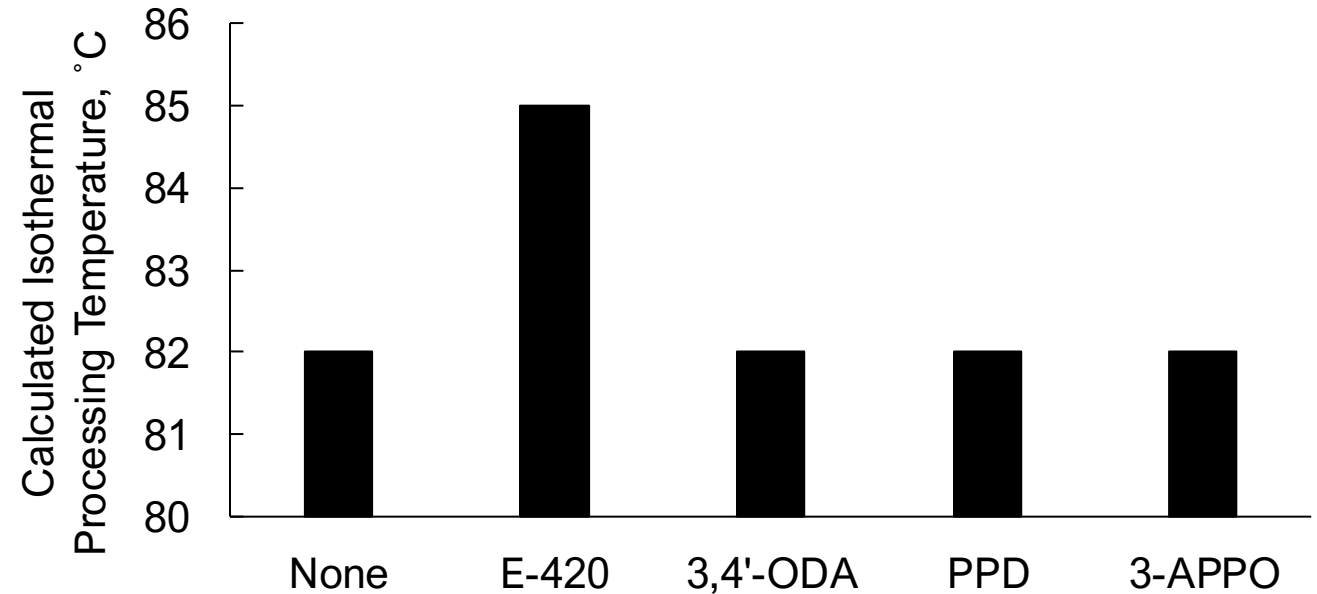
3-APPO

Core Resin Composition

- Epoxy: TGMDA, DGOA
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- Catalyst: 2Et4Melm, 1.0 wt%
- Additive Loading: ≤ 5 wt%

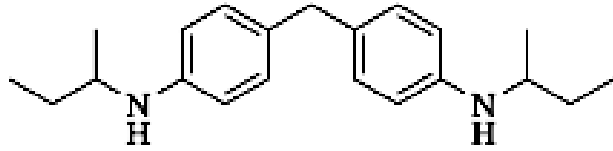
Hindered diamines increase the isothermal processing temperature

Effect of Hardener Additives

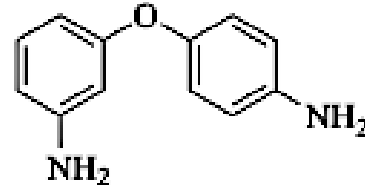




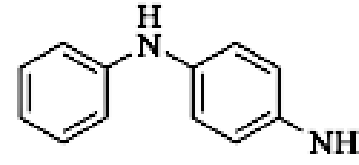
Effect of Hardener Additives



E-420



3,4'-ODA



PPD



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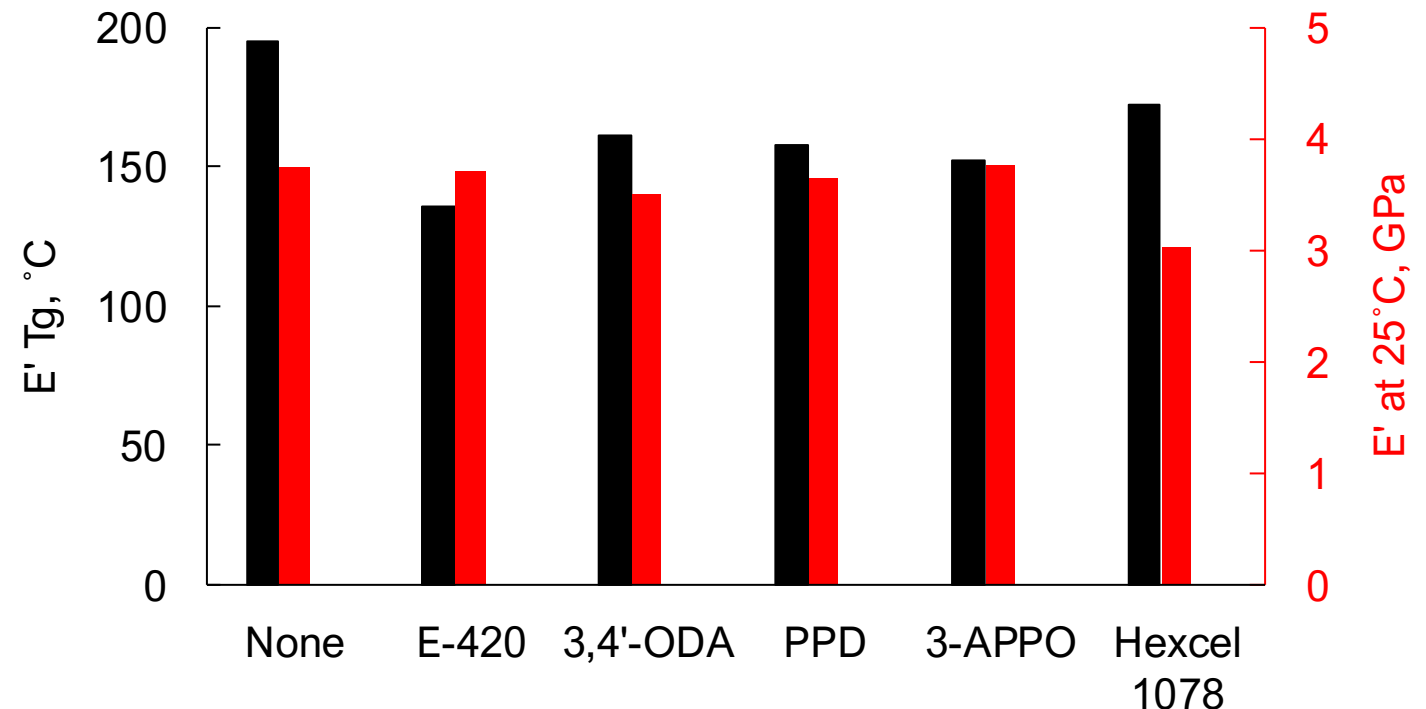
Storage Modulus

- Insignificant impact

Glass Transition Temperature

- Reduced by 17 – 30%

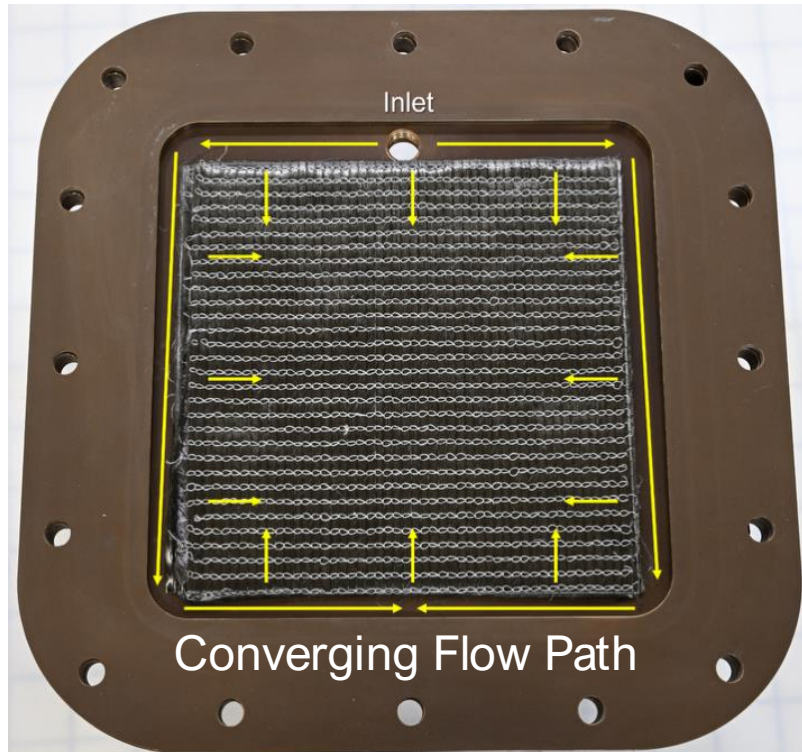
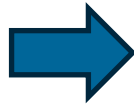
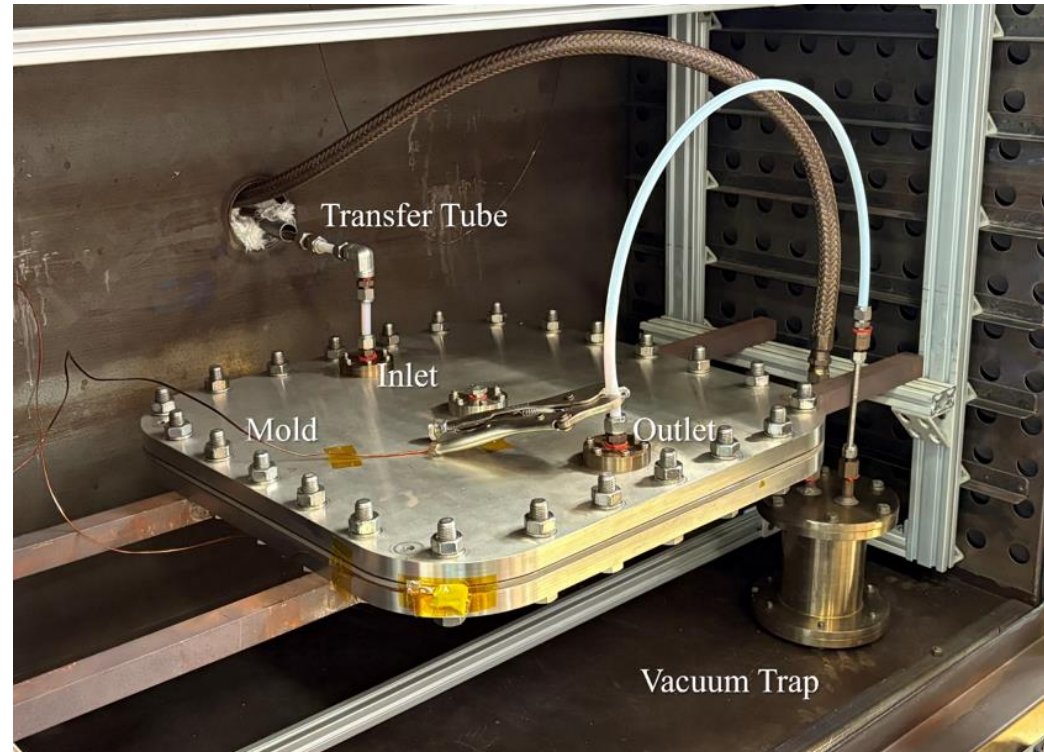
Effect of Hardener Additives





Composite Fabrication

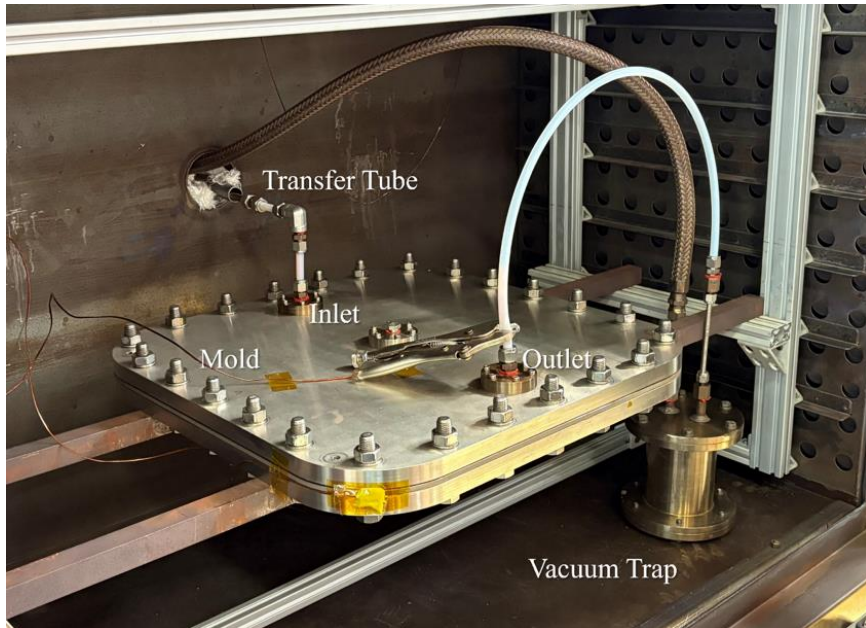
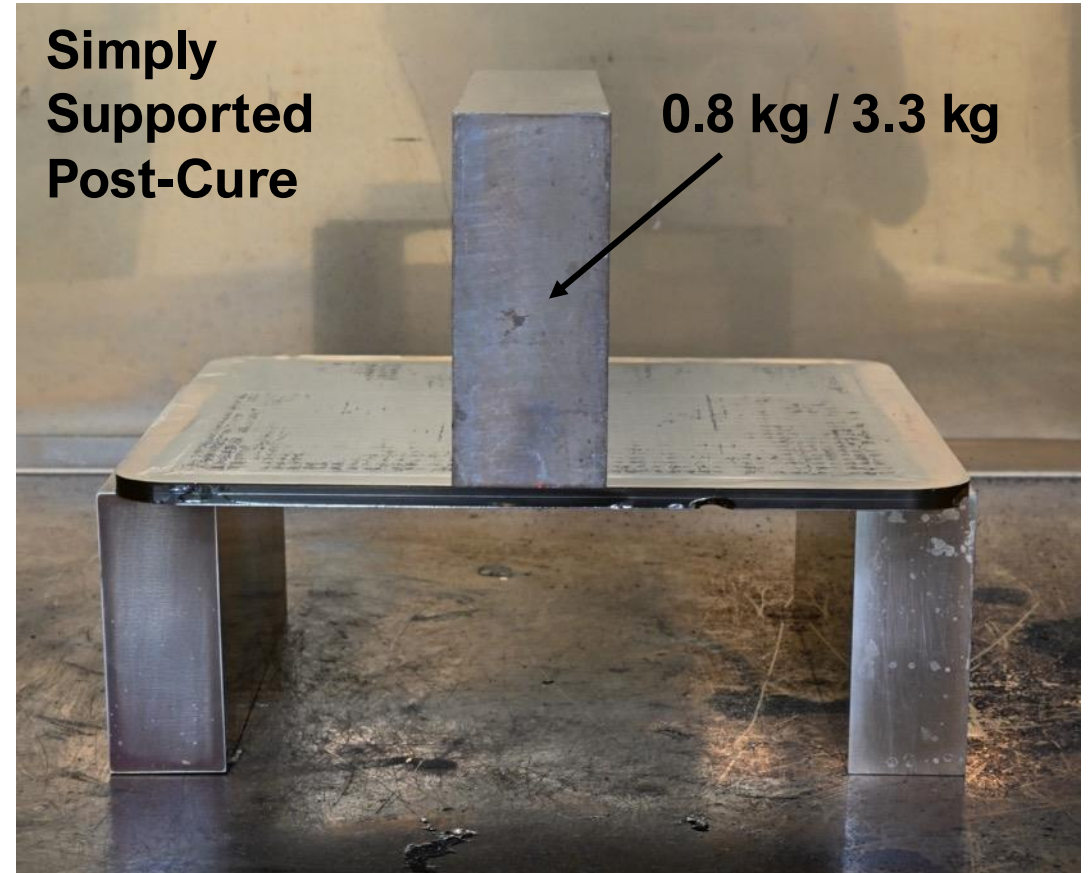
- Reinforcement: Saertex 568 g/m², non-crimp, quasi-isotropic, symmetric layup
- Thickness:
 - Tension Test Samples: 4-Ply
 - Compression and Fracture Toughness Test Samples: 8-Ply
- Target Fiber Volume Fraction (FVF): 58.5%





Composite Fabrication

- Reinforcement: Saertex 568 g/m², non-crimp, quasi-isotropic, symmetric layup
- Thickness:
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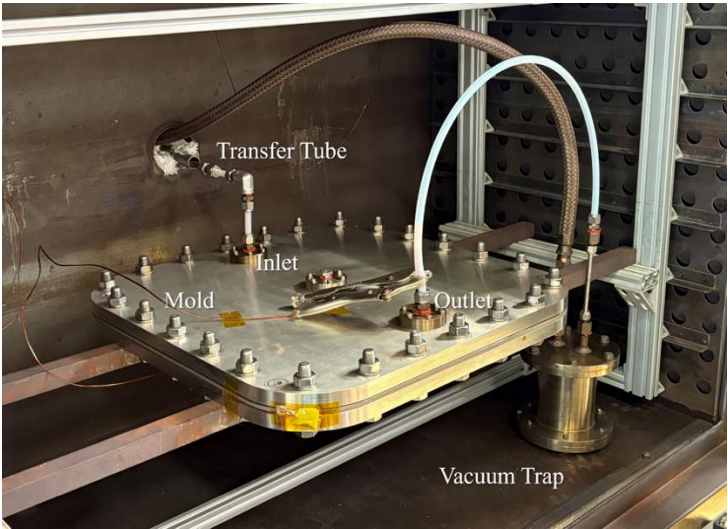
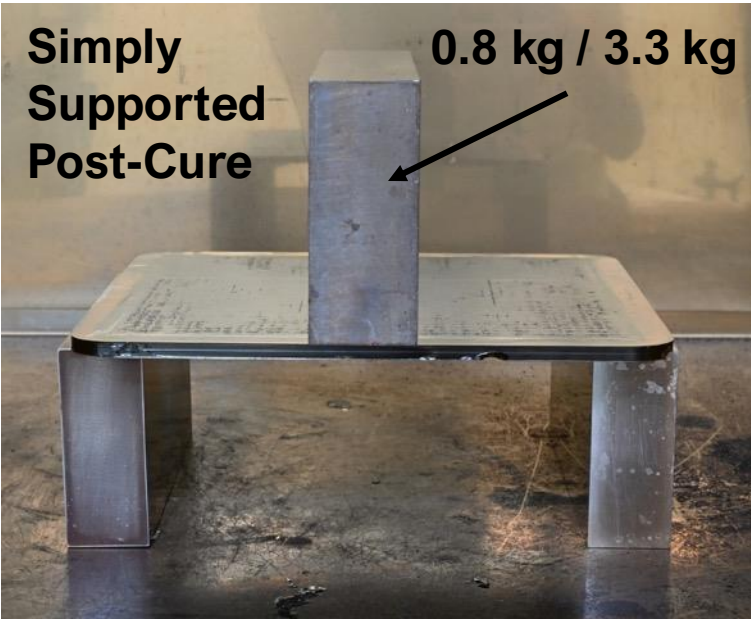
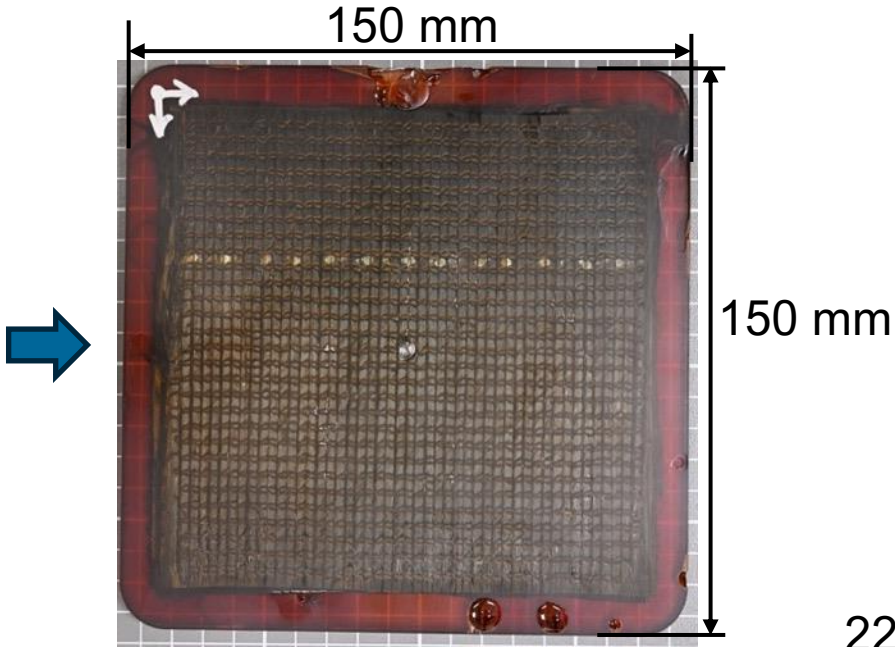




Composite Fabrication

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- Thickness:
 - Tension: 4-Ply
 - Compression and Fracture Toughness: 8-Ply
- Target FVF: 58.5%

Property	Percentage of Hexcel HexFlow® 1078 Value
Tensile Strength	90%
Tensile Modulus	98%
Compressive Strength	113%
G _{IIC}	62%





Concluding Remarks

- An isothermal rapid-curing resin was formulated with an isothermal processing temperature $< 90^{\circ}\text{C}$ to achieve infusion and cure in 1 hour
- The isothermal rapid-curing resin was used to fabricate a composite panel via RTM with properties comparable to Hexcel HexFlow[®] 1078
- Increasing catalyst loading results in decreasing isothermal processing temperatures and diminishing mechanical properties
- The aliphatic epoxy additives investigated increase reaction rate and have structure dependent impacts on thermomechanical properties
- The amine additives investigated have negligible impact on isothermal processing temperature (with the exception of hindered E-420) and reduce thermomechanical properties