Polyimide Composites Properties of RTM370 Fabricated by VARTM

Kathy C. Chuang
NASA Glenn Research Center, Cleveland, OH 44135
(216) 433-3227; Kathy.Chuang@grc.nasa.gov

Jim M. Criss
M & P Technologies, Inc. Marietta, GA 30314
(770) 652-7638; jim.m.criss@mandptechnologies.com

Eric A. Mintz
Center for High Performance Polymers and Composites,
Clark Atlanta University, Atlanta, GA 30314

High Temperature Polyimide Composites Materials & Processing

- **Conventional PMR-15, PMR-II-50, AFR-PE4 polyimide composites all require solvent-based prepregs for part fabrication ⇒ *time consuming, costly and the use of solvents and diamines are hazardous***

- **Fabricate net-shape polymer matrix composites from resins in the melt via RTM or VARTM using preforms ⇒ eliminate costly hand lay-up and hazard**
  ⇒ *produce 30% cost saving & 20% weight saving for complex parts*
  ⇒ adaptable to automatic process

Challenge: RTM or VARTM requires low-melt viscosity that conventional polyimides derived from commercially available s-dianhydride (> 10^3-10^5 poise) cannot meet

- **a-BPDA based imide resins have shown to exhibit low-melt viscosity (10-30 poise) at 280 °C ⇒ *amenable to low-cost RTM or VARTM process***

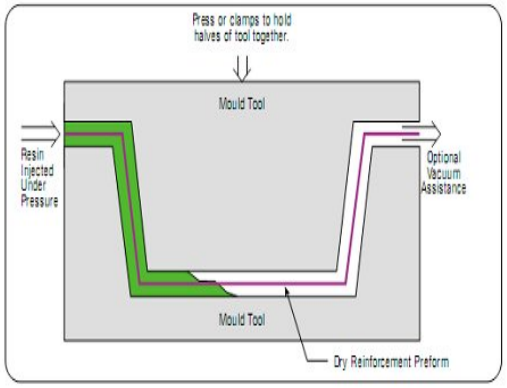
- **Advance PMC temperature capability to 260-315°C beyond the state-of the-art RTM resins, such as epoxy (177 °C) & BMI (232 °C)**

Objectives

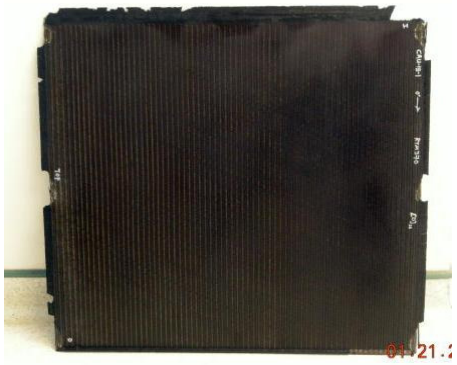
- Fabricated composite panels with RTM370 imide resin (~10-30 poise) by vacuum assisted resin transfer molding (VARTM)
- Compare mechanical properties of VARTM panels to RTM panels at 288-315°C (550-600°F)

VARTM *vs* RTM

⇒

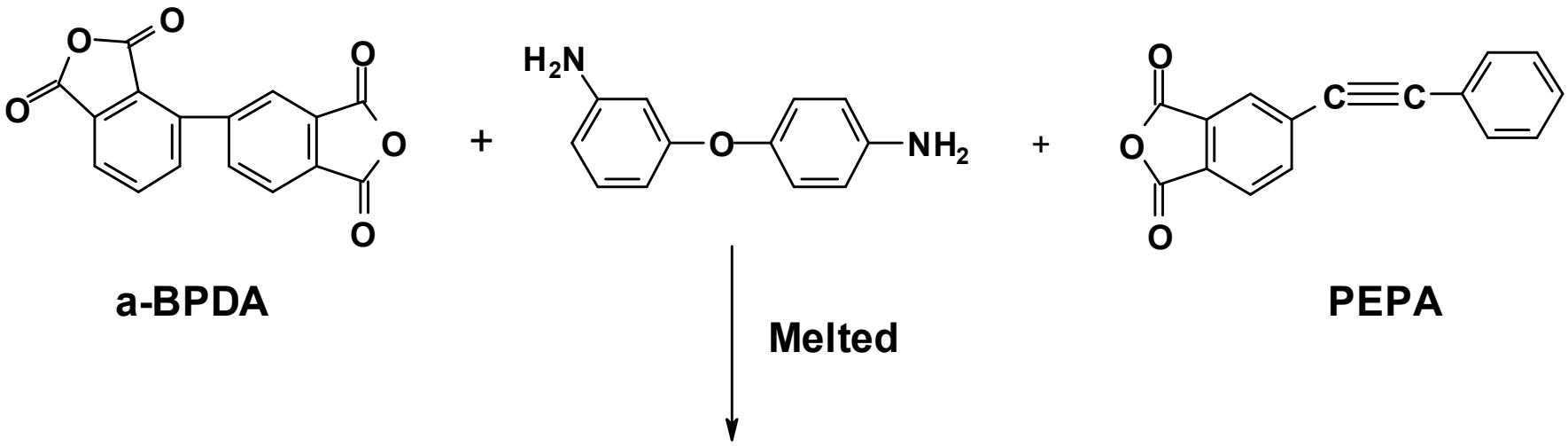


⇒



VARTM	RTM
Use vacuum bag	Need a mold (expensive)
Use vacuum only	Use pressure and/or vacuum
15-20 psi	200 psi
Suitable for large part	Suitable for small part/mold

RTM370 Imide Resins



Imidized Oligomers

Advantages of imide resins containing a-dianhydrides:

- ***Lower melt viscosities***
- ***Higher T_g's***

Solvent-Free process:

- ***No organic volatiles*** **(Green)**
- ***Adaptable to reactive extrusion*** ***(cost saving)***

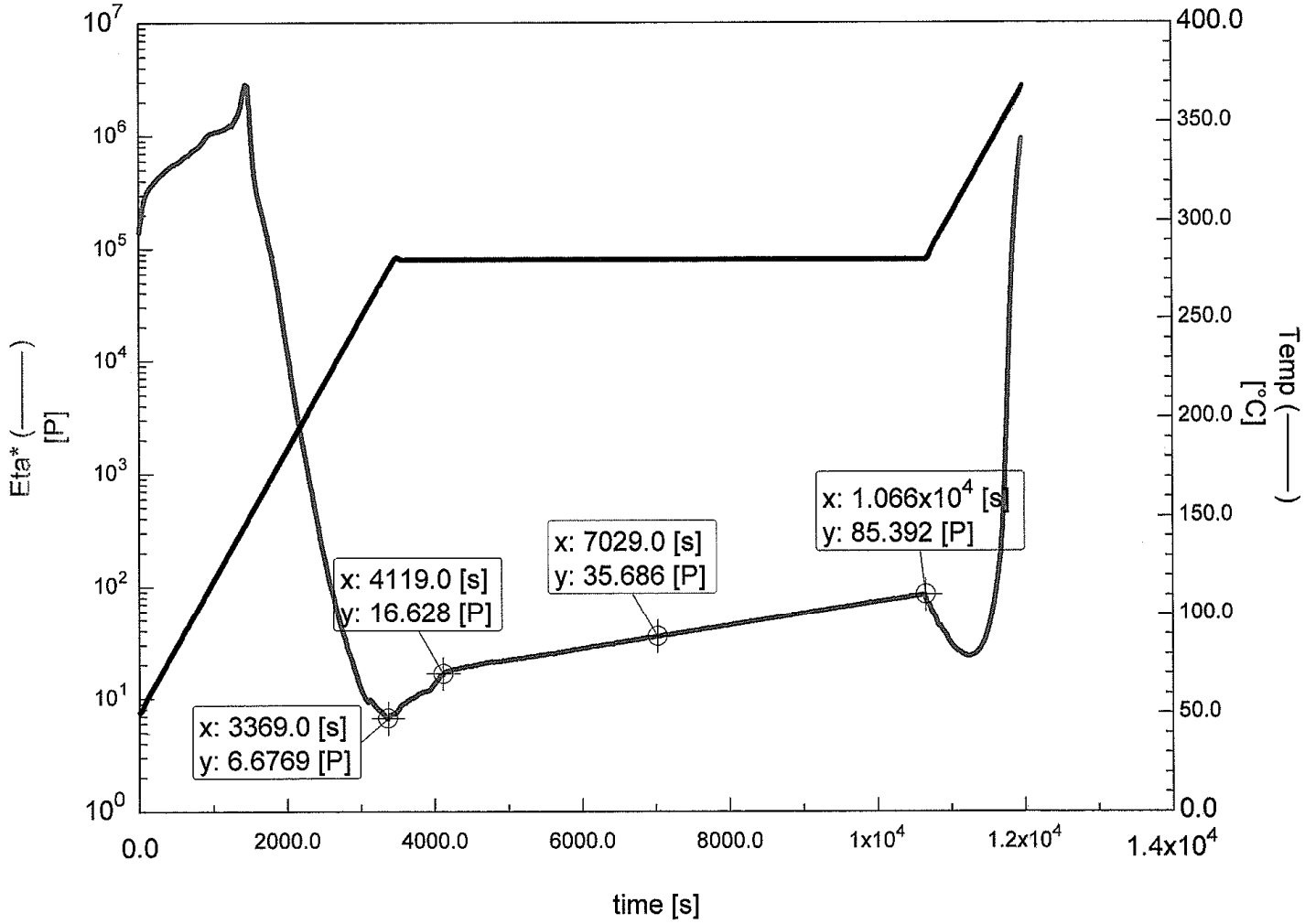
Physical Properties of Imide Oligomers/ Resins Based on a-BPDA and 4-PEPA

Resin	Diamine	Oligomer Min. η @280 °C by Brookfield[1] (Poise)	Oligomer Min. Complex [η]* @280°C[2] (Poise)	Cured Resin T_g (°C) NPC[3] byTMA[5]	Cured Resin T_g (°C) PC[4]@ 650°F
RTM370	3,4′-ODA	8.8	6.5	342	370[5]
RTM370 Composite		---	---	338 (DMA)[6]	350 (DMA)[6]

3,4′-ODA = 3,4′-Oxydianiline

[1] Absolute viscosity measured by Brookfield Viscometer at 280 °C.
[2] Complex viscosity measured by Aries Rheometer, using parallel plates.
[3] NPC = No Post cure
[4] PC = Resin Postcured at 343 °C (650 °F) for 16 hrs while composite postcured@650°
[5] TMA =Thermal mechanical analysis heated at 10 °C/min, using expansion mode.
[6] DMA = Dynamic mechanical analysis were performed at 5 °C/min heating rate, using single cantilever.

Rheology of APS's RTM370 Imide Resin
2 hr hold at 280 °C

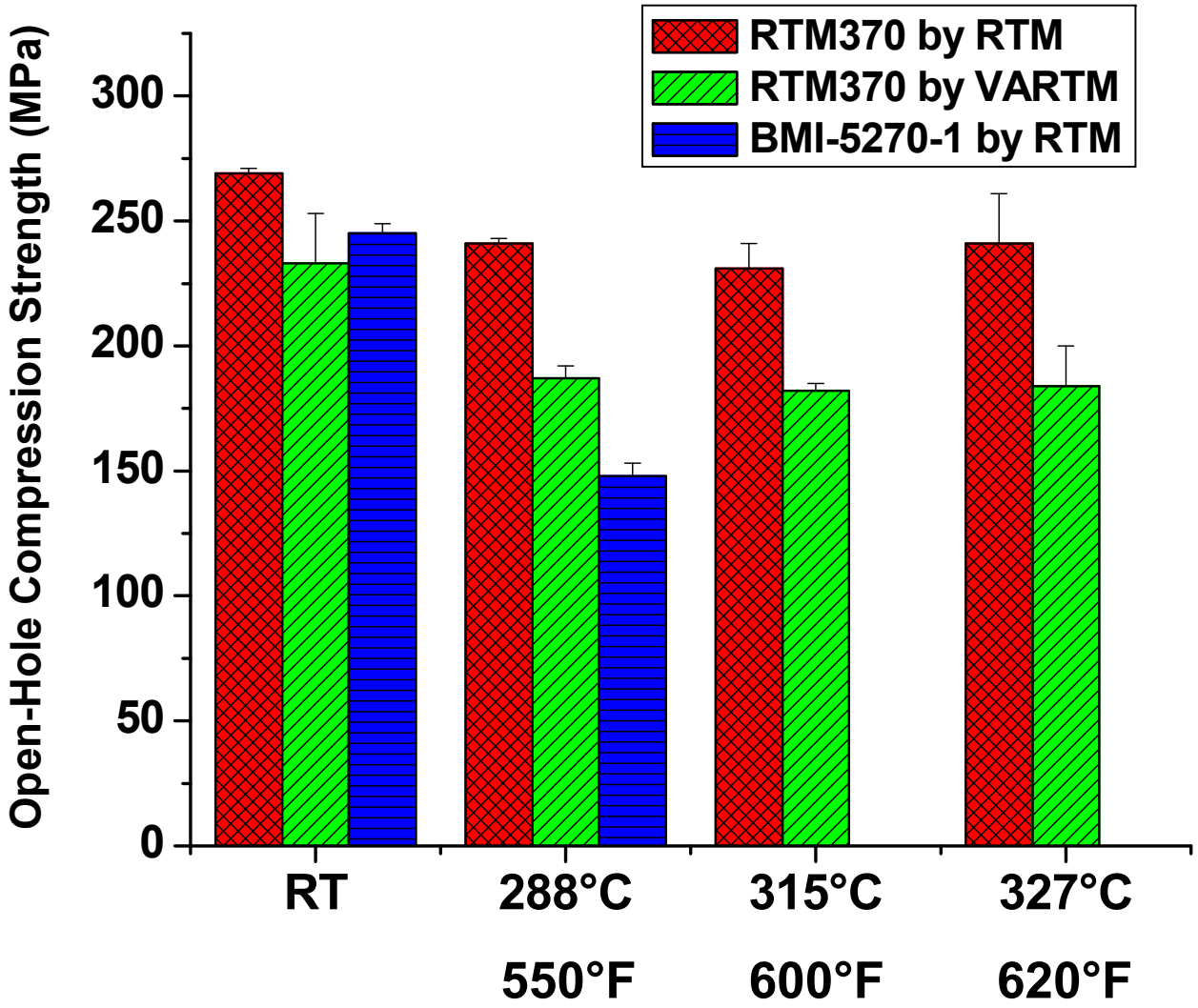


RTM370 Composite Property Comparison VARTM *vs* RTM (T650-35/HT sizing)

	Test Temp.	OHC Strength (MPa)			OHC Modulus (GPa)			SBS Strength (MPa)		
		Initial	500h @550°F 288°C	1000h @550°F 288°C	Initial	500h @550°F 288°C	1000h @550°F 288°C	Initial	500h @550°F 288°C	1000h @550°F 288°C
VARTM	RT	233	194	120	37	38	33	43	37	22
	288°C	186	197	135	40	42	38	31	27	17
	315°C	182	---	118	42	---	38	29	26	15
	327°C	184	---	---	41	---	---	30	---	16
RTM	RT	269	287	230	44	47	44	51	54	43
	288°C	242	244	198	48	44	45	41	41	41
	315°C	231	---	---	46	---	---	31	---	---
	327°C	241	---	---	48	---	---	30	---	---

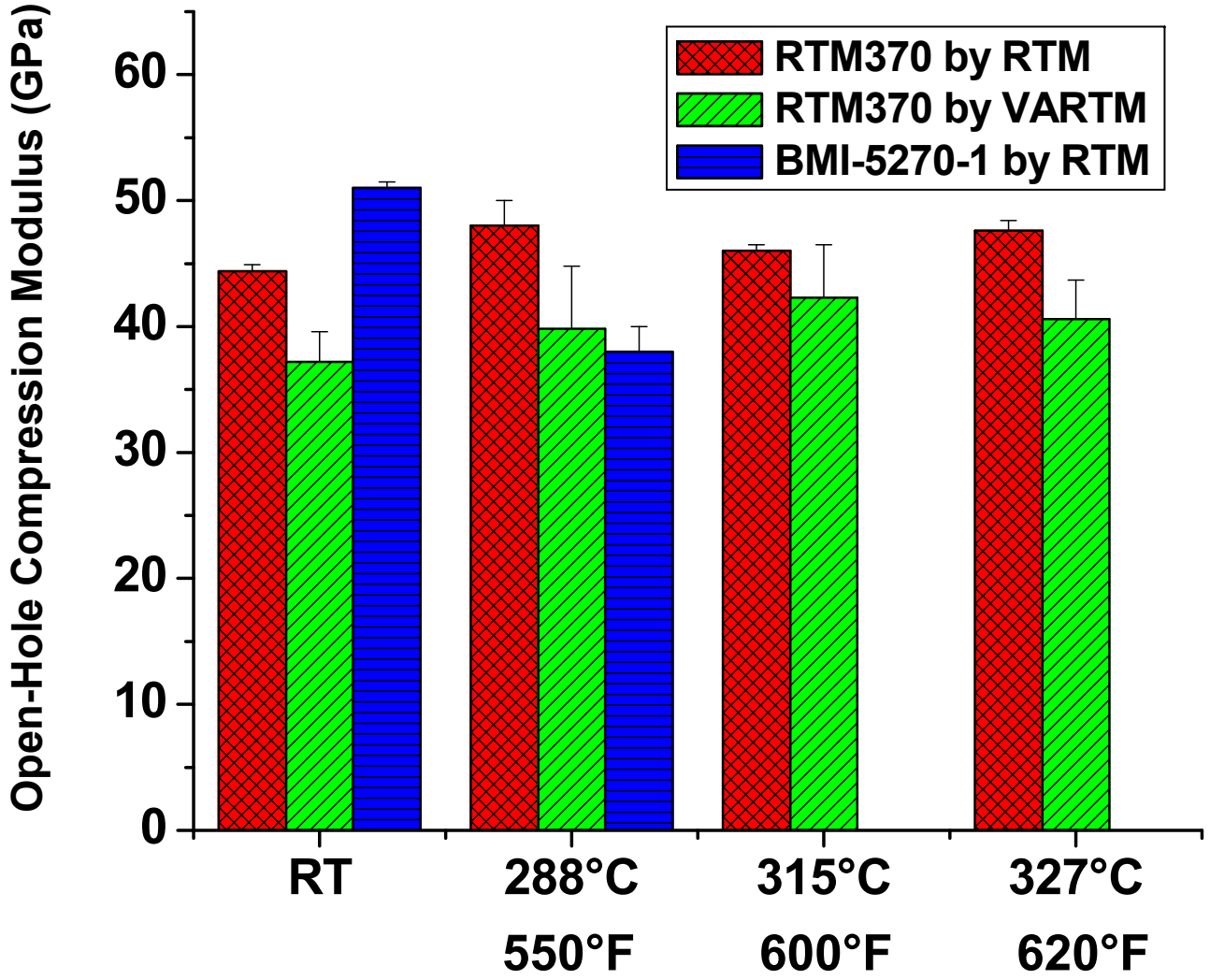
RTM370 OHC Strength VARTM *vs* RTM

(T650-35/8HS/HT Sizing)



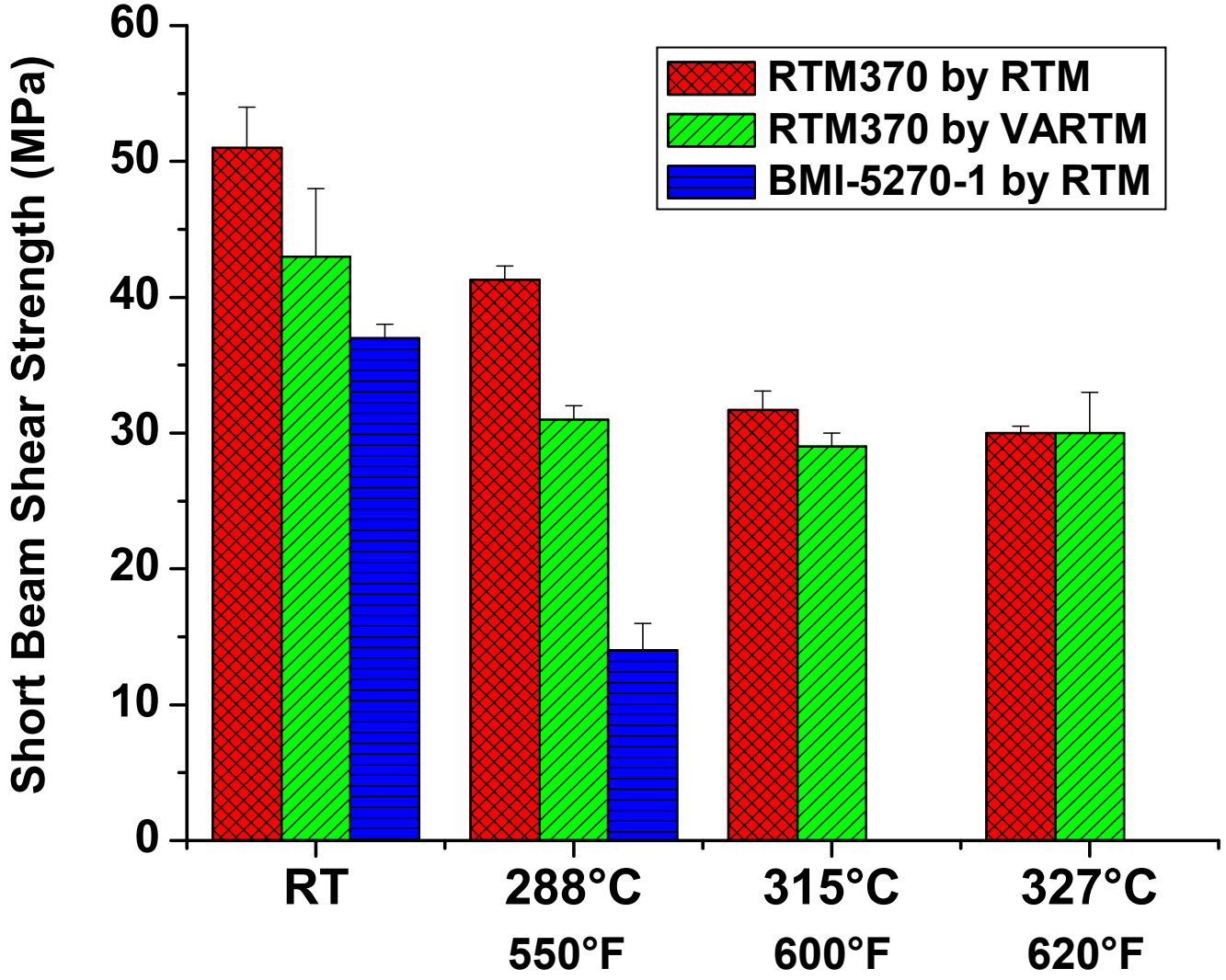
RTM370 OHC Modulus VARTM *vs* RTM

(T650-35/8HS/HT Sizing)

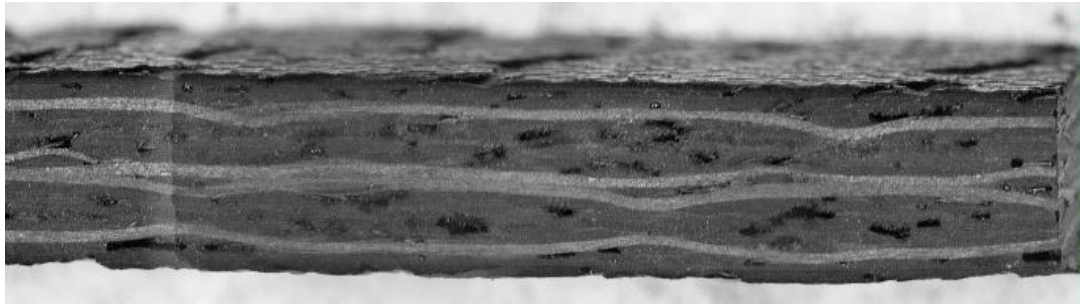


RTM370 SBS Strength VARTM *vs* RTM

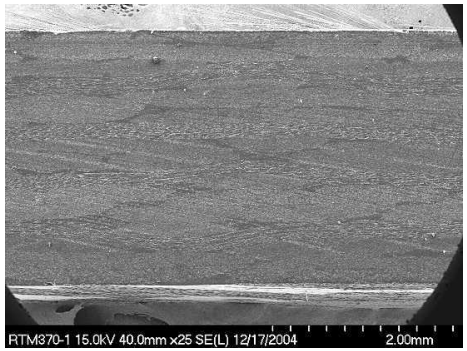
(T650-35/8HS/HT Sizing)



VARTM *vs* RTM



SEM of RTM370 made by VARTM
Void content = ~6.5% after postcured at 650°F/8 h
Resin *vs* fiber content =47-50% *vs* 53-50%



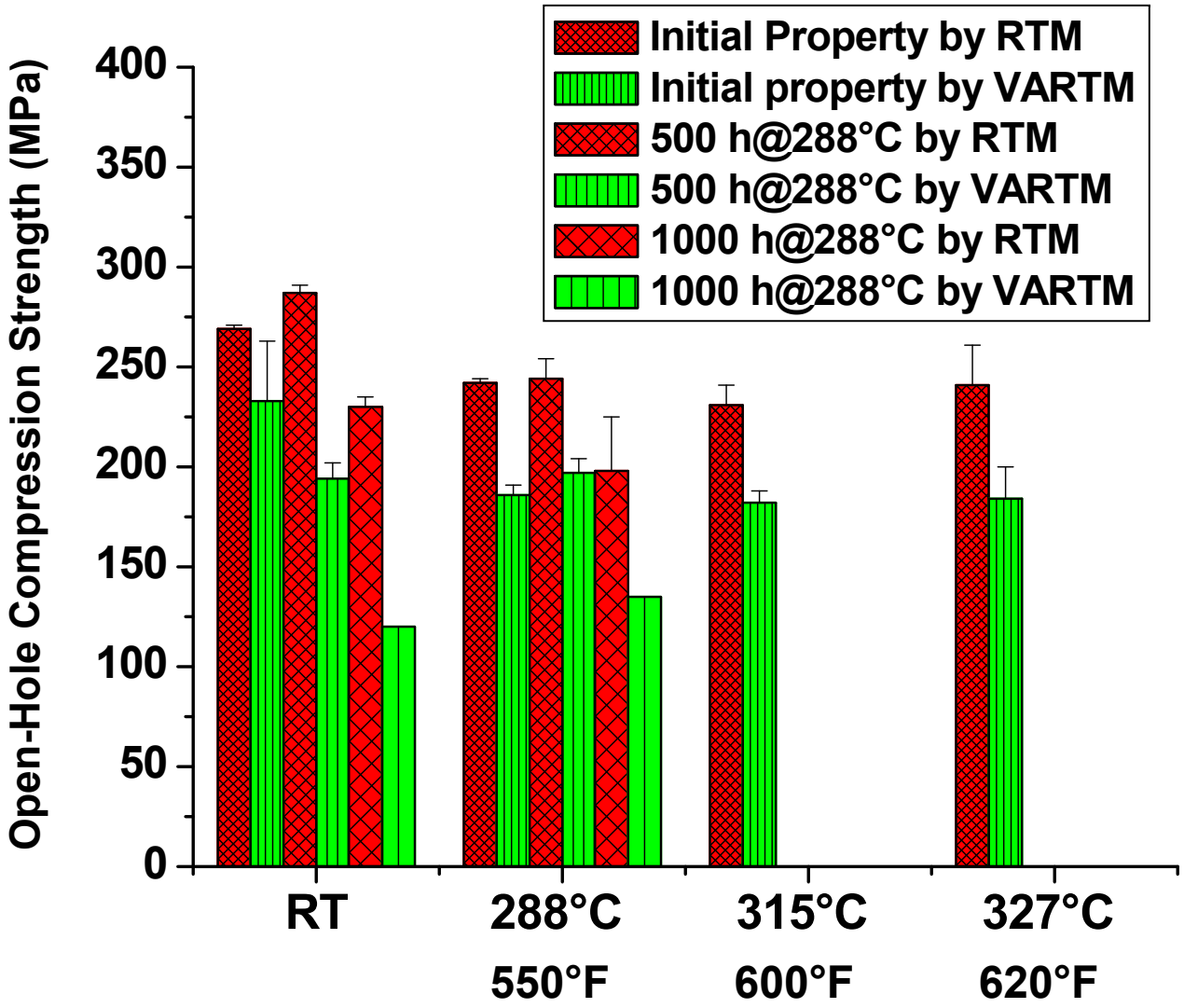
RTM370 Made by RTM
Void content= ~1%
Resin content = 44-47%

VARTM	RTM
15-20 psi	200 psi
Higher void content	Lower void content

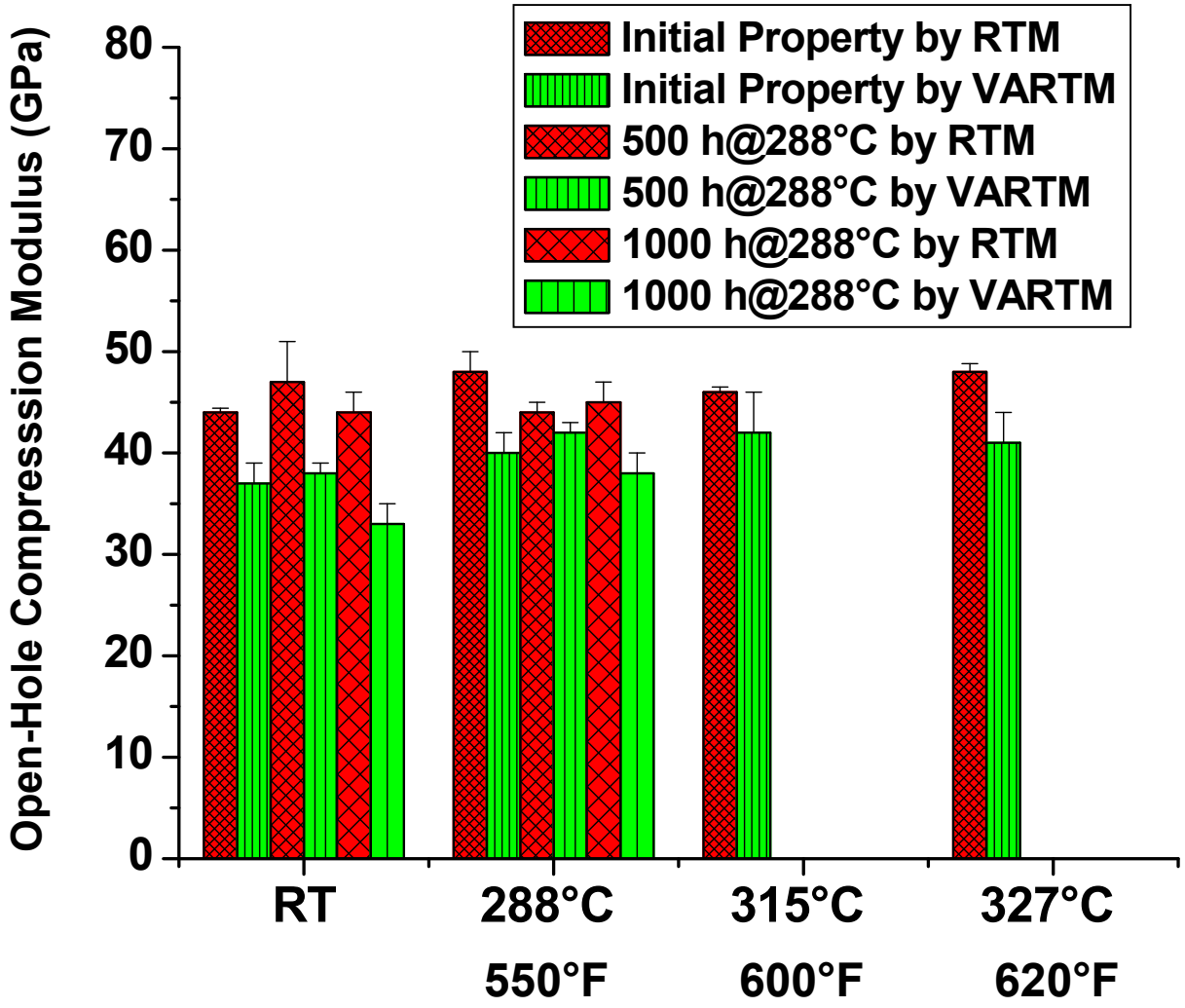
Process Improvement:

- **Injection at 260°C instead of 280°C ⇒ Longer pot-life, but longer injection time**
- **Add hold time at ~300°C, instead of direct ramp from 280°C to 371°C**

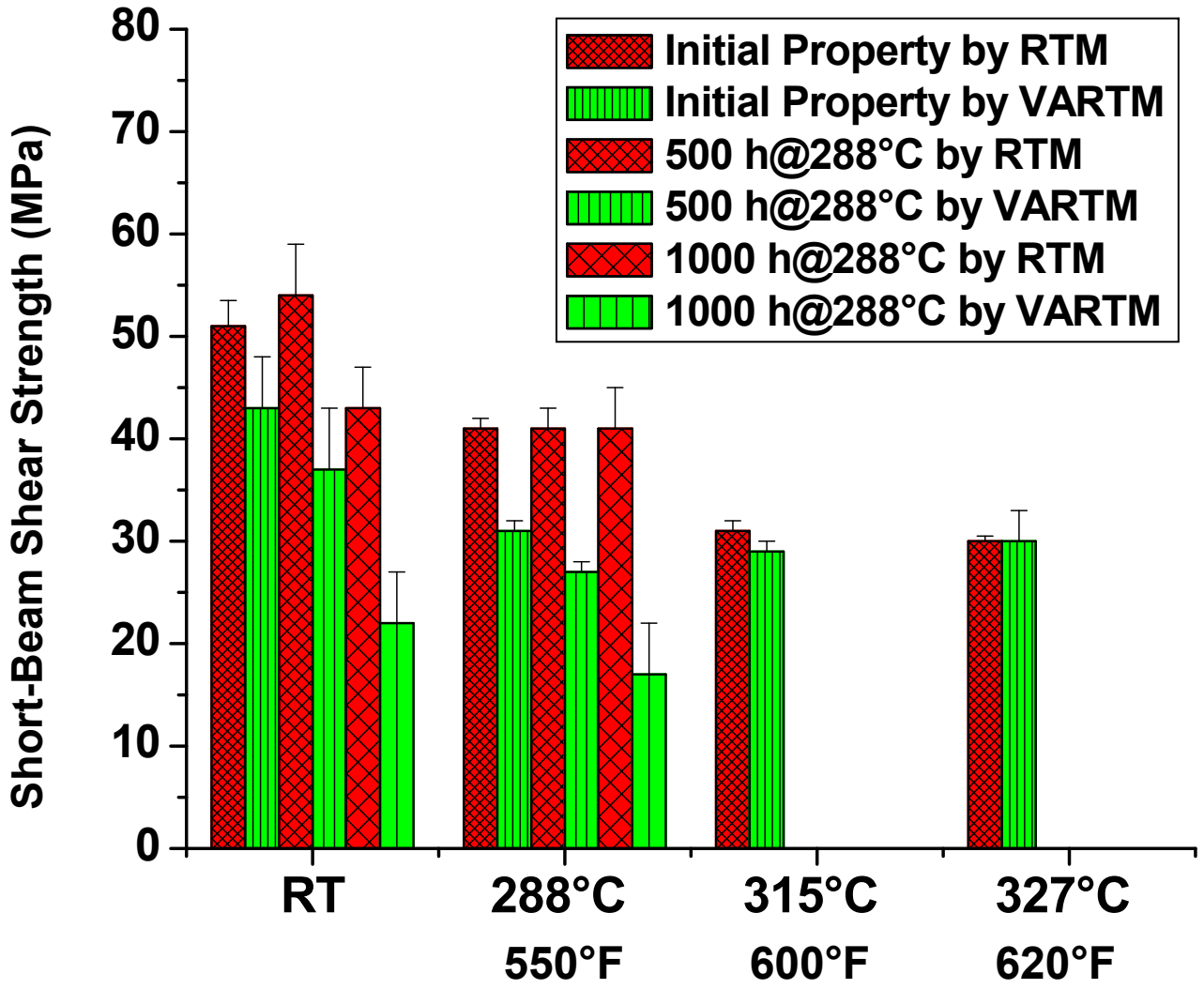
Open-Hole Compression Strength of RTM370 Composites Subjected to Isothermal Aging at 288°C for 1000 h in Air



Open-Hole Compression Modulus of RTM370 Composites Subjected to Isothermal Aging at 288°C for 1000 h in Air



Short-Beam Shear Strength of RTM370 Composites Subjected to Isothermal Aging at 288°C for 1000 h in Air



Summary

- Demonstrated RTM370 imide resin can be processed by VARTM out of autoclave
- VARTM panels have higher void content than those made by RTM, due to lack of pressure

  ⇒ VARTM panels appeared to have slightly lower OHC, but comparable SBS at high temp. & retained good mechanical properties after aging
- Need process development to reduce void content to < 2% for aerospace applications
- Need to improve the resin content of VARTM panels from 47-50% to 35-40%.

Acknowlegements

- **NASA Glenn Tech Transfer Fund**
- **Akron Polymer Systems for supplying RTM370 resin**
- **Ohio Third Frontier Funding (Maverick)**
- **NASA Co-Op Grant to Clark Atlanta U.**

- Linda McCorkle (OAI) : SEM, Rheology
  Acid Digestion
- Brian Shonkwiler(CAU): Mechanical Testing
- Dan Scheiman (ASRC): Thermal Analysis