Fire Retardant Pultrusion Profiles with Improved Mechanical Strength

Doug Copeland

Polynt

ACMA American Composites Manufacturers Association | EPTA

Approach

- Background – Flammability Standards, Flame Retardant (FR) Resin Options, Alumina Trihydrate (ATH) Filler
- Pultrusion Formulas, ATH Ladder Study
- Screening Pultrusion Runs - 2 inch panel, Short Beam Shear
- Cone Calorimeter Results
- Pultrusion Runs – 8 inch panel, Mechanical Results, ASTM E84
- Effect of Filler's Particle Size, Precipitated ATH
- Toughened FR Resin

Test Standards

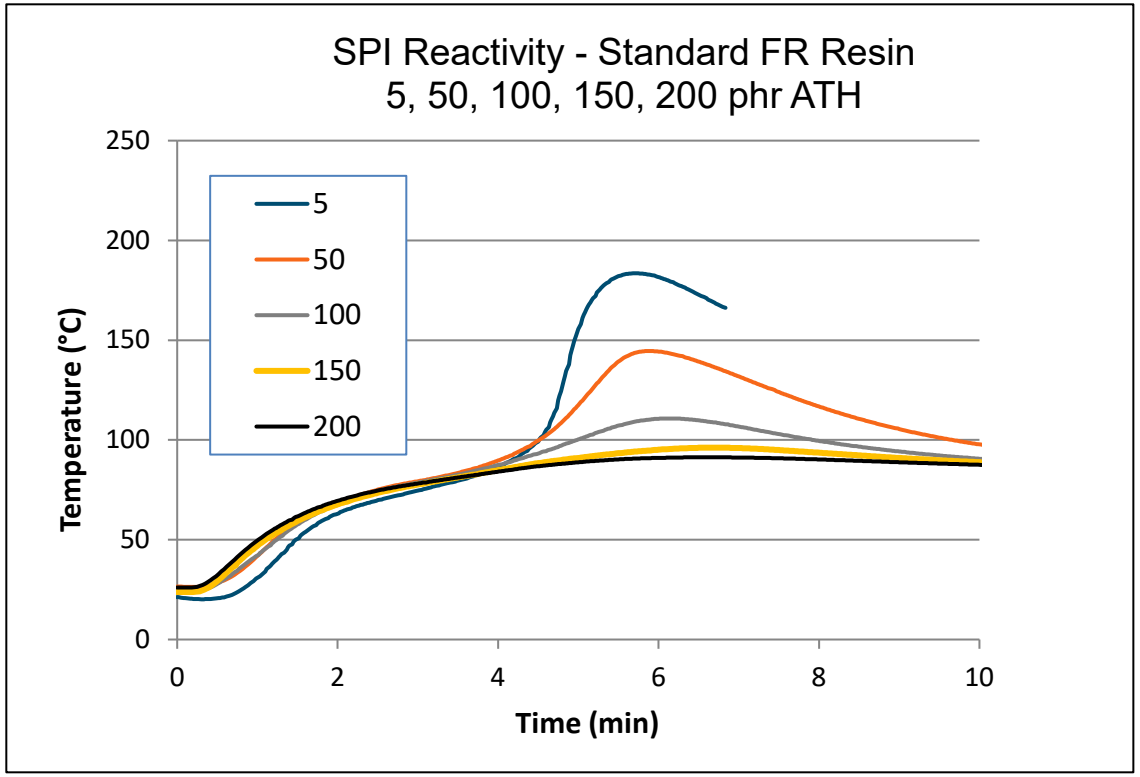
Market	Test Standards	Application
Transportation	FMVSS 302, CMVSS 302	Car, truck and bus vehicle interiors
Electrical	UL-94	Electrical boxes
Transportation	Docket 90 • ASTM E162 Flame Spread • ASTM E662 Smoke Generation • Smoke Toxicity	Mass transit (Bus and Train)
Architecture	ASTM E84 Steiner Tunnel Test – Flame Spread and Smoke Generation	Building Exteriors (limited surface area)
Architecture	NFPA 268 Radiant Burn and NFPA 285 Fire Propagation	Building Exteriors (expanded surface area)

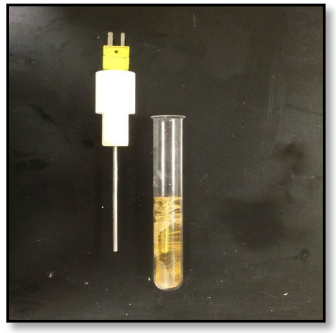
Pultrusion Machine Setup

ATH Ladder in FR Pultrusion Resin Formulation

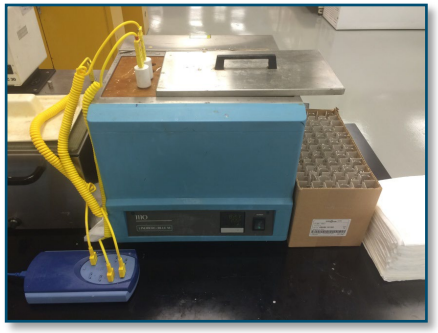
Flame Retardant Resin	100	100	100	100	100
Alumina Trihydrate	5	50	100	150	200
Low Temperature Initiator	0.4	0.4	0.4	0.4	0.4
Mid/High Temperature Initiator	0.8	0.8	0.8	0.8	0.8
Internal Mold Release	1	1.25	1.75	2.25	2.75
Wetting/Dispersing Additive		1	2	3	4
Styrene	0.4	0.4	0.4	0.4	0.4
Viscosity - RV#4 @ 20 rpm, 25ºC	140	280	700	1435	3650
Number of 113 roving	54	48	42	37	30
Pull Force – 1/8 x 2 in. Profile (lb.)					
12 inch/min	77	102	99	72	57
18	122	160	179	133	121
24	188	230	214	182	157

SPI Reactivity ATH Ladder in Standard FR Resin





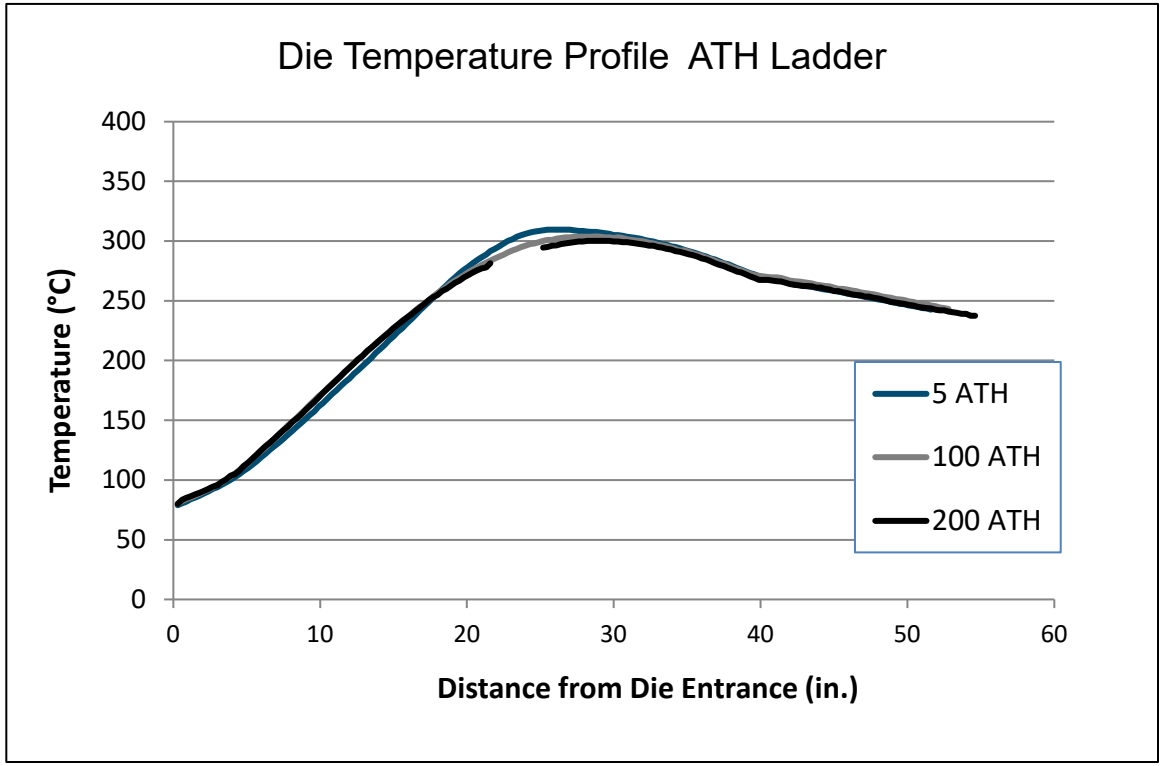
Test Tube and Thermocouple



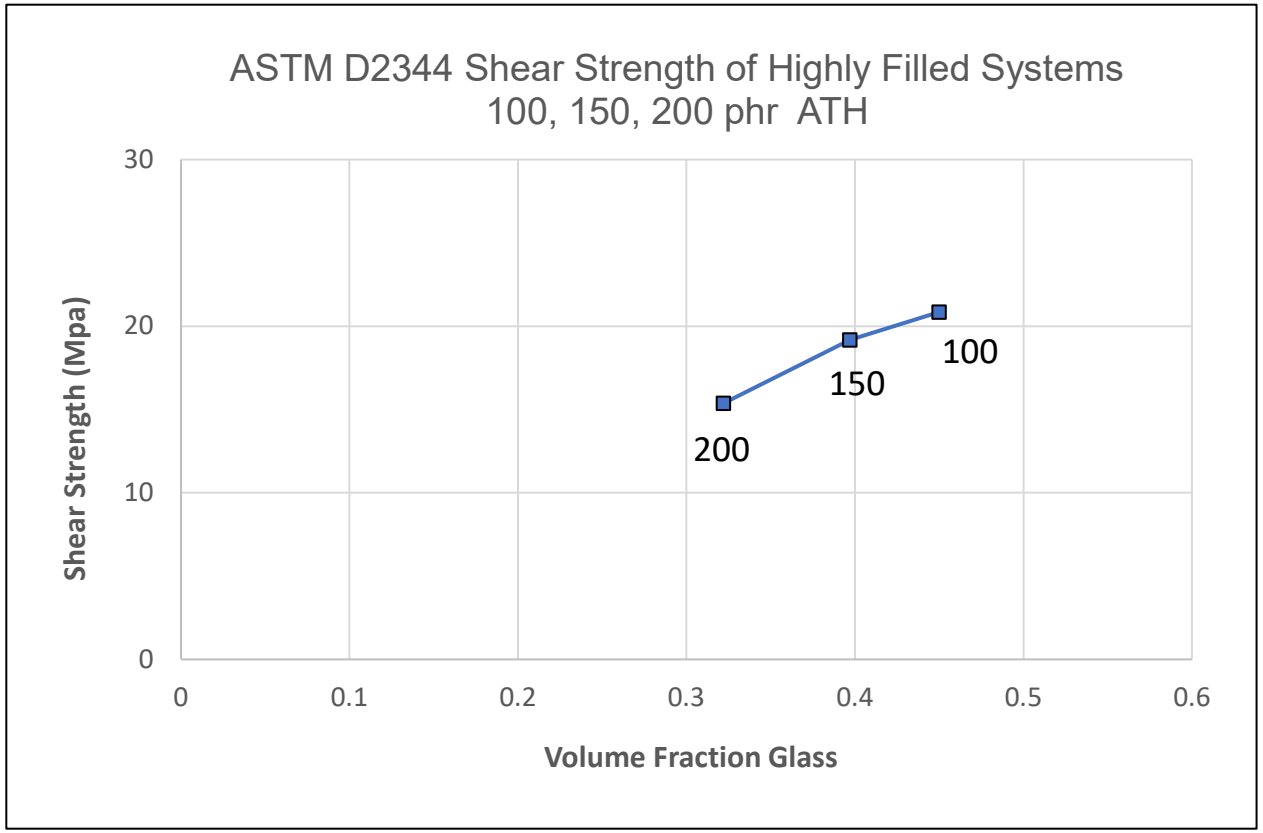
Circulating Water Bath - 180°F

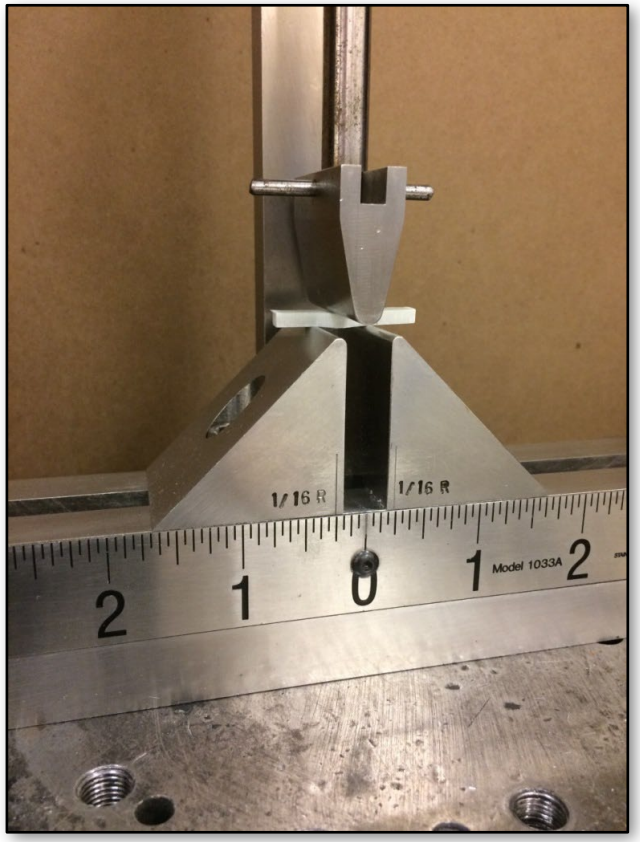
	ATH Filler (phr)				
	5	50	100	150	200
Gel Time (min)	1.9	2.1	2.2	2.6	3.0
Interval (min)	1.6	2.0	2.1	2.3	1.9
Peak Exotherm (°C)	184	144	111	96	91

Temperature Profile Through the Die



ASTM D2344 Short Beam Shear Test





ASTM E1354 Oxygen Consumption Calorimeter

	ATH Filler (phr)				
	5	50	100	150	200
Ends of 113 yield glass	54	48	42	37	30
Volume Fraction	0.58	0.51	0.45	0.40	0.32
Cone Data					
Time to Sustained Ignition (s)	52	51	62	88	101
Peak Rate of Heat Release (kW/m2)	391	283	232	196	199
Average SEA (m2/kg)	753	555	399	313	258

Forest Products Laboratory

Peak Heat Release < 300 and SEA < 300 often translate to Class A in ASTM E84 Test

ASTM E84 Tunnel Test - 150 phr ATH

Time to Ignition (min)	Maximum Flamespread (ft)	Time to Max Flamespread (min)	Flame Spread Index (FSI)	Smoke Developed Index (SDI)	Rating
1.0	5.4	5.6	20	160	Class A

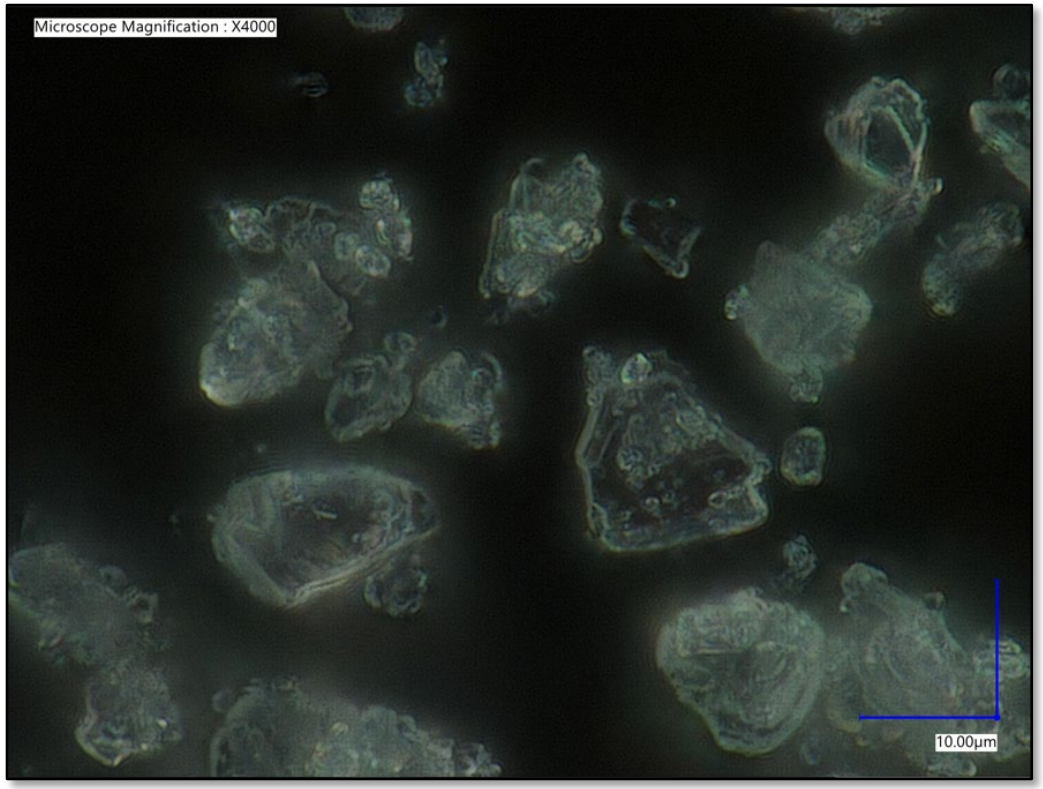
Layup – 56 yield Continuous Glass Roving, 1.0 oz./ft2 Continuous Glass Mat (top and bottom) 0.56 oz./yd2 spunbond polyester veil (top and bottom)

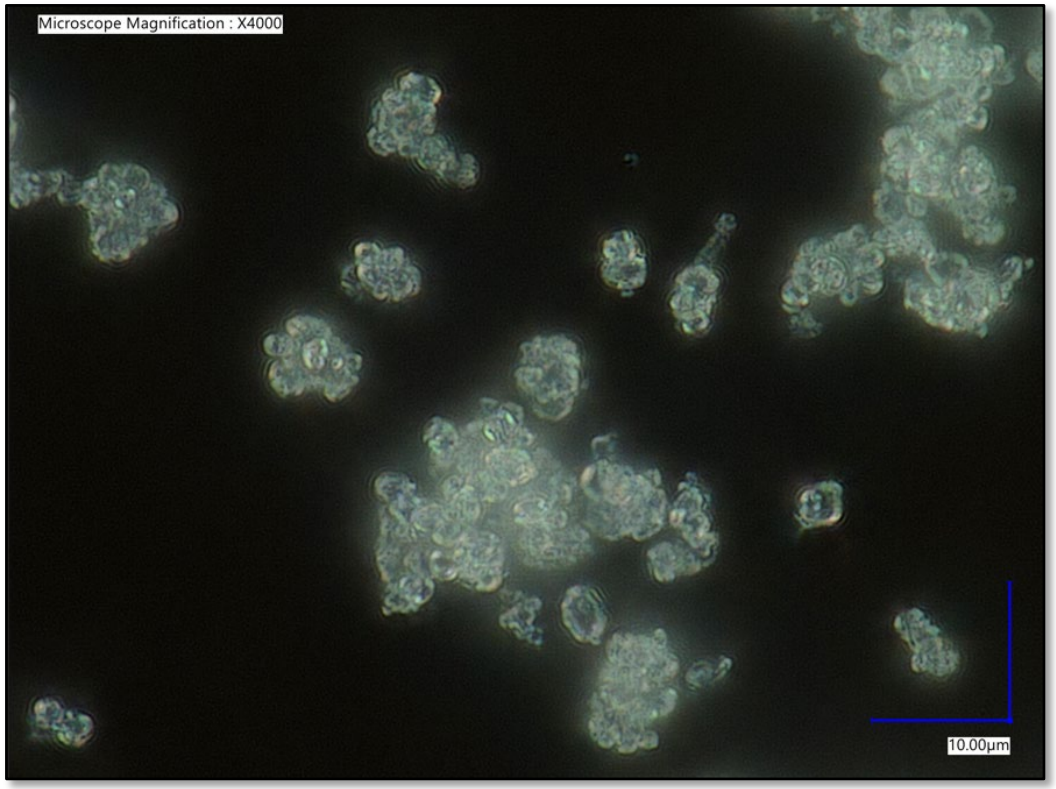
Mechanical Properties – Pultruded 1/8 x 8 inch Panel

	wt% Glass	Resin	Filler
ATH 150 phr	42.7	17.6	39.7

	Tensile (ksi)	Elongation (%)	Compression (ksi)		Flexural (ksi)		Short Beam Shear (Mpa)	
	Longitudinal		Longitudinal	Transverse	Longitudinal	Transverse	Longitudinal	Transverse
ATH 150 phr	96	1.8	43.8	8.7	44.6	14.8	18.8	17.6

Ground vs Precipitated ATH, 4000x

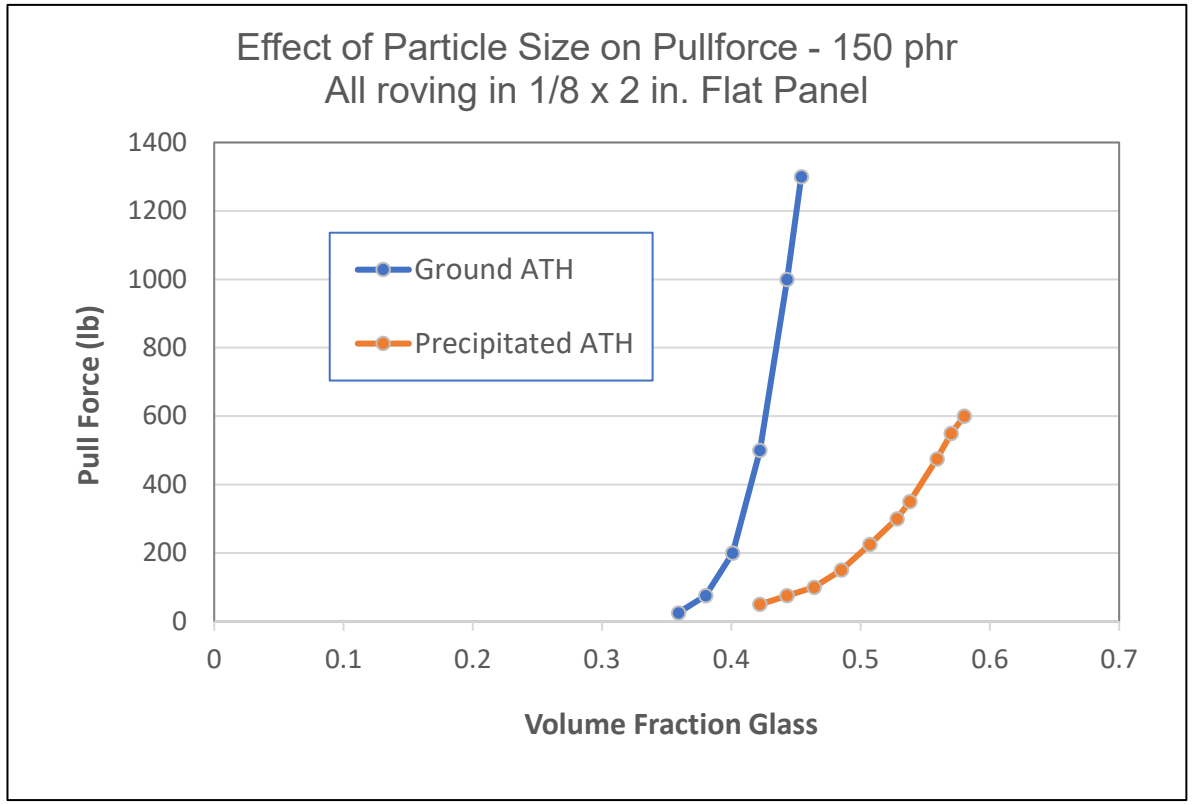


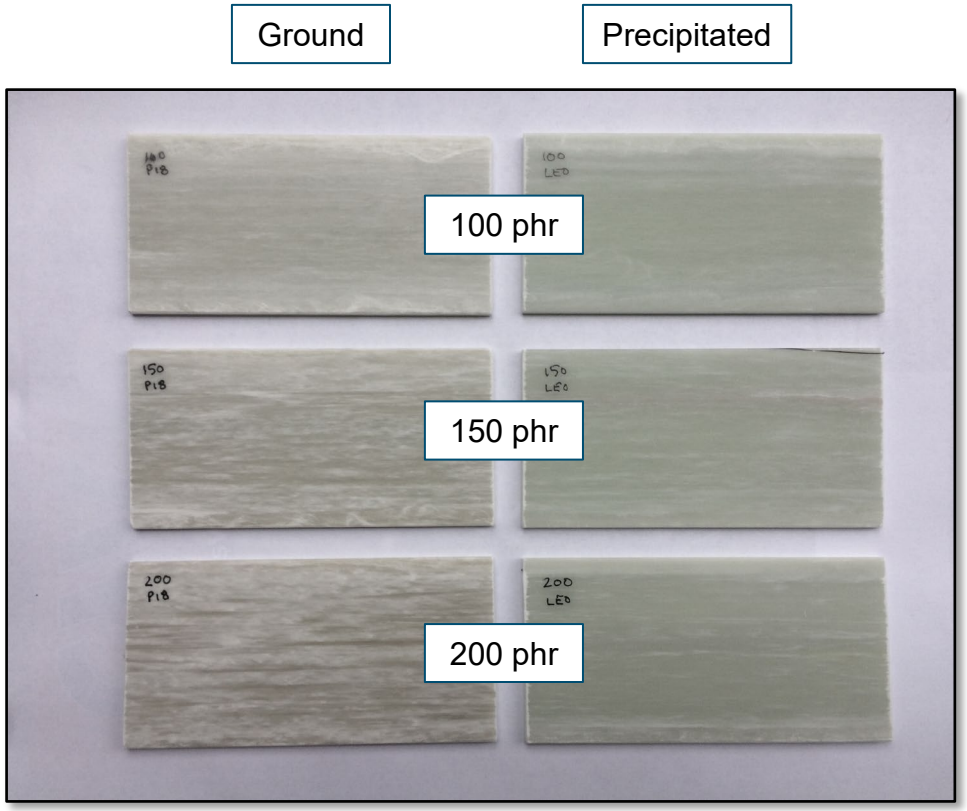


Viscosity and Pull Force - Ground vs Precipitated ATH

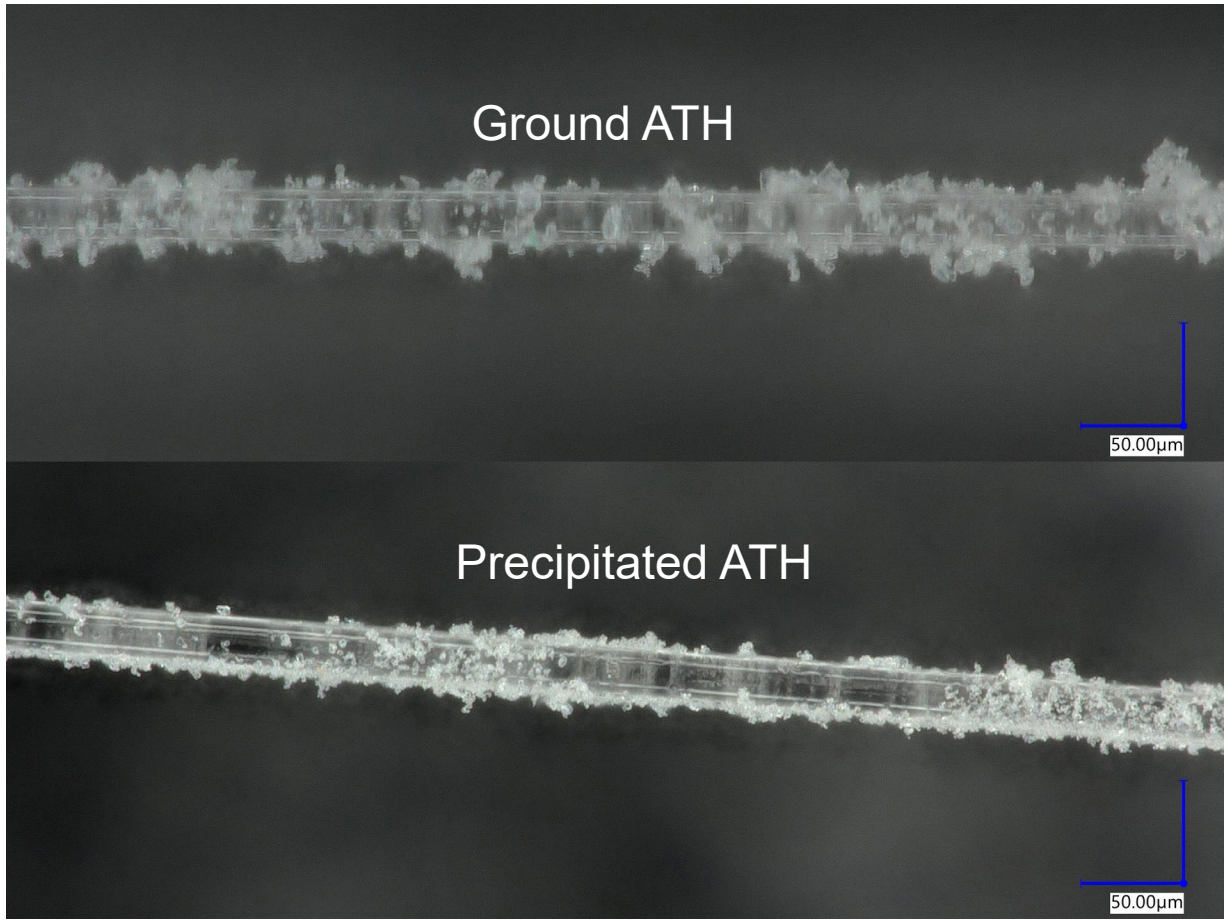
	Ground ATH			Precipitated ATH		
ATH	100	150	200	100	150	200
Viscosity (cP)	700	1435	3650	600	1425	3050
Number of 113 roving	42	37	30	52	47	42
Line Speed (inch/min.)	**Pull Force – 1/8 x 2 in. Profile (lb.)**					
12 inch/min	99	72	57	116	93	94
18	179	133	121	140	136	139
24	214	182	157	181	200	192

Ground vs Precipitated ATH - 2 inch Pultruded Panel

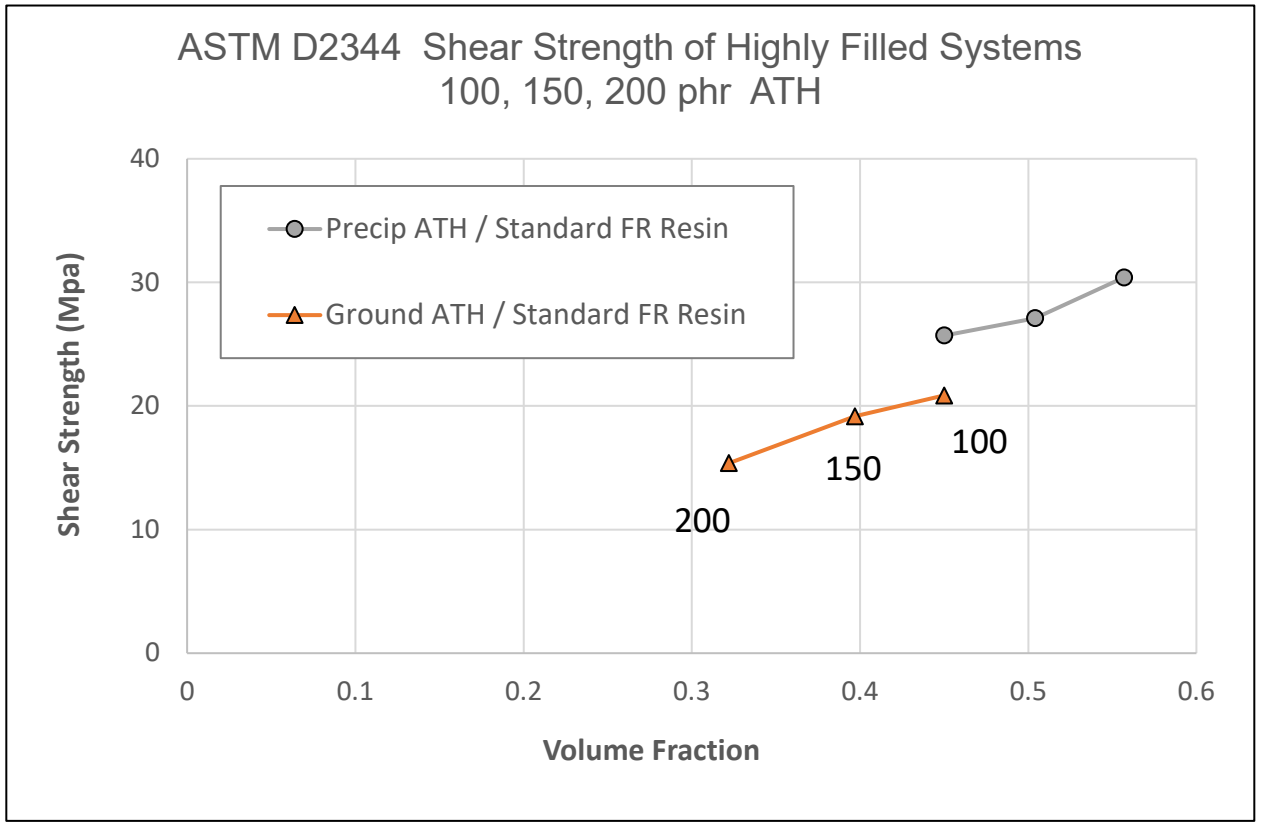


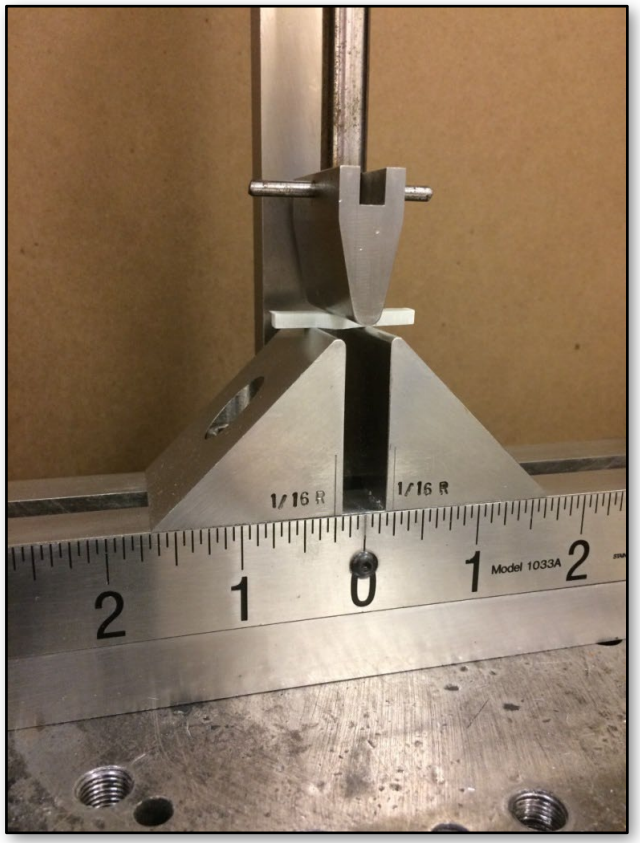


Ground vs Precipitated, 500x - 113 yield Glass Filament



Short Beam Shear Test – Ground vs Precipitated ATH

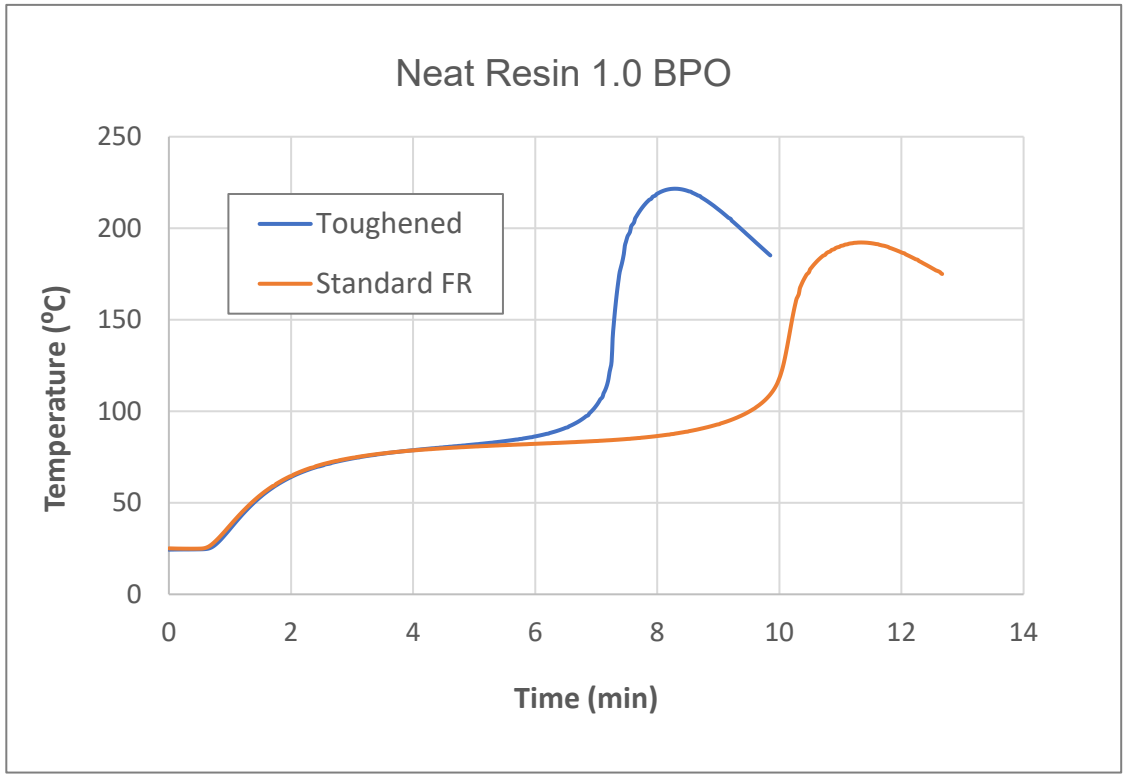


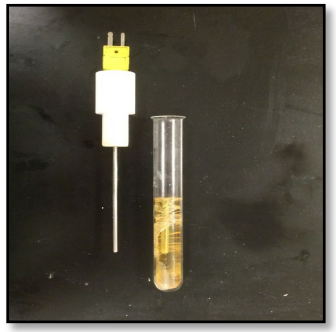


ASTM E1354 Oxygen Consumption Calorimeter

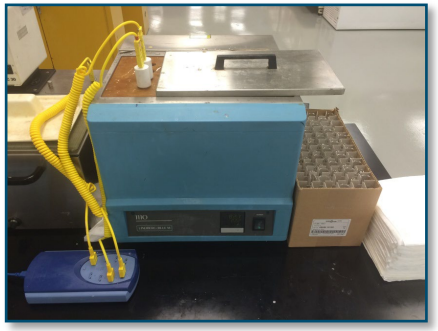
	ATH Filler (phr)					
	Ground ATH			Precipitated ATH		
	100	150	200	100	150	200
Ends of 113 yield glass	42	37	30	52	47	42
Volume Fraction	0.45	0.40	0.32	0.56	0.50	0.45
Cone Calorimeter Data						
Time to Sustained Ignition (s)	62	88	101	71	75	90
Peak Rate of Heat Release (kW/m2)	232	196	199	231	191	186
Average SEA (m2/kg)	399	313	258	416	322	261

SPI Reactivity Neat FR Resin, Standard and Toughened





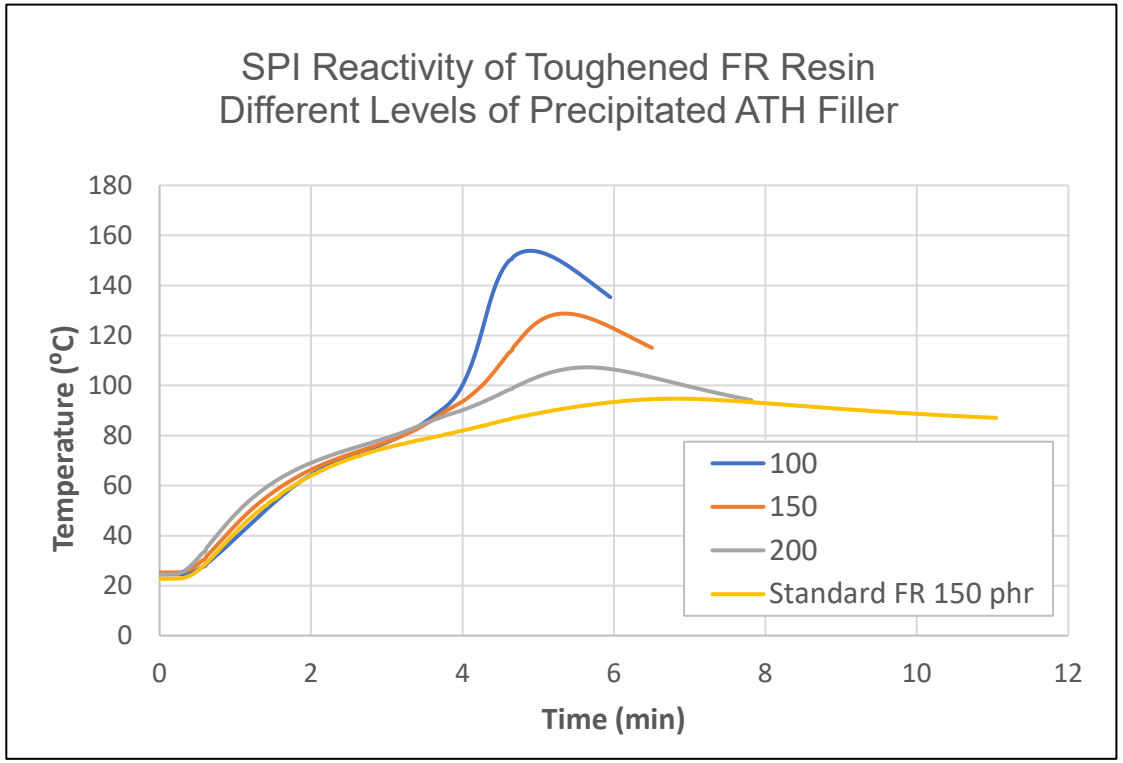
Test Tube and Thermocouple

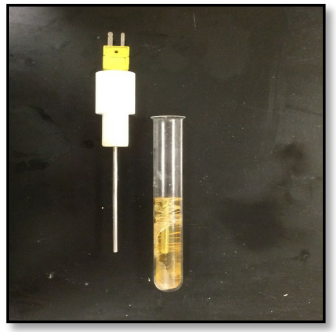


Circulating Water Bath - 180°F

	Resin	
	Standard	Toughened
Gel Time (min)	6.2	4.1
Interval (min)	3.1	2.1
Peak Exotherm (°C)	192.3	221.6

SPI Reactivity ATH Ladder in Toughened FR Resin





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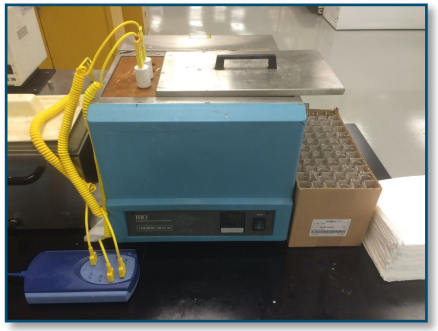


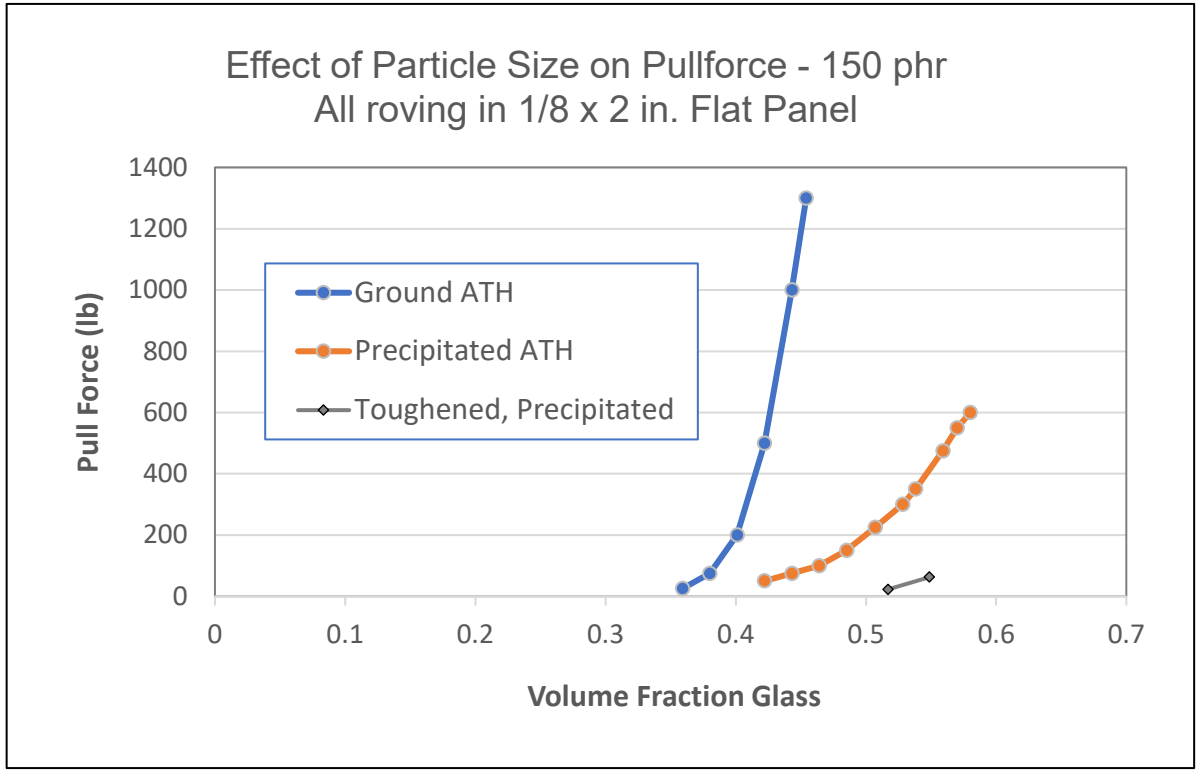
Circulating Water Bath - 180°F

	ATH Filler (phr)			
	100	150	200	150 std
Gel Time (min)	1.6	1.7	2.0	2.6
Interval (min)	1.3	1.7	1.9	2.3
Peak Exotherm (°C)	154	129	107	96

Viscosity and Pull Force - Precipitated ATH / Toughened Resin

	Precipitated ATH			Toughened Resin / Precipitated ATH		
ATH	100	150	200	100	150	200
Viscosity (cP)	600	1425	3050	736	1360	3020
Number of 113 roving	52	47	42	54	49	46
Line Speed (inch/min.)	**Pull Force – 1/8 x 2 in. Profile (lb.)**					
12 inch/min	116	93	94	49	23	44
18	140	136	139			
24	181	200	192			

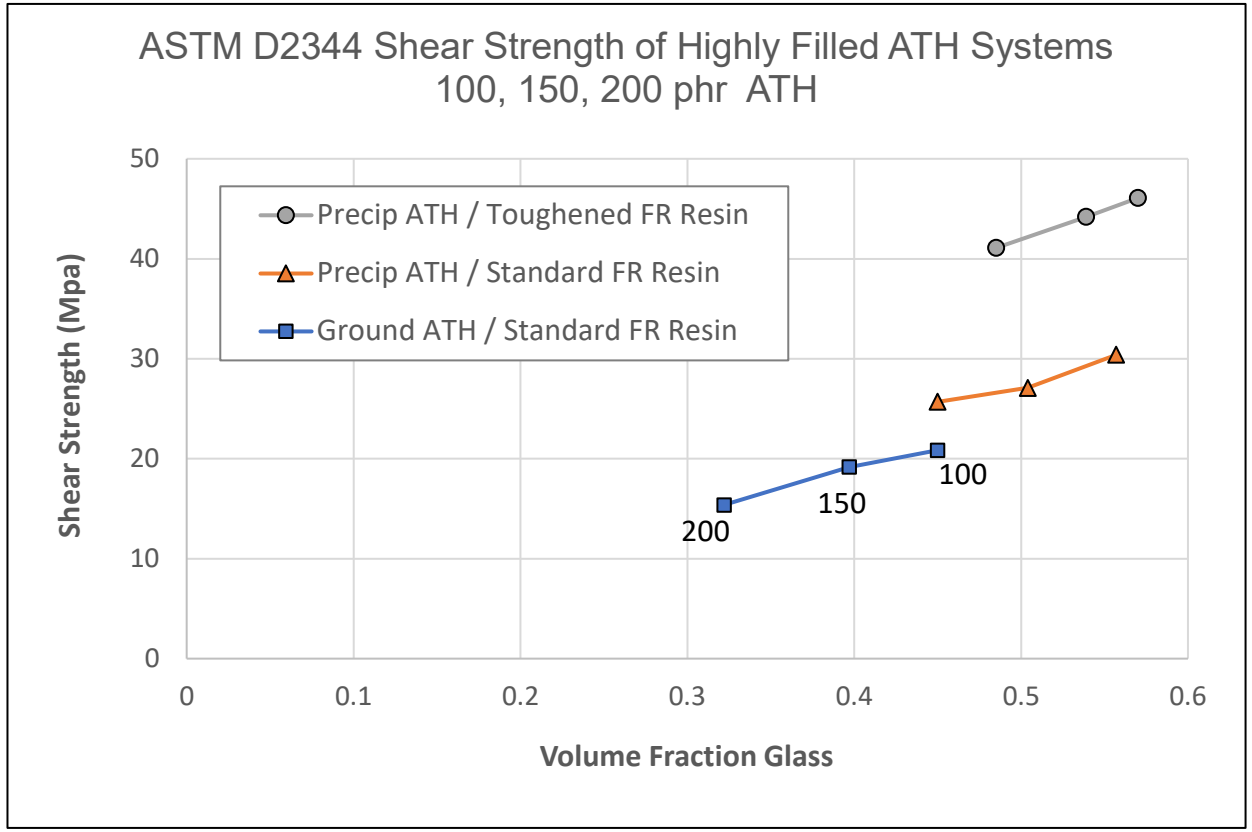
Ground vs Precipitated ATH - 2 inch Pultruded Panel

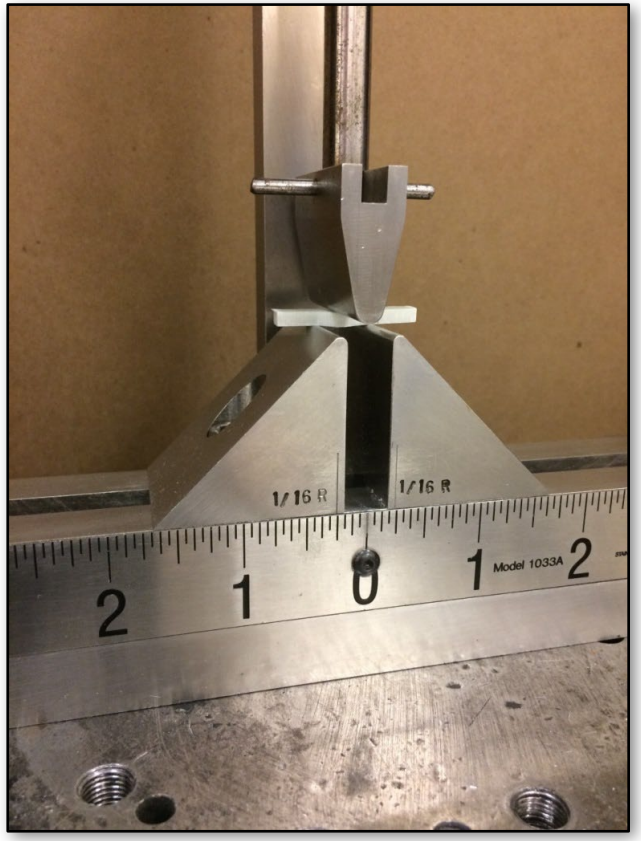


ASTM E1354 Oxygen Consumption Calorimeter

	ATH Filler (phr)								
	Ground ATH			Precipitated ATH			Toughened / Precipitated ATH		
	100	150	200	100	150	200	100	150	200
Ends of 113 yield glass	42	37	30	52	47	42	54	51	46
Volume Fraction	0.44	0.39	0.32	0.55	0.50	0.44	0.57	0.54	0.49
Cone Calorimeter Data									
Time to Sustained Ignition (s)	62	88	101	71	75	90	89	98	113
Peak Rate of Heat Release (kW/m2)	232	196	199	231	191	186	212	178	153
Average SEA (m2/kg)	399	313	258	416	322	261	457	344	313

Short Beam Shear Test – Ground vs Precipitated ATH





ASTM E84 Tunnel Test

	Glass wt %	Time to Ignition (min)	Maximum Flamespread (ft)	Time to Max Flamespread (min)	Flame Spread Index (FSI)	Smoke Developed Index (SDI)	Rating
Standard Resin 150 phr Ground ATH	43	1.0	5.4	5.6	20	160	Class A
Toughened Resin 100 phr Precipitated ATH	60	1.7	5.0	9.5	20	95	Class A

Mechanical Properties – Pultruded 1/8 x 8 inch Panel

	wt% Glass	Resin	Filler
Standard Resin, Ground ATH 150 phr	42.7	17.6	39.7
Toughened Resin, Precipitated ATH 100 phr	60.4	15.9	23.7

	Tensile (ksi)	Elongation (%)	Compression (ksi)		Flexural (ksi)		Short Beam Shear (Mpa)	
	Longitudinal		Longitudinal	Transverse	Longitudinal	Transverse	Longitudinal	Transverse
Ground 150 phr	96	1.8	43.8	8.7	44.6	14.8	16.3	13.7
Precip / Tough 100 phr	119	2.2	54.0	13.2	71.5	24.2	28.8	15.4
% Change	+13	+ 22	+ 23	+ 52	+ 60	+64	+ 77	+ 12

Layup – 56 yield Continuous Glass Roving, 1.0 oz./ft2 Continuous Glass Mat (top and bottom)
0.56 oz./yd2 spunbond polyester veil (top and bottom)

Conclusions

- Class A performance for ASTM E84 test can be achieved with 150 phr ATH and Standard FR Resin
- Precipitated ATH allows for higher glass content and mechanical properties
- Class A performance can be achieved with 100 phr ATH and precipitated ATH
- A Toughened FR resin significantly increases mechanical performance