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Bond Process Qualification Protocols & Adhesive Qualification Guidance for Aircraft Design and Certification

2018 Technical Review

Waruna Seneviratne, John Tomblin, and Upul Palliyaguru

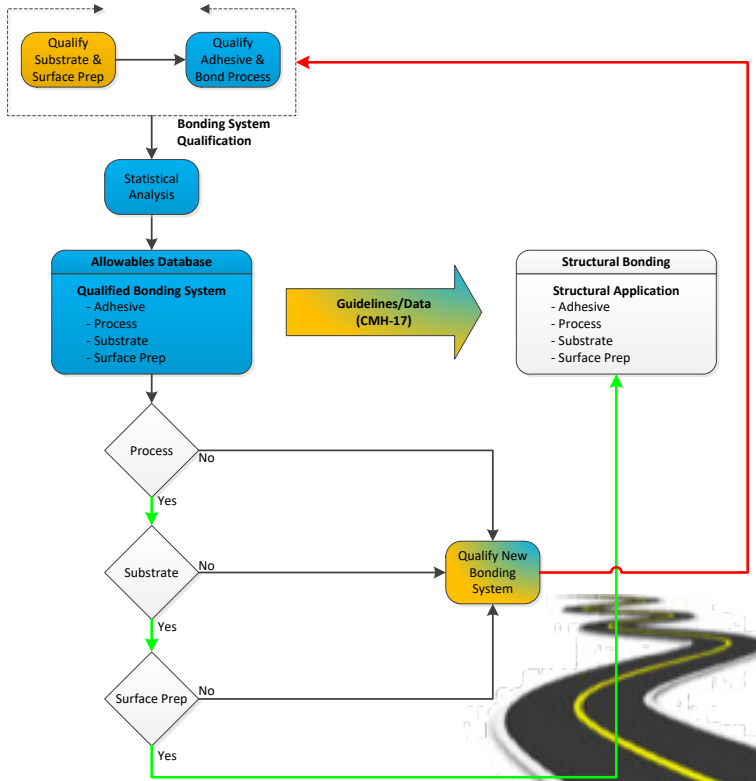
NIAR | Wichita State University
National Institute for Aviation Research

Bond Process Qualification Protocols & Adhesive Qualification Guidance for Aircraft Design and Certification

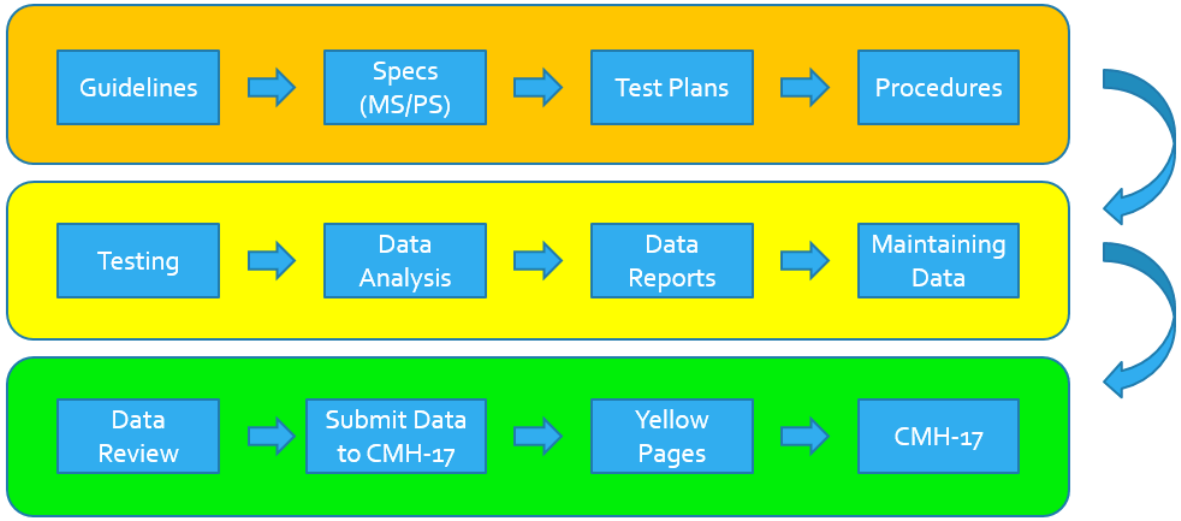
- Principal Investigators & Researchers
  - John Tomblin, *PhD*, and Waruna Seneviratne, *PhD*
  - *Upul Palliyaguru, Anushi Amaranayake, and Tammy Nguyen*
- FAA Technical Monitor
  - Ahmet Oztekin
- Other FAA/CMH-17 Personnel Involved
  - Larry Ilcewicz, *PhD, Cindy Ashforth, and* Curtis Davies,
- DoD & Industry Participation
  - AFRL, Boeing, Bell Helicopter, Henkel, Honda Aircraft Co., Lockheed Martin, MMM, MTech Engineering Services, NAVAIR, Solvey Industries , Textron Aviation, Boom Aerospace

Road Map - Adhesive Qualification Guidance

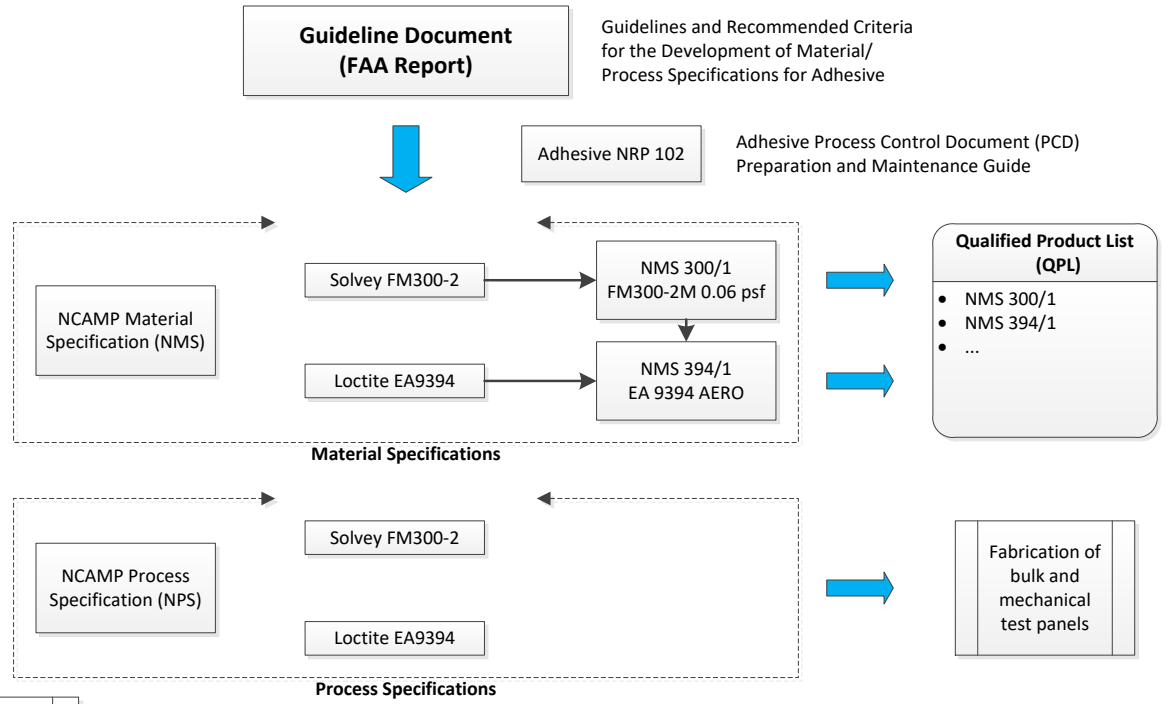
- Adhesive Characterization Qualification (ACQ)
  - Develop test matrices
    - Bulk physical, chemical, and mechanical test matrices
    - Adhesive (joint) mechanical tests
    - Fluid sensitivity
    - Equivalency tests
  - Develop databases
    - Select adhesive bond systems
    - Inclusion to CMH-17 data review group



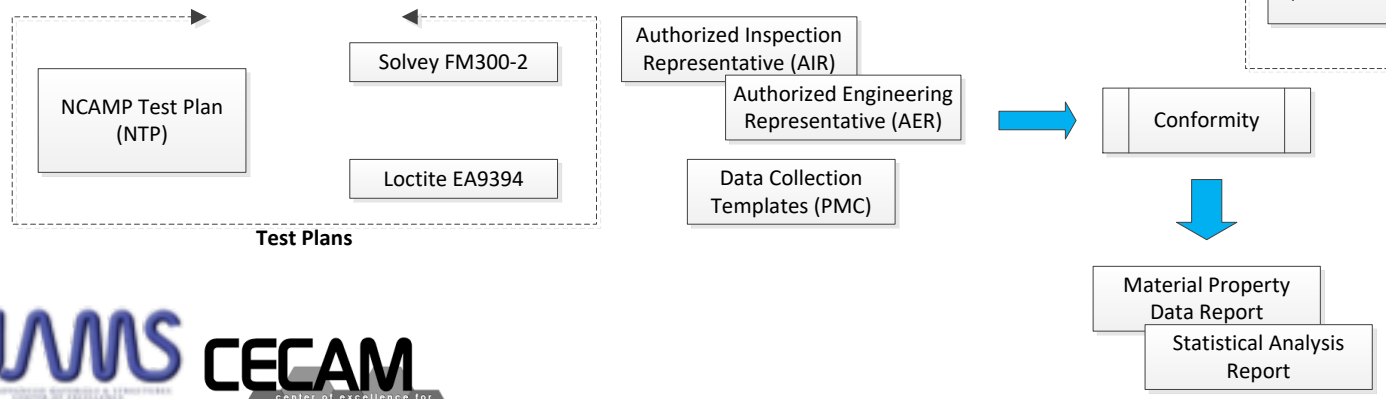
Road Map - Adhesive Qualification Guidance



Development of NCAMP Specifications

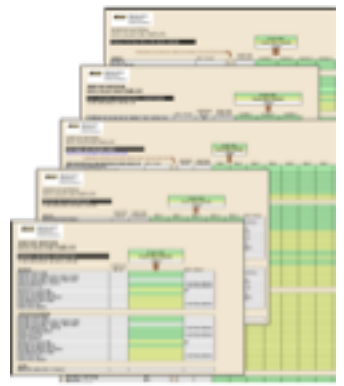


Development of NCAMP Test Plans



Development of NCAMP Specifications, Test Plans & Guidelines

- Adhesive System 1 – FM300-2M (Film Adhesive)
  - NCAMP Material Specification (Base) – NMS300
  - NCAMP Material Specification (Slash) – NMS300/1
  - NCAMP Process Specifications - NPS 83002
  - NCAMP Test Plan - NTP AC-3002Q1
- Adhesive System 2 – EA9394 bare (Paste Adhesive)
  - NCAMP Material Specification (Base) – NMS394
  - NCAMP Material Specification (Slash) – NMS394/1
  - NCAMP Process Specifications - NPS 89394
  - NCAMP Test Plan - NTP AC-9394Q1
- Adhesive Process Control Document (PCD) – NRP 105
- NCAMP - Adhesive Data Collection Forms



NCAMP Material Specifications
Summary of documents -> NMS 300 (Base)
250°F Cure High Temperature and High Toughness Structural Film Adhesive

- Covers Solvay FM300-2 types
  - Carrier material types
    - Type K – Wide open knit
    - Type M – Random mat
    - Type U – Unsupported film
  - Nominal weights
    - 0.10 psf
    - 0.08 psf
    - 0.06 psf
    - 0.03 psf
  - Changes to the qualified materials
  - Tests performed on adhesive

Uncured adhesive physical and chemical properties

Property	Product Form	Test Standard	# of Replicates
HPLC	Uncured	SACMA SRM 20R-94	3 per Batch
FTIR	Uncured	ASTM E-168 ASTM E-1252	3 per Batch
Viscosity & Gel Time	Uncured	ASTM D4473	3 per Batch
Resin Flow	Uncured	ASTM D3531	3 per Batch
Volatile Content	Uncured	ASTM D3530	3 per Batch

Cured adhesive physical and thermal properties

Property	Product Form	Test Standard	# of Replicates
Cured Panel Thickness	Cured	SACMA SRM 10R-94	10 per Panel
Tg	Cured	ASTM D7028	3 per Batch
Density	Cured	ASTM D792 Method A	3 per Batch
Thermal Conductivity & Thermal Diffusivity	Cured	ASTM E1952	3 per Batch
	Cured	ASTM E1530	3 per Batch

Cured adhesive mechanical properties

Property	Test Temperature	Test Method	# of Replicates
Tension Strength & Modulus	RT	ASTM D638	5
Compression Strength and Modulus	RT	ASTM D695	5
Shear Strength and Modulus	RT	ASTM D3846	5

NCAMP Material Specifications
Summary of documents -> NMS 394 (Base)
150°F Cure High Temperature and High Toughness Structural Paste Adhesive

- Covers Henkel EA9394 types
  - Bondline Control Mechanisms
    - EA9394.3 -0.005 Glass beads
  - Adhesive variations weights
    - EA 9394
    - EA 9394/C-2
    - EA 9394.2
  - Changes to the qualified materials
  - Tests performed on adhesive

Uncured adhesive physical and chemical properties

Property	Product Form	Test Standard	# of Replicates
HPLC	Uncured	SACMA SRM 20R-94	3 per Batch
FTIR	Uncured	ASTM E-168 ASTM E-1252	3 per Batch
Viscosity & Gel Time	Uncured	ASTM D4473	3 per Batch
Resin Flow	Uncured	ASTM D3531	3 per Batch
Volatile Content	Uncured	ASTM D3530	3 per Batch

Cured adhesive physical and thermal properties

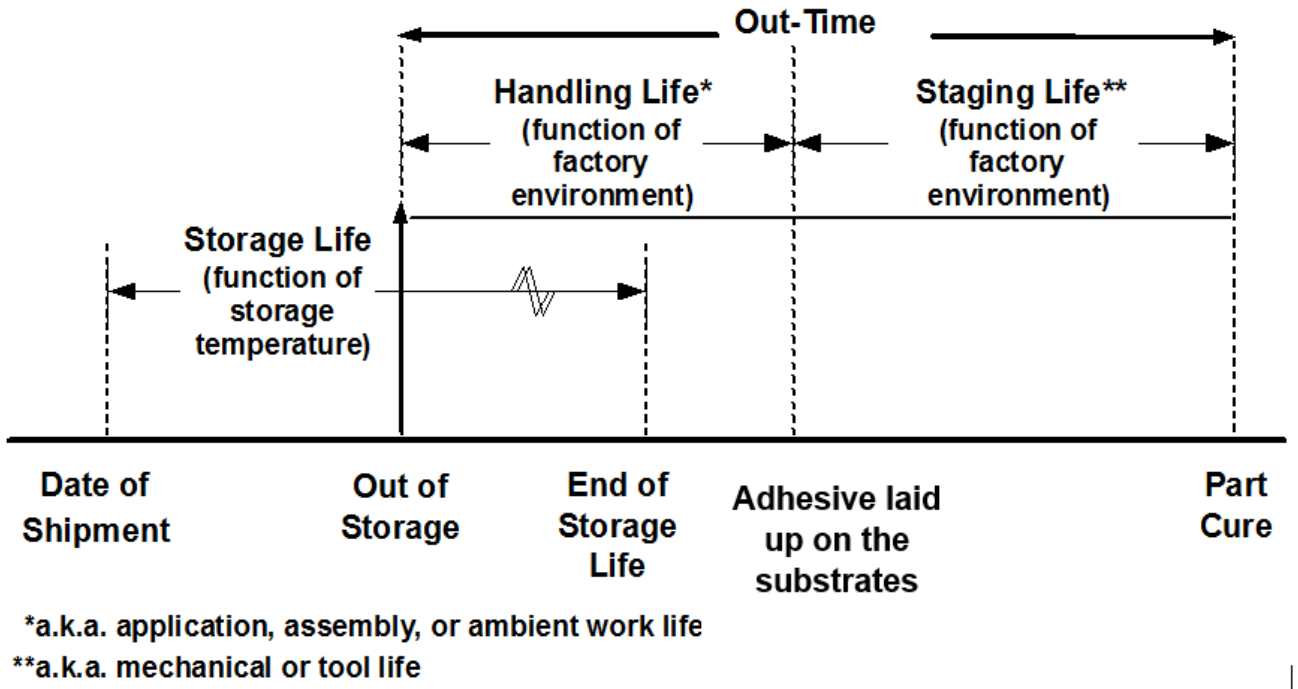
Property	Product Form	Test Standard	# of Replicates
Cured Panel Thickness	Cured	SACMA SRM 10R-94	10 per Panel
Tg	Cured	ASTM D7028	3 per Batch
Density	Cured	ASTM D792 Method A	3 per Batch
Thermal Conductivity & Thermal Diffusivity	Cured	ASTM E1952	3 per Batch
	Cured	ASTM E1530	3 per Batch

Cured adhesive mechanical properties

Property	Test Temperature	Test Method	# of Replicates
Tension Strength & Modulus	RT	ASTM D638	5
Compression Strength and Modulus	RT	ASTM D695	5
Shear Strength and Modulus	RT	ASTM D3846	5

NCAMP Material Specifications
Summary of document -> NMS 300 (Base)
250°F Cure High Temperature and High Toughness Structural Film Adhesive

- Storage and Handling Requirements
  - General requirements
  - Adhesive life requirements
  - Safety requirements
- Quality Assurance
  - Classification of tests and Inspections
  - Receiving inspections
- Delivery of Materials
  - Packaging
  - Shipping Requirements
- Material Rejection



NCAMP Material Specifications
Summary of document -> NMS 300/1 (Slash)
(Solvay FM300-2M – 0.06psf)

- QPL - Solvay FM300-2M – 0.06psf
- Technical Requirements

Uncured adhesive physical and chemical properties

Property	Product Form	Test Standard[1]	# of Replicates (Every roll)	Requirements
HPLC	Uncured	SACMA SRM 20R-94	3	TBD
FTIR	Uncured	ASTM E168 ASTM E 1252	3	TBD
Viscosity & Gel Time	Uncured	ASTM D4473	3	TBD
Resin Flow	Uncured	ASTM D2183	3	Minimum Average – 575%; Ind. Min 540.5%
Volatile Content	Uncured	ASTM D3530	3	Average maximum – 1%

Cured bulk adhesive mechanical properties

Property	Test Temperature	Test Method[1]	Number of Replicates	Requirements
Tension Strength & Modulus	RT	ASTM D638	5	TBD
Compression Strength and Modulus	RT	ASTM D695	5	TBD
Shear Strength and Modulus	RT	ASTM D3846	5	TBD

Adhesive mechanical properties

Property	Test Temperature	Test Method[1]	Number of Replicates	Requirements
Lap Shear Strength	RT	ASTM D1002	5	Minimum Average 4850 psi; Ind. Min 4559 psi
Flatwise Tensile Strength	RT	ASTM D897	5	Minimum Average 892 psi; Ind. Min 838 psi
Peel Strength	RT	ASTM D3167	5	Minimum Average 31 in-lb/in psi; Ind. Min 29 in-lb/in

NCAMP Material Specifications
Summary of document -> NMS 394/1 (Slash)
(Henkel EA9394 bare)

- QPL - Henkel EA9394 bare
- Technical Requirements

Uncured adhesive physical and chemical properties

Property	Product Form	Test Standard[1]	# of Replicates (Every roll)	Requirements
HPLC	Uncured	SACMA SRM 20R-94	3	TBD
FTIR	Uncured	ASTM E 168 ASTM E 1252	3	TBD
Viscosity & Gel Time	Uncured	ASTM D4473	3	Minimum Average = 160 Pa·S Ind. Avg =150 Pa.S
Resin Flow	Uncured	ASTM D2183	3	TBD
Volatile Content	Uncured	ASTM D3530-97	3	TBD

Cured bulk adhesive mechanical properties

Property	Test Temperature	Test Method[1]	Number of Replicates	Requirements
Tension Strength & Modulus	RT	ASTM 638	5	Minimum Average 6675 psi; Ind. Min 6274 psi
Compression Strength and Modulus	RT	ASTM D695	5	Minimum Average 10000 psi; Ind. Min 9400 psi
Shear Strength and Modulus	RT	ASTM D3846	5	TBD

Adhesive mechanical properties

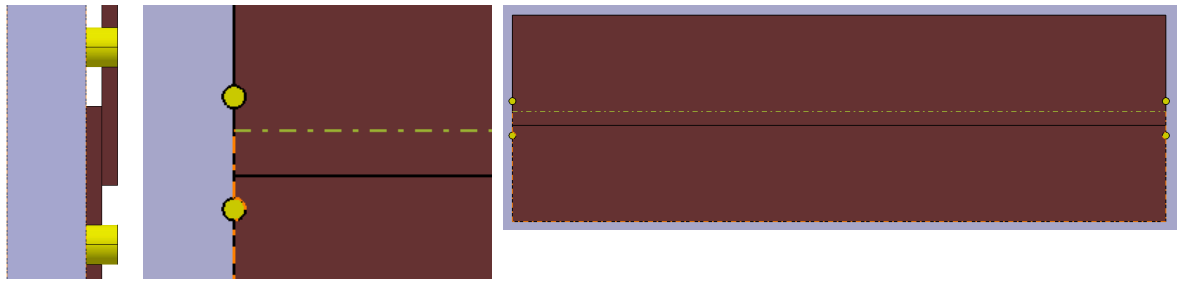
Property	Test Temperature	Test Method[1]	Number of Replicates	Requirements
Lap Shear Strength	RT	ASTM D1002	5	Minimum Average 4850 psi; Ind. Min 4559 psi
Flatwise Tensile Strength	RT	ASTM D897	5	Minimum Average 892 psi; Ind. Min 838 psi
Peel Strength	RT	ASTM D3167	5	Minimum Average 31 in-lb/in psi; Ind. Min 29 in-lb/in

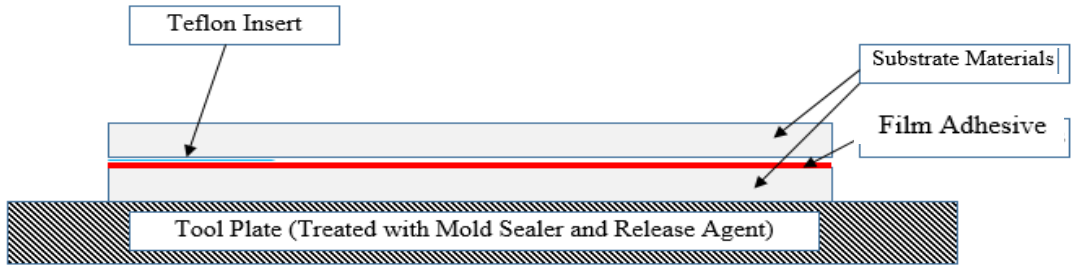
NCAMP Process Specifications
Summary of document -> NPS 83002
Fabrication of NMS 300 Qualification, Equivalency and Acceptance Test Panels

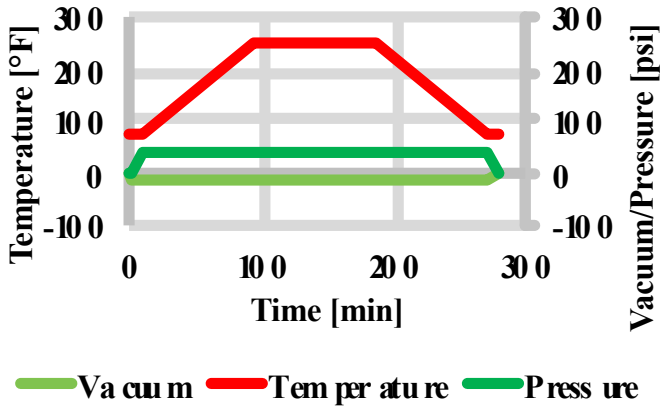
- Materials used for bonded panel fabrication
- Adhesive preparation
- Substrate preparation
  - Aluminum (Al2024 T3) – PAA (ASTM D3933) + BR 127
    - Pre-bond measurements
      - Thickness
      - Surface roughness
      - Primer thickness
  - Composite (Epoxyglass G10) – 120 grit sanding + water break test + cleaning
    - Pre-bond measurements
      - Thickness
      - Surface roughness

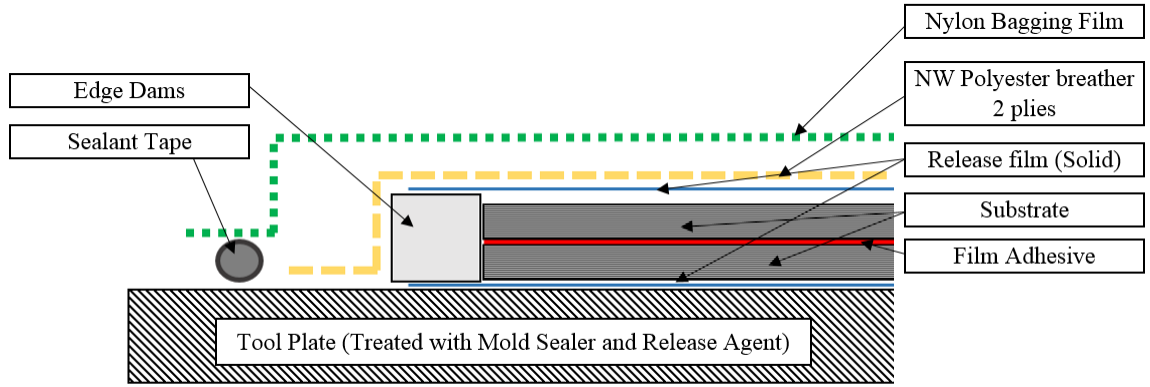
NCAMP Process Specifications
Summary of document -> NPS 83002
Fabrication of NMS 300 Qualification, Equivalency and Acceptance Test Panels

- Fabrication/Bonding of mechanical test panels
  - Bonding of panels with a lap area
  - Bonding of panels without a lap area (Full surface bond)
  - Bonding of panels with a disbond
- Fabrication of bulk adhesive panels
- Bagging Scheme
- Cure Cycle –250°F for 90 minutes with 40 psi pressure.
- Quality control
  - Visual inspection
  - TTU c-scan









NCAMP Test Plan
Summary of document -> NTP AC-3002Q1
Adhesive Property Data Acquisition and Qualification Test Plan for Solvay FM300-2M 0.06psf

- Nomenclature for specimen identification
- Physical, Chemical and Thermal tests performed on adhesive

Uncured adhesive physical and chemical properties

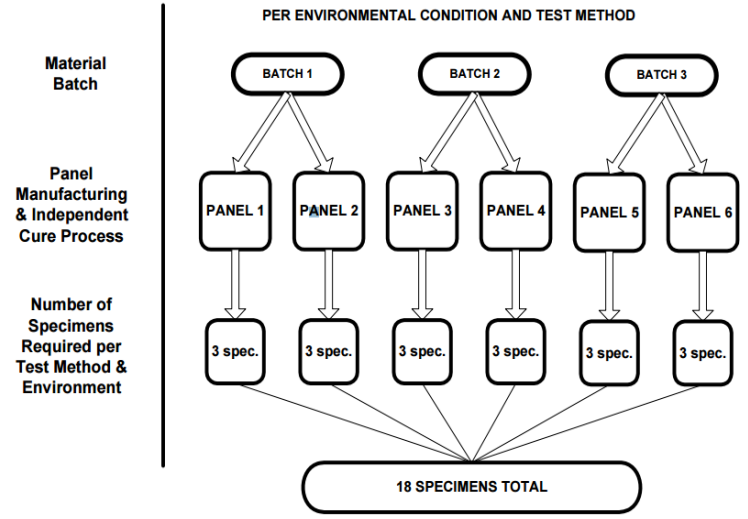
Property	Product Form	Test Standard	Min Replicates per batch
HPLC	Uncured	SACMA SRM 20R-94	3
FTIR	Uncured	ASTM-E168 & ASTM-E1252	3
Viscosity & Gel Time	Uncured	ASTM D4473	3
Resin Flow	Uncured	ASTM D2183	3
Volatile Content	Uncured	ASTM D3530	3

Cured adhesive physical and thermal properties

Property	Product Form	Test Standard	Min Replicates per batch
Tg	Cured	ASTM D7028	3
Density	Cured	ASTM D792 Method A	3
Thermal Conductivity & Thermal Diffusivity	Cured	ASTM E1952	3
	Cured	ASTM E1530	3

NCAMP Test Plan
Summary of document -> NTP AC-3002Q1
Adhesive Property Data Acquisition and Qualification Test Plan for Solvay FM300-2M 0.06psf

- Adhesive Mechanical Property Testing
  - Test Environments -> CTD (-65F), RTD (70F), ETD (180F Dry), and ETW (180F Wet)
  - Dry – as fabricated moisture content
  - Wet – specimens conditioned at 145°F/85%RH for 1000 hrs. (FM300-2M)
- Specimen distribution methodology



NCAMP Test Plan
Summary of document -> NTP AC-3002Q1
Adhesive Property Data Acquisition and Qualification Test Plan for Solvay FM300-2M 0.06psf

- Adhesive Mechanical Property Testing
  - Test Matrix

Test Description	Test Code	Test Standard	Property	Number of Batches x No. of Panels x No. of Specimens			
				Test Temperature/Moisture Condition			
				CTD	RTD	ETD	ETW
Thin Metal Adherend Single Lap Shear	SLS – MThin	ASTM D1002	Strength	3x2x3	3x2x3	1x2x3	3x2x3
Thick Metal Adherend Single Lap Shear	SLS - MThick	ASTM D5656	Strength and Modulus	1x2x3	1x2x3	1x2x3	1x2x3
Thin Laminate Adherend Single Lap Shear	SLS - CThin	ASTM D3165	Strength	3x2x3	3x2x3	1x2x3	3x2x3
Mode I Fracture Toughness	M-I	ASTM D3433	Fracture Toughness Gic	1x2x3	1x2x3	1x2x3	1x2x3
Mode II Fracture Toughness	M-II	ASTM D7905	Fracture Toughness Giic	1x2x3	1x2x3	1x2x3	1x2x3
Floating Roller Peel	FRP	ASTM D3167	Peel Load	3x2x3	3x2x3	1x2x3	3x2x3
Flatwise Tensile	FWT	ASTM D897	Strength	3x2x3	3x2x3	1x2x3	3x2x3

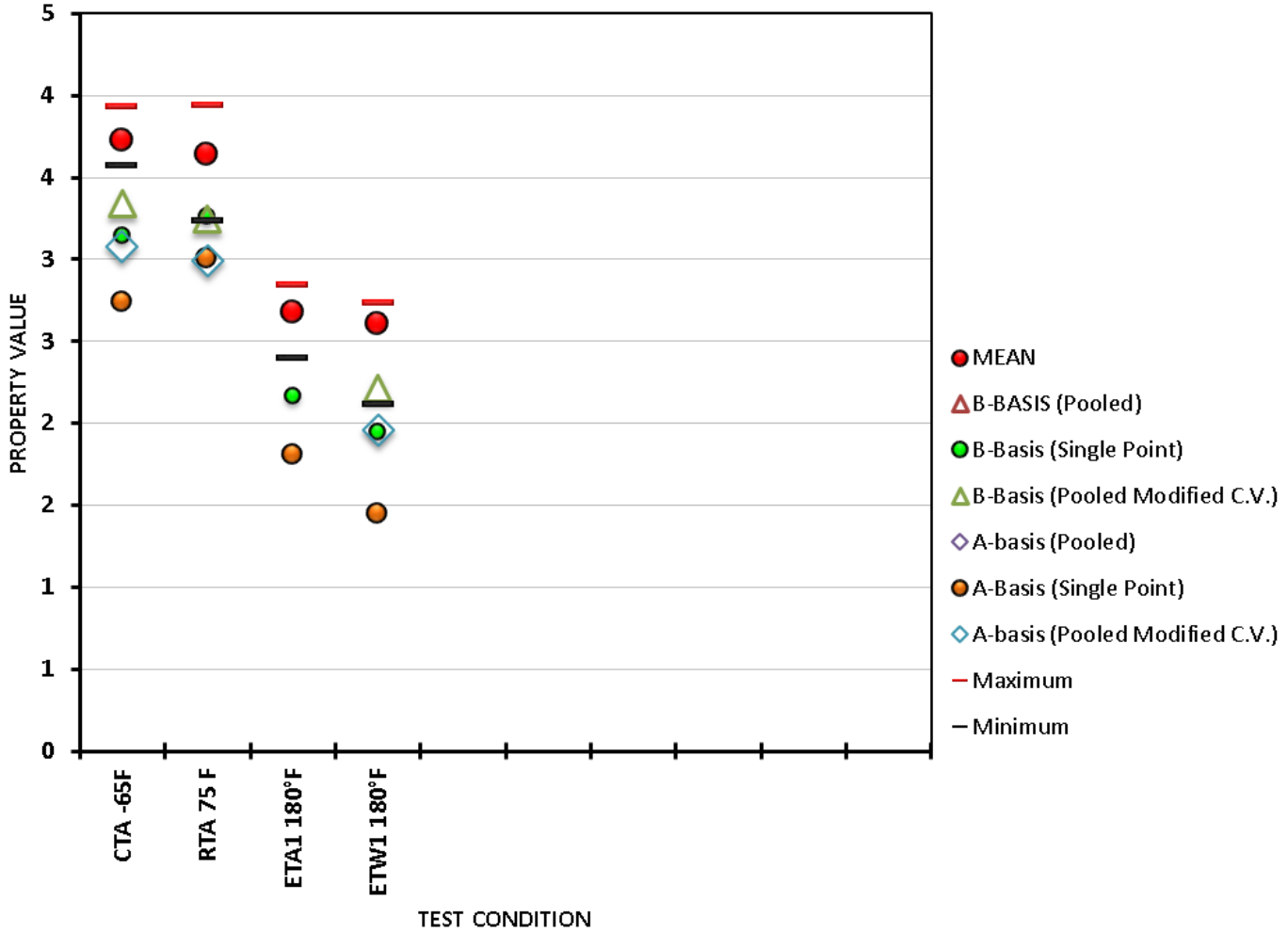
NCAMP Test Plan
Summary of document -> NTP AC-3002Q1
Adhesive Property Data Acquisition and Qualification Test Plan for Solvay FM300-2M 0.06psf

- Adhesive Mechanical Property Testing – Fluid Sensitivity
  - Test Matrix

Extended Contact:	Exposure	Test Condition	Code
SAE AMS 2629 Jet Reference Fluid	30 days min. @ 70°F±10°F	70°F	FS11RT
	30 days min. @ 70°F±10°F	180°F	FS11ET
MIL-PRF-5606 Hydraulic Oil	30 days min. @ 70°F±10°F	70°F	FS12RT
	30 days min. @ 70°F±10°F	180°F	FS12ET
Sea Water (ASTM D1141 or equiv.)	30 days min. @ 70°F±10°F	70°F	FS13RT
	30 days min. @ 70°F±10°F	180°F	FS13ET
Skydrol LD-4 (SAE AS1241, Type IV, Class 1)	30 days min. @ 70°F±10°F	70°F	FS14RT
	30 days min. @ 70°F±10°F	180°F	FS14ET
Short Duration Contact:			
MEK washing fluid. ASTM D740	90 minutes min. @ 70°F±10°F	70°F	FS21RT
	90 minutes min. @ 70°F±10°F	180°F	FS21ET
Control Tests:			
85% Relative Humidity	1000 hrs. at 145°F±5°F /85%±5% RH	70°F	FS31RT
	1000 hrs. at 145°F±5°F /85%±5% RH	180°F	FS31ET

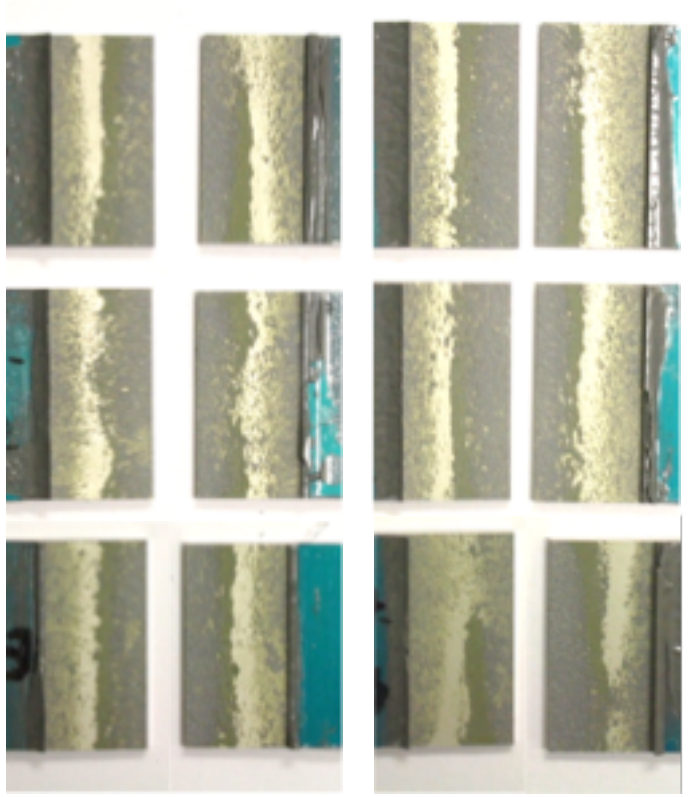
Preliminary Test Results

EA 9394 – D1002 – Property Value – Shear Strength (ksi)



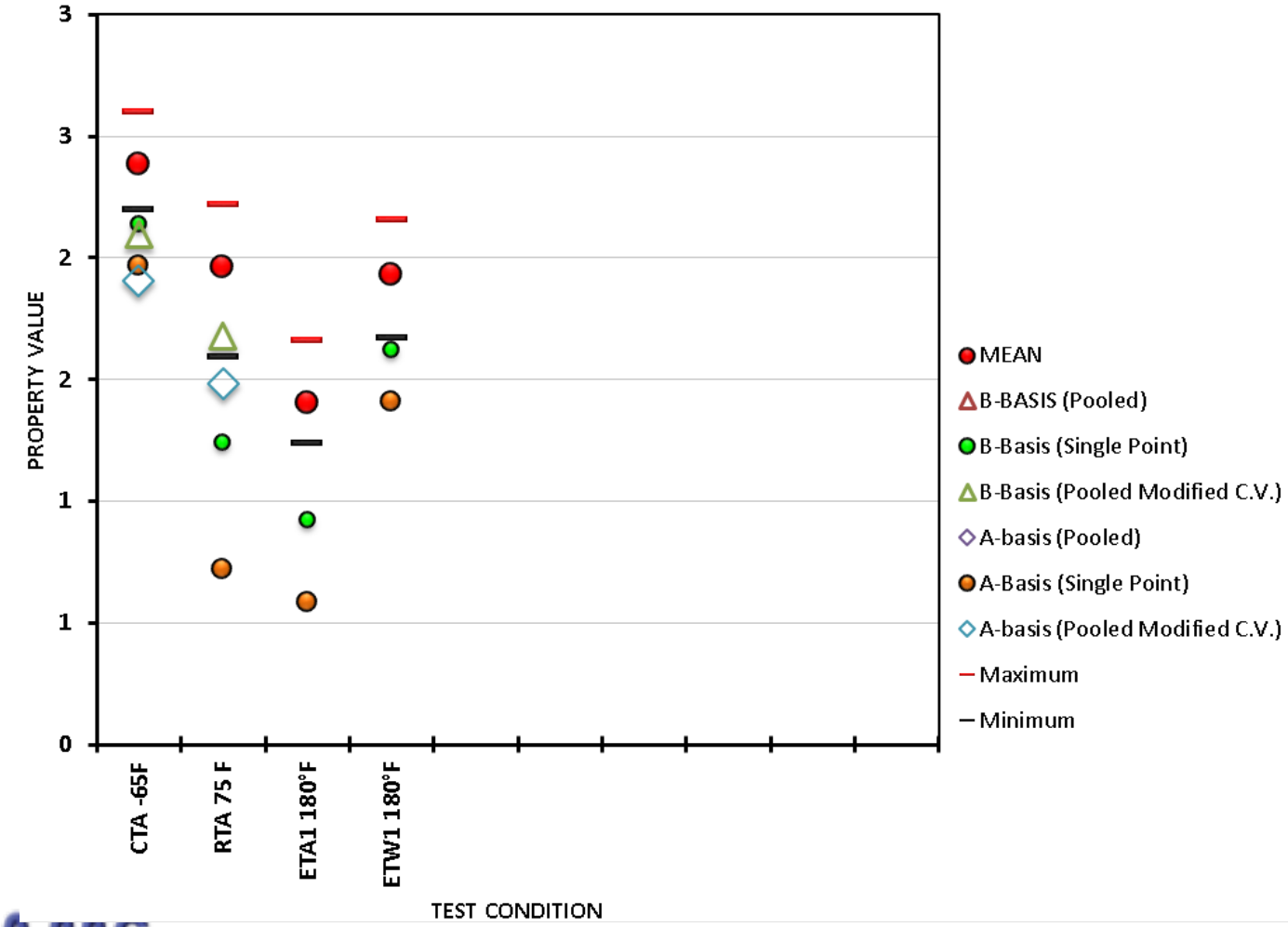
Batch A

Cure Cycle 1 Cure Cycle 2



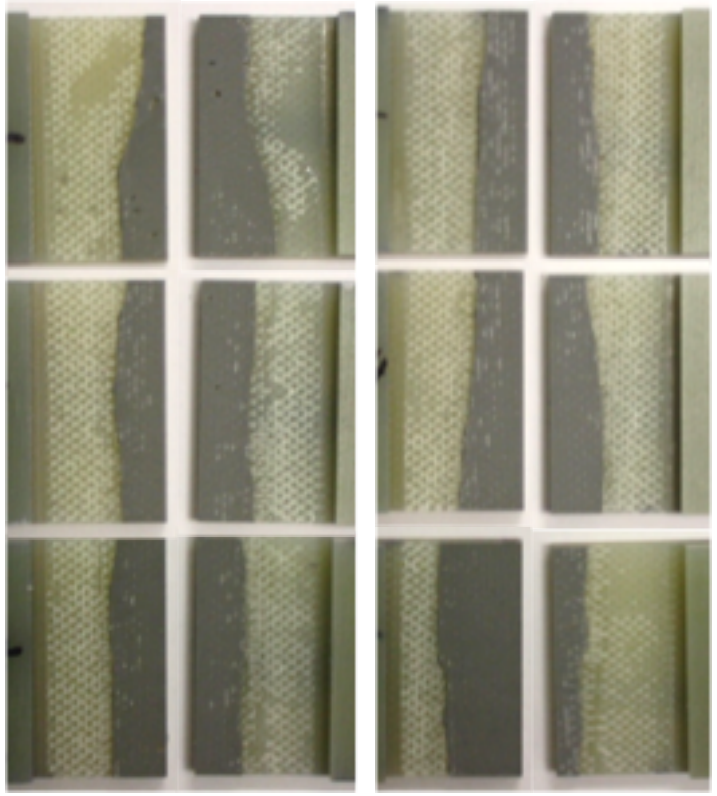
Higher % of Cohesive / Adhesive

Preliminary Test Results
EA 9394 – D3165 – Property Value – Shear Strength (ksi)



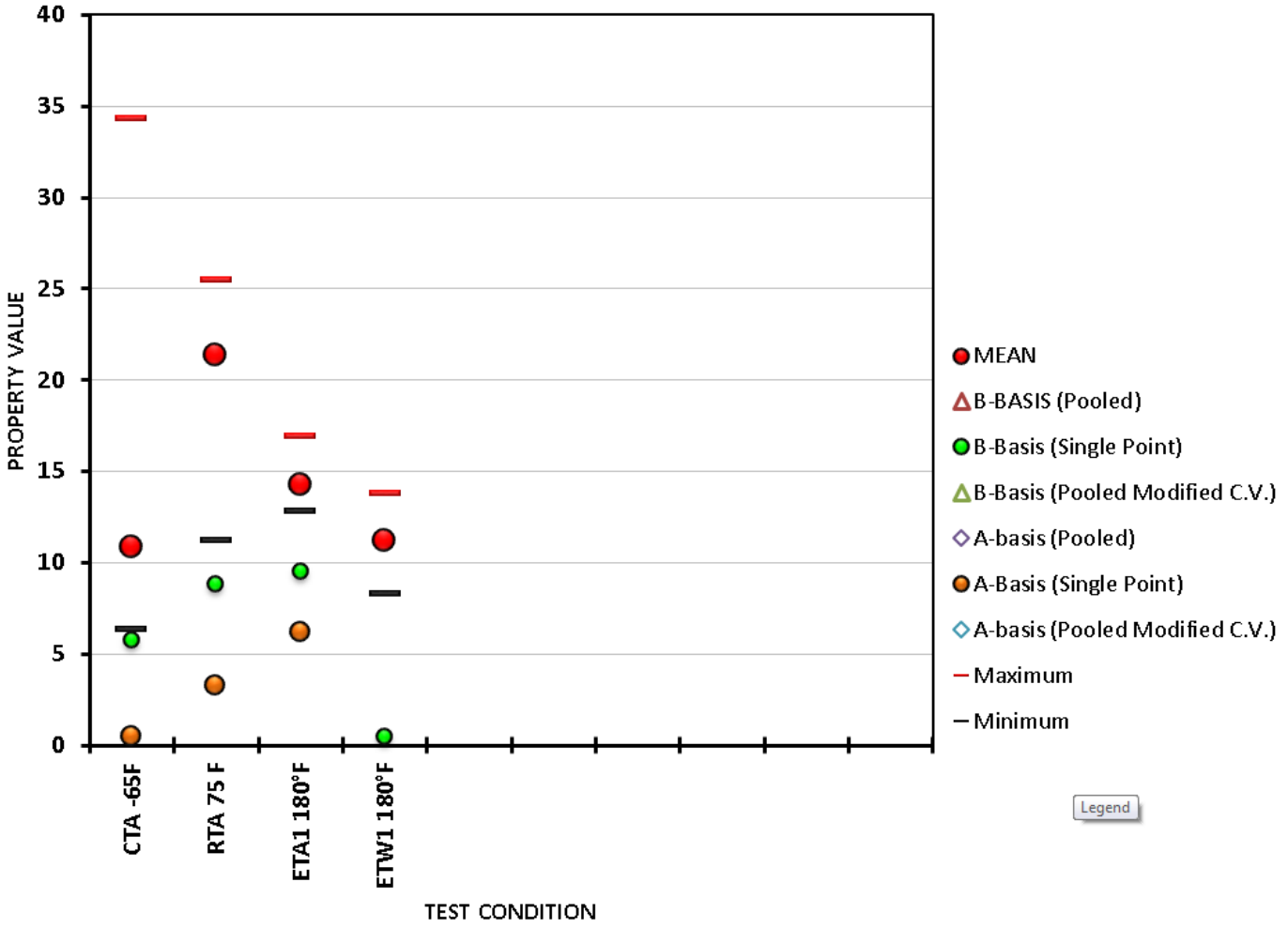
Batch A

Cure Cycle 1 | Cure Cycle 2



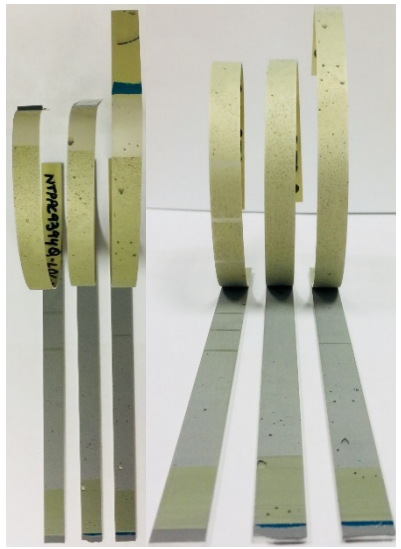
Adhesive Failure | Adhesive Failure

Preliminary Test Results
EA 9394 – D3167 – Property Value – Peel Load (lbf)



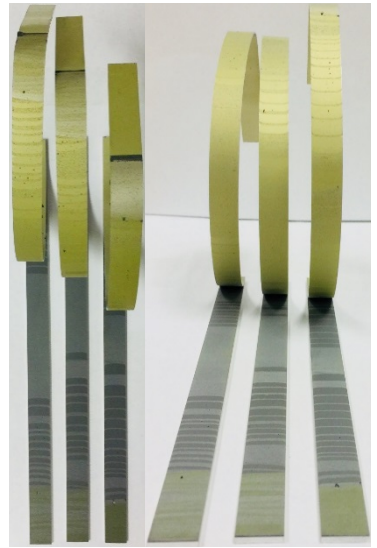
Batch A

Cure Cycle 1



Adhesive Failure

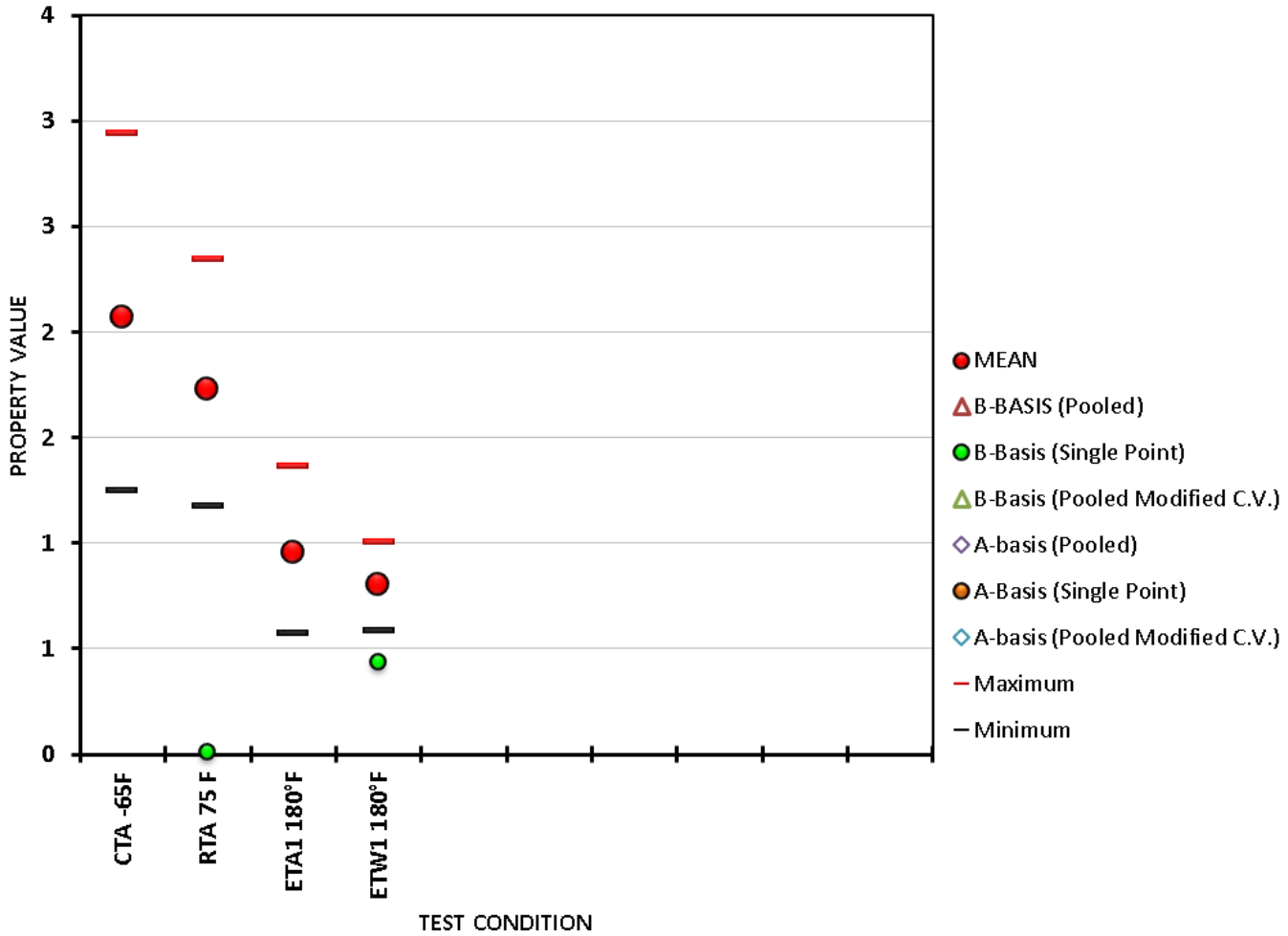
Cure Cycle 2



Adhesive Failure

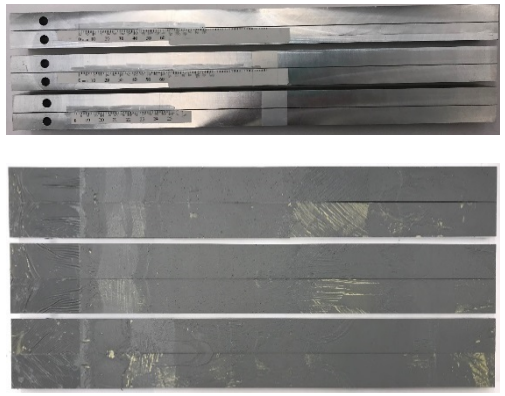
Preliminary Test Results

EA 9394 – D3433 – Property Value – Fracture Toughness (lbf/in)



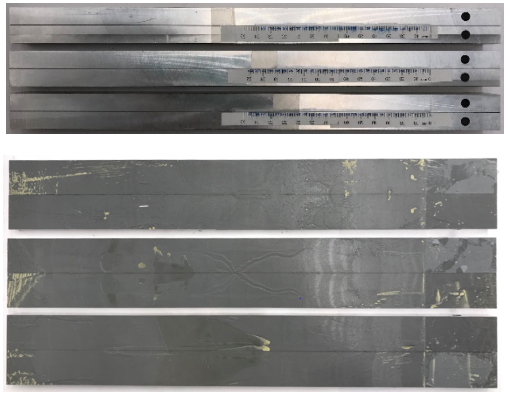
Batch A

Cure Cycle 1



Cohesive Failure

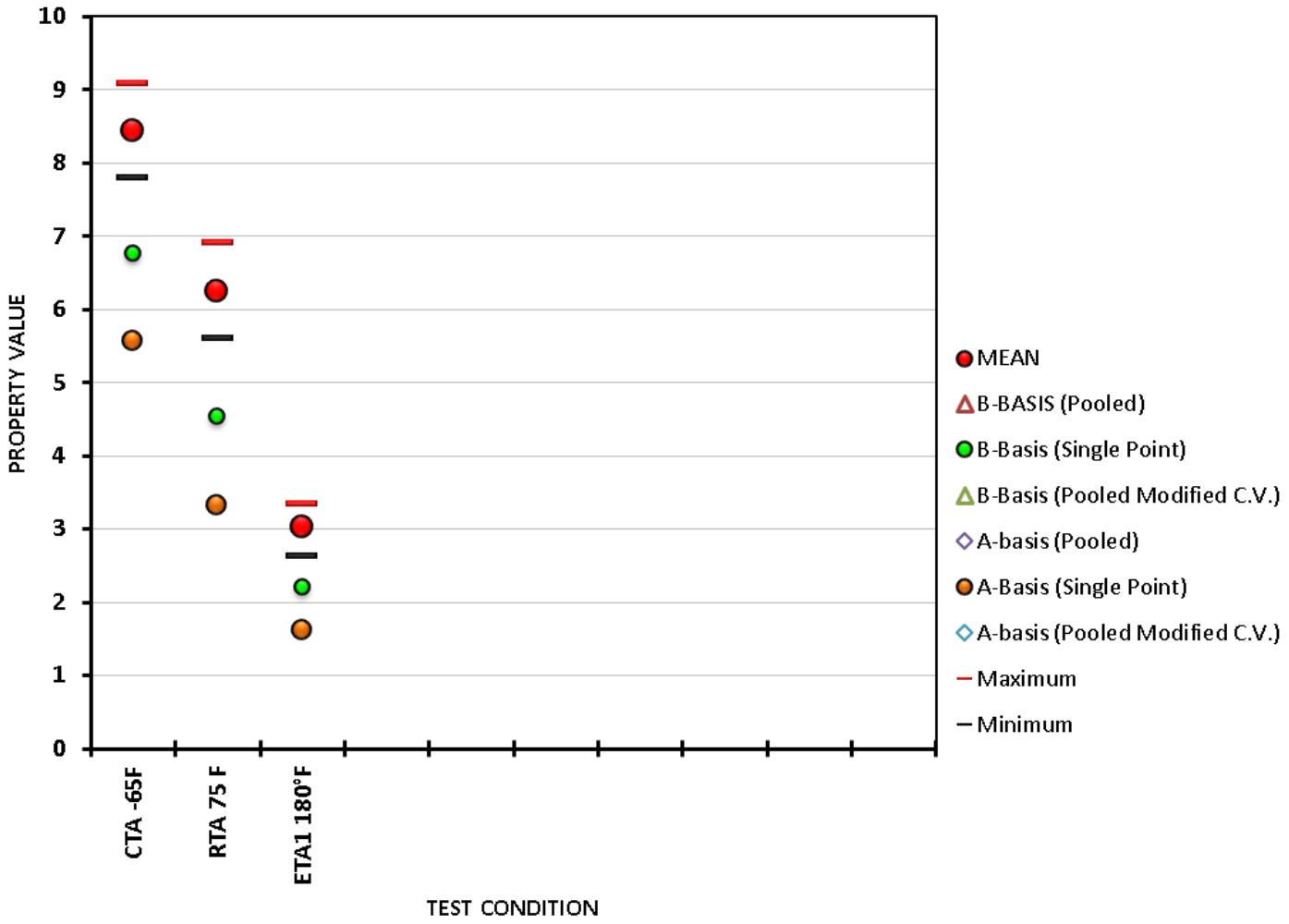
Cure Cycle 2



Cohesive Failure

Preliminary Test Results

EA 9394 – D5656 (T1) – Property Value – Shear Strength (ksi)



T1

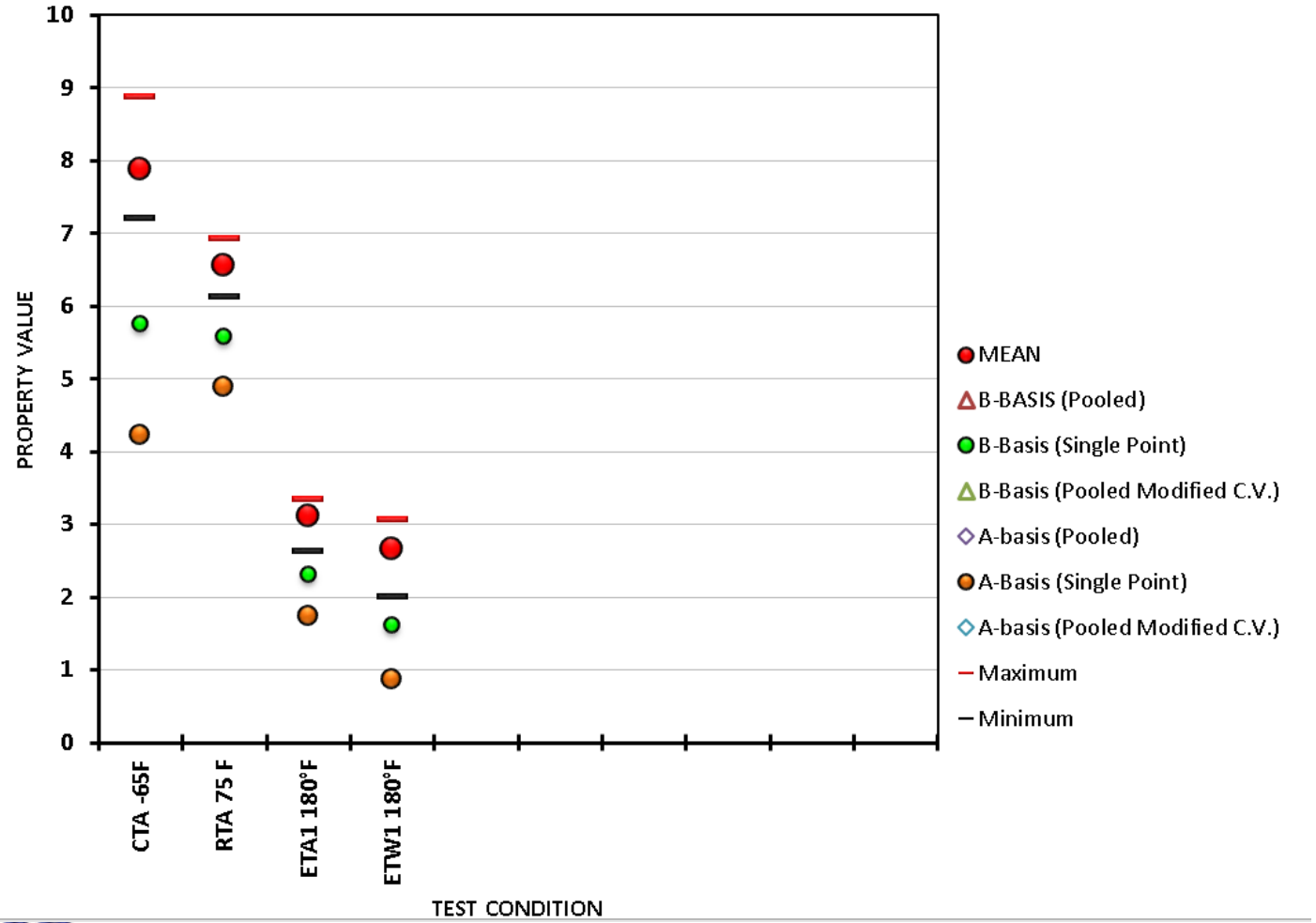
Batch A

Cure Cycle 1 Cure Cycle 2

Cohesive / Adhesive

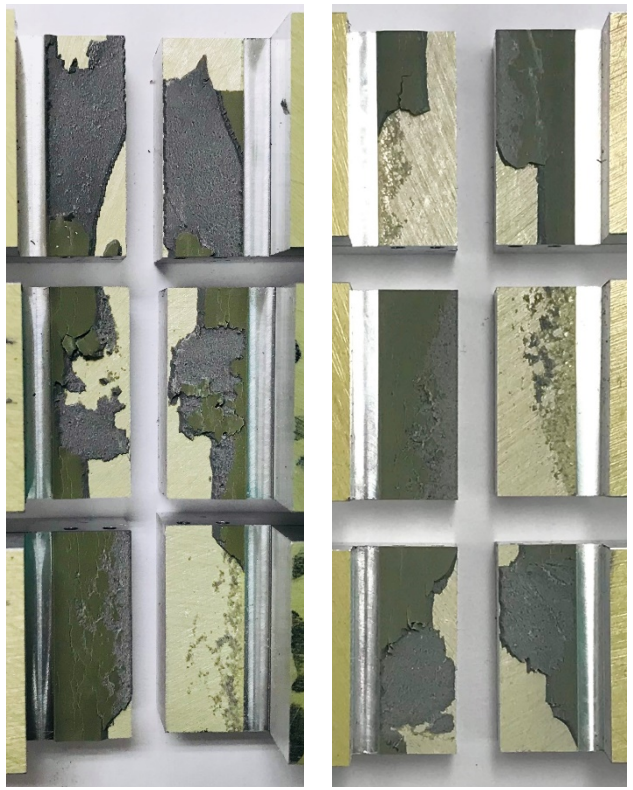
Preliminary Test Results

EA 9394 – D5656 (T2) – Property Value – Shear Strength (ksi)



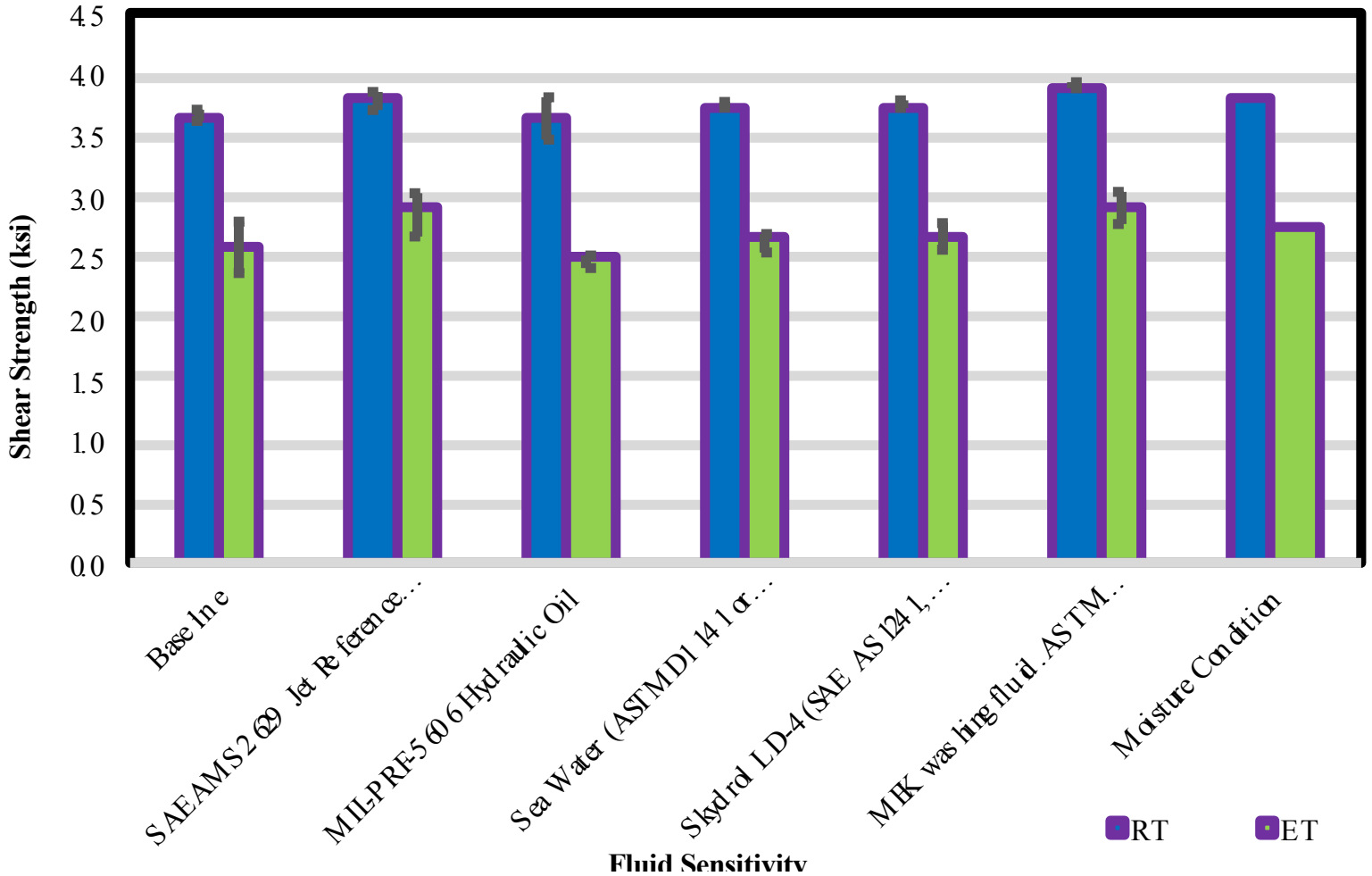
T2
Batch A

Cure Cycle 1 Cure Cycle 2



Cohesive / Adhesive

Preliminary Test Results
EA 9394 – D1002 – Fluid Sensitivity Test Results



Current Status

EA 9394

Property	Test Method	Test Environment	EA9394					
			Batch A		Batch B		Batch C	
			C1	C2	C1	C2	C1	C2
Thin Metal Adherend Lap Shear	D1002	CTD	3	3	3	3	3	3
		ETD	3	3				
		ETW	3	3	3	3	3	3
		RTD	3	3	3	3	3	3
Thick Metal Adherend Lap Shear	D5656 (T1)	CTD	3	3				
		ETD	3	3				
		ETW	3	3				
		RTD	3	3				
Thick Metal Adherend Lap Shear	D5656 (T2)	CTD	3	3				
		ETD	3	3				
		ETW	3	3				
		RTD	3	3				
Composite Adherend Lap Shear	D3165	CTD	3	3	3	3	3	3
		ETD	3	3				
		ETW	3	3	3	3	3	3
		RTD	3	3	3	3	3	3
Mode I Fracture Toughness	D3433	CTD	3	3				
		ETD	3	3				
		ETW	3	3				
		RTD	3	3				
Mode II Fracture Toughness	D7905	CTD	3	3				
		ETD	3	3				
		ETW	3	3				
		RTD	3	3				
Floating Roller Peel	D3167	CTD	3	3	3	3	3	3
		ETD	3	3				
		ETW	3	3	3	3	3	3
		RTD	3	3	3	3	3	3
Flatwise Tensile	D897	CTD	3	3	3	3	3	3
		ETD	3	3				
		ETW	3	3	3	3	3	3
		RTD	3	3	3	3	3	3
Fluid Sensitivity	D1002FS	ET	30					
		RT	30					

FM300-2M

Property	Test Method	Test Environment	FM300-2M					
			Batch A		Batch B		Batch C	
			C1	C2	C1	C2	C1	C2
Thin Metal Adherend Lap Shear	D1002	CTD	3	3	3	3	3	3
		ETD	3	3				
		ETW	3	3	3	3	3	3
		RTD	3	3	3	3	3	3
Thick Metal Adherend Lap Shear	D5656	CTD	3	3				
		ETD	3	3				
		ETW	3	3				
		RTD	3	3				
Composite Adherend Lap Shear	D3165	CTD	3	3	3	3	3	3
		ETD	3	3				
		ETW	3	3	3	3	3	3
		RTD	3	3	3	3	3	3
Mode I Fracture Toughness	D3433	CTD	3	3				
		ETD	3	3				
		ETW	3	3				
		RTD	3	3				
Mode II Fracture Toughness	D7905	CTD	3	3				
		ETD	3	3				
		ETW	3	3				
		RTD	3	3				
Floating Roller Peel	D3167	CTD	3	3	3	3	3	3
		ETD	3	3				
		ETW	3	3	3	3	3	3
		RTD	3	3	3	3	3	3
Flatwise Tensile	D897	CTD	3	3				
		ETD	3	3				
		ETW	3	3				
		RTD	3	3	3	3	3	3
Fluid Sensitivity	D1002FS	ET	30					
		RT	30					

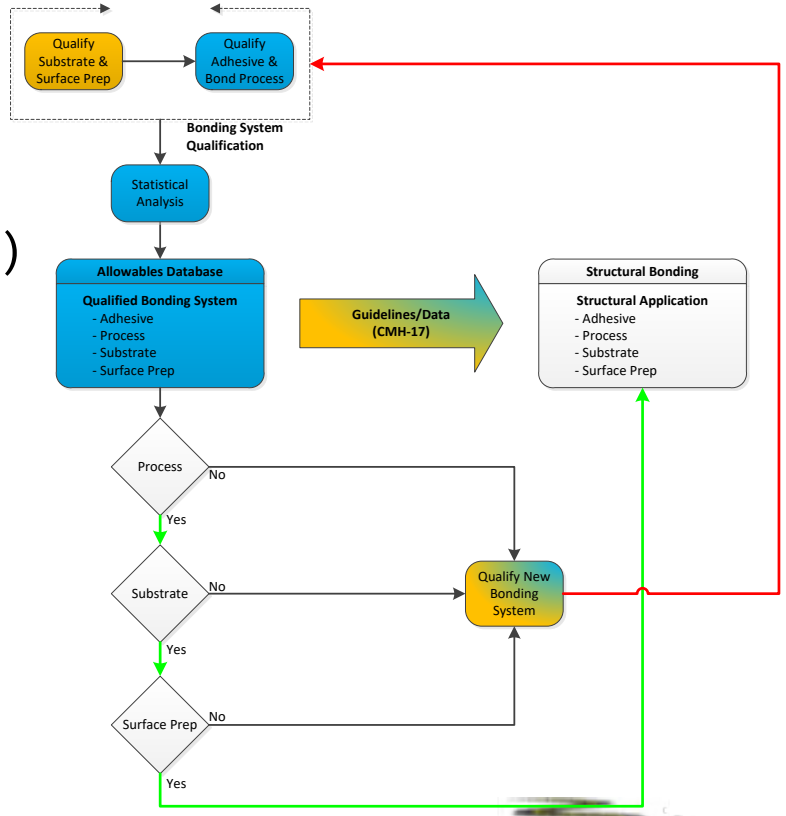
- Waiting on Adhesive Batch
- Specimen/Panel Bonding in Progress
- Specimen Machining in Progress
- Conditioning in Progress
- Testing in Progress
- Testing Complete

Look Forward

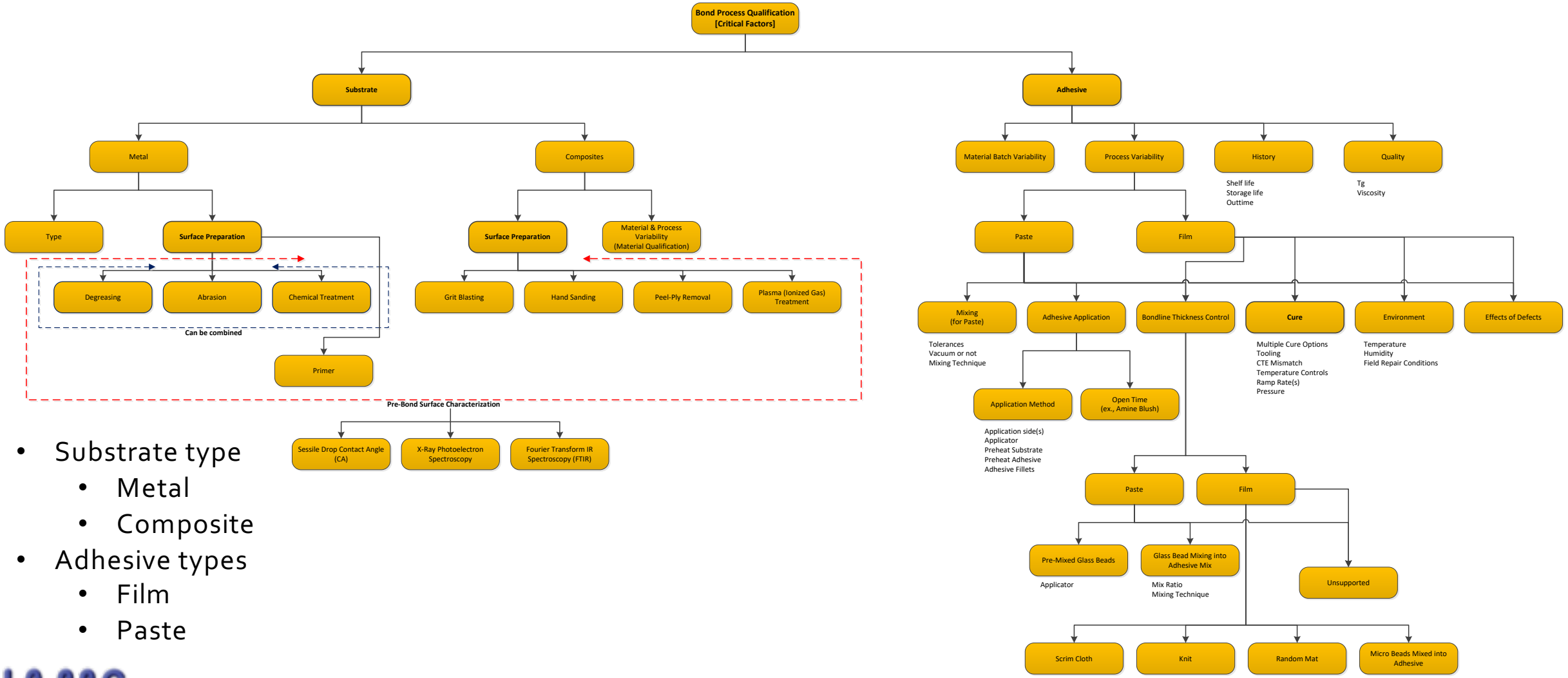
- **Future Activities**
  - Generate the B-Basis allowable for EA9394 and FM300-2M material systems
  - Focus on performing equivalency on adhesive materials.
  - Analyze failure modes for different test environments and report them accordingly
- **Benefit to the Aviation Community**
  - Guidance on test matrices for mechanical, physical and chemical characterization of adhesives
  - Generate adhesive material databases under NCAMP protocols that can be used for a wide variety of applications be different end users

Bond Process Qualification Protocols - Road Map

- Bond Process Qualification (BPQ)
  - Develop an acceptance criteria
    - Requirements (based on information in AC's and FAR's , etc.)
    - Applicability of existing standards and/or develop new standards
  - Select known bond system failures
    - Simulate and investigate the BPQ methodology flags the "bad" bonds
  - Develop protocols
    - Quantify process reliability
    - Assess repeatability/maturity



Bond Process Qualification (Critical Factors)



- Substrate type
  - Metal
  - Composite
- Adhesive types
  - Film
  - Paste

Summary of Activities

- Current Activities
  - Task 1 – Substrate and adhesive compatibility
  - Task 2 – Use of peel ply for composite substrate preparation
- Completed Activities
  - Effects of Mix-ratio in two part paste adhesives
  - Evaluation of assembly time in paste adhesives
    - Amine blush effects
  - Fluid Sensitivity of adhesive
  - Efficient adhesive screening method testing.

Qualification of a Bond Process

Substrate + **Adhesive System** + **Substrate Surface Preparation** + **Bonding Process/Curing**

1. Composite
2. Metal

1. Paste
2. Film

1. Abrasion
   1. Grid Blasting
   2. Hand Sanding
2. Peel Ply
   1. Nylon
   2. Polyester
3. Chemical
   1. PAA + Priming
4. Other
   1. Plasma Treatment

1. Cure Cycle – Compatibility with Substrate
2. Vacuum or Pressure
3. Bondline Control
4. Assembly Time
5. Adhesive Preparation
6. Environment
7. Bond Configuration – Co-bond/Secondary bond

Qualification of a Bond Process

Task 1

Substrate	Adhesive System	Substrate Surface Preparation	Bonding Process/Curing

1. Composite
2. Metal
	1. Paste
	2. Film
		1. Abrasion
		  1. Grid Blasting
		  2. Hand Sanding
		2. Peel Ply
		  1. Nylon
		  2. Polyester
		3. Chemical
		  1. PAA + Priming
		4. Other
		  1. Plasma Treatment
			1. Cure Cycle – Compatibility with Substrate
			2. Vacuum or Pressure
			3. Bondline Control
			4. Assembly Time
			5. Adhesive Preparation
			6. Environment
			7. Bond Configuration – Co-bond/Secondary bond

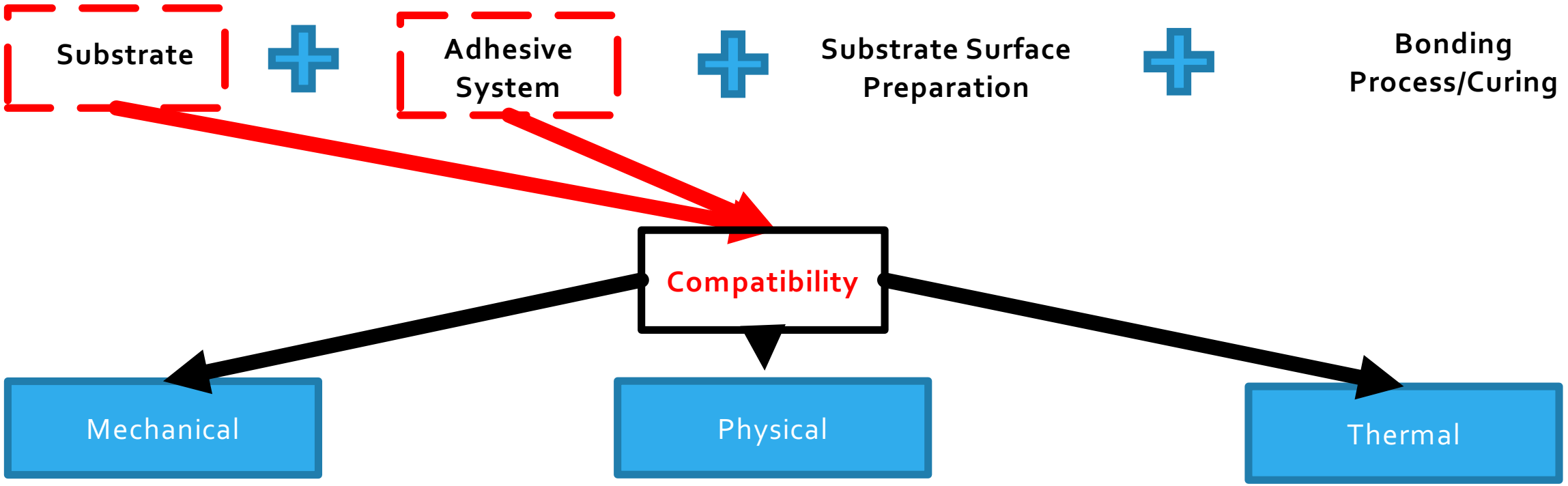
Task 2

Task 1 –Qualification of a Bond Process – Substrate and Adhesive Compatibility - Background and Goals

- When using bonded joints for primary or secondary structure applications, there is a wide variety of substrates and adhesive materials that are available for use. **Providing Guidance on selecting a compatible substrate and a adhesive combination** is important for designers.
- Main factors to consider during adhesive and substrate selection are the **mechanical property requirements**, **physical compatibility** of the substrates (hybrid and non-hybrid) and adhesives for bonding, **thermal compatibility** of the bond system during the bonding process and service life.
- Objective of this task is to provide establish a set of guidelines to use when selecting an adhesive and substrate combination for a given bond process.

Goal – **Develop guidelines on how to select compatible substrate and adhesive combinations to obtain a robust bond system**

Task 1 - Qualification of a Bond Process – Substrate and Adhesive Compatibility



1. Material allowable properties
   1. Substrate data -> NCAMP
   2. Adhesive data -> NCAMP (in progress)
2. Joint mechanical capability
   1. Static
   2. Durability

1. Surface Characterization
   1. Surface roughness
   2. Surface energy/ Contact angle
   3. Wettability envelope

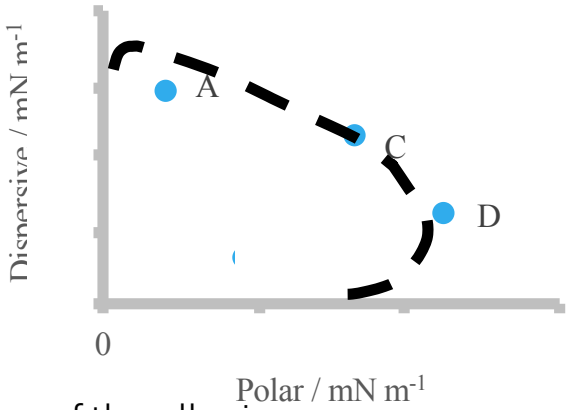
1. CTE Mismatch
2. Glass Transition
3. Cure Cycle Compatibility
   1. Secondary bond
   2. Co-bonded

Task 1 - Substrate and Adhesive Compatibility

- Substrates
  - Carbon Fiber Composites
    - UNI – T800/3900-2
    - PW - T300/3900-2
  - Glass Fiber Composites
    - Fabric – Epoxyglass G10
  - Metallic
    - AL 2024-T3
    - Ti – Grade 2
- Adhesives
  - Paste Adhesives
    - Henkel EA9394
    - Henkel EA9390
    - Cytec 680-3
  - Film Adhesive
    - Cytec FM300-2M
    - 3M-AF163
- Cure Cycles
  - Manufacturer recommended cure cycle
- Surface Preparation Methods
  - Carbon fiber composites
    - As fabricated – MEK wipe only
    - Peel ply only
    - Peel ply + Light abrasion (180 grit)
    - Abrasion only (120 grit)
  - Glass fiber composites
    - As fabricated
    - Abrasion only(120 grit)
  - Metallic substrates
    - MEK wipe only
    - PAA+BR127
    - 3M - AC 130-2 surface treatment
    - Abrasion
    - Chemical treatment – ASTM D2651 (Ti)

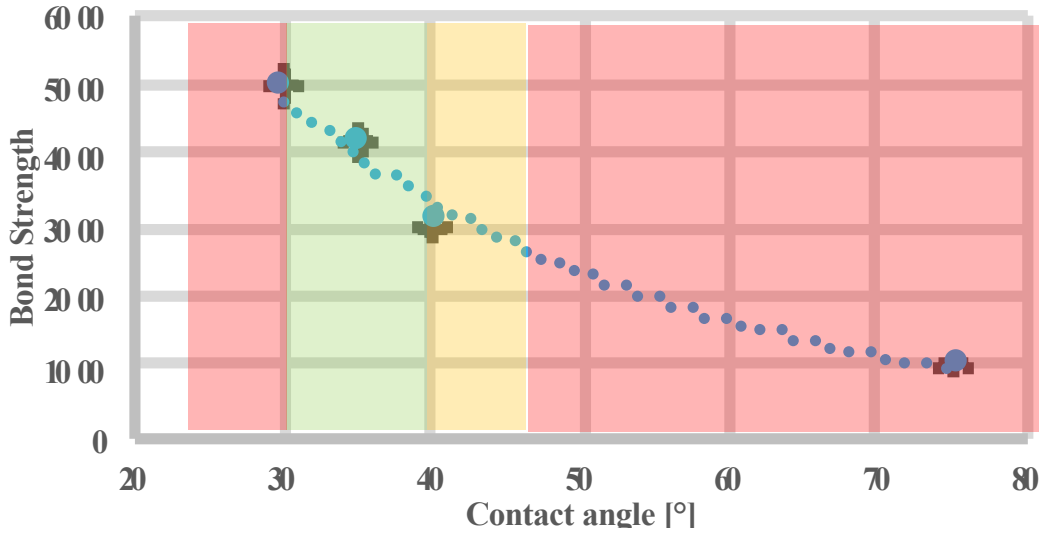
Task 1 - Substrate and Adhesive Compatibility Assessment
Physical Compatibility

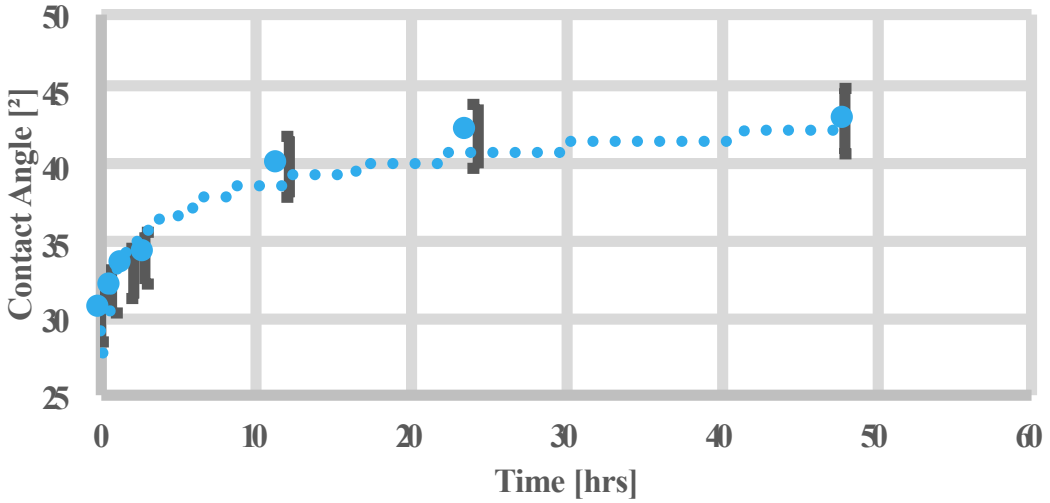
- Objective
  - **Generate guidelines to ensure the surface preparation + substrates are physically compatible for bonding.**
- Physical Compatibility
  - Surface morphology related tests for substrates
    - Surface roughness
    - Contact angle
    - Surface Energy
  - Wettability envelope development
    - Measure polar and dispersive surface energies for substrate and compare the surface energy of the adhesive.
    - Objective:- A simplistic rapid approach to evaluate if the adhesive surface tension falls within the wettability envelope == Good bond
  - Perform qualitative tests to assess the bond failures
    - Wedge crack
    - Rapid adhesion test
    - Flatwise tensile



Task 1 - Substrate and Adhesive Compatibility Assessment
Physical Compatibility

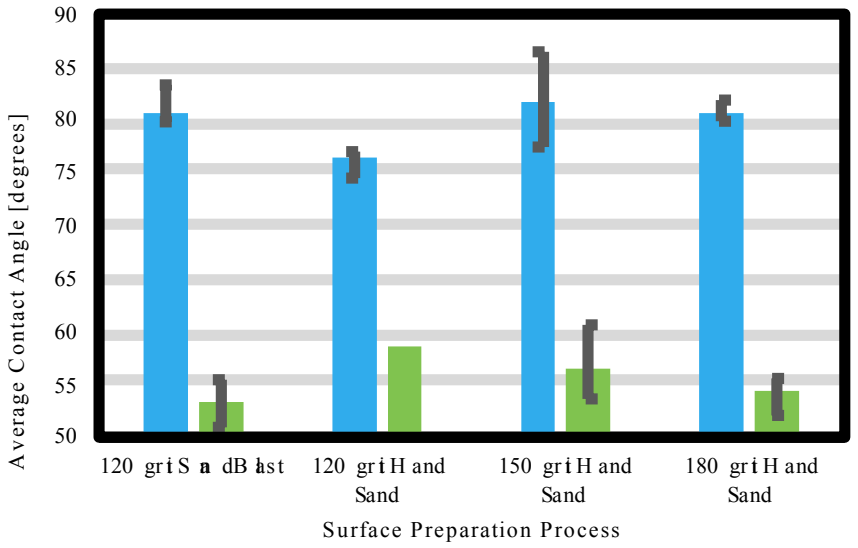
- Develop the relationship between the
  - Contact angle of substrates vs. bond quality.
  - Elapsed time vs contact angle
- Use in-situ surface energy measurement techniques (BTG-Labs - Surface Analysts) to assess the substrate characterizations
- Repeat the process for a given adhesive for different mechanical properties (Shear, Peel, Fracture Toughness)

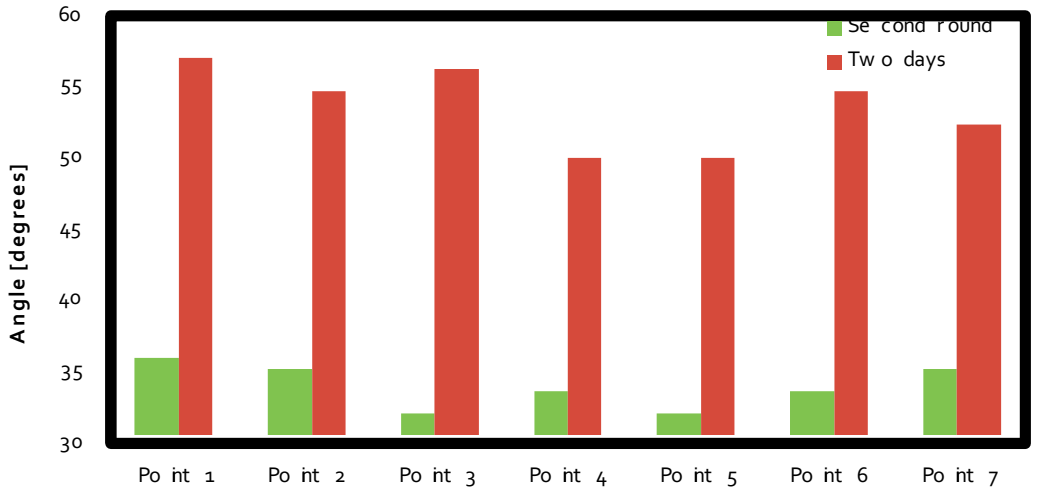




Task 1 - Substrate and Adhesive Compatibility Assessment
Physical Compatibility – Current Status

- Fabrication of composites test panels completed.
- Preliminary experiments completed for surface energy/contact angle measurements
- Guideline development for substrate preparation timeline is in progress





Task 1 - Substrate and Adhesive Compatibility Assessment
Thermal Compatibility

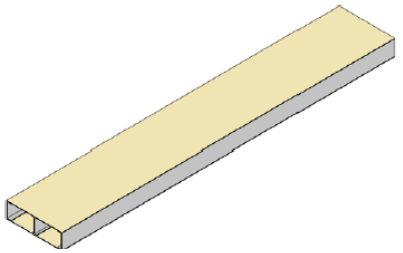
- Objective
  - **Generate guidelines to ensure the thermal properties of substrates and adhesives are compatible for bonding and during service life.**
- Thermal Compatibility
  - CTE mismatch
    - Between substrates and adhesives cured at elevated temperatures
    - CTE mismatch in bonded structures during service life – Cold and elevated temperature environments – formation of micro cracks during thermal cycling
  - Glass Transition Temperatures
    - Mismatch in glass transition temperatures and out it could potentially effect the bond integrity
  - Pose cure effect on the substrates (composites) for secondary bonded structures
    - Understand how the critical mechanical and thermal properties change after exposing to high temperatures for long durations (cure profile of a adhesive)

In - Progress

Substrate and Adhesive Compatibility Assessment
Mechanical Properties

- Mechanical Properties
  - Coupon level testing
    - Perform coupon level testing to evaluate static and durability capability. (using the actual bond process that will be used for the application)
      - Shear
      - Peel
      - Fracture toughness
  - Element/Component level testing
    - Fabricate a representative bonded structures
    - Perform mechanical testing (static and fatigue)
    - Assess the bond quality / Perform NDI

Future Activities



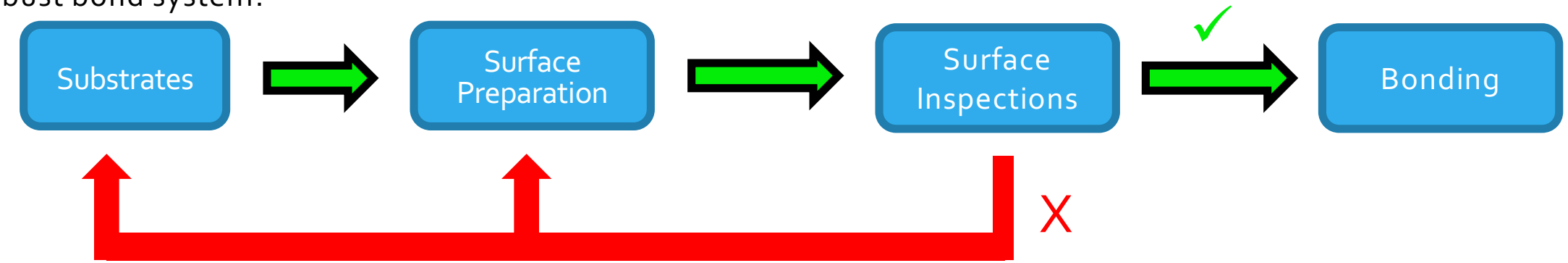
Task 1 - Substrate and Adhesive Compatibility Additional Tasks

- Extend current research with industry partners to further investigate substrate adhesive compatibility.
- Substrate Material – Tencate T350-1/IM7
- Adhesive System – EA 9394

Test Type	Test Method	Bondline Thickness [in]	CTD		RTD		ETD		ETW	
			#Batch	#Spec.	#Batch	#Spec.	#Batch	#Spec.	#Batch	#Spec.
Single Lap Shear	ASTM D3165 - Composites	0.015	1	6	1	6	1	6	1	6
		0.04	3	18	3	18	3	18	3	18
		0.08	1	6	1	6	1	6	1	6
		0.125	1	6	1	6	1	6	1	6
Single Lap Shear	ASTM D1002 - (Al -thin)	0.015	1	6	3	18	1	6	1	6
Lap Shear and Stress/Strain	ASTM D5656 - (Al – thick)	0.015	1	6	3	18	1	6	1	6
T-Peel	ASTM D1876 - Composites	0.015	1	6	3	18	1	6	1	6
Floating Roller Peel	ASTM D3167	0.015	1	6	3	18	1	6	1	6
Fracture Toughness	ASTM D3433	0.015	1	6	3	18	1	6	1	6
Flatwise Tensile	ASTM D897	0.015	1	6	3	18	1	6	1	6

Task 2 - Peel Ply Surface Preparation Evaluation Background and Goals

- Use of **peel ply** as a surface preparation method reduces the amount of labor involved and simplify the substrate preparation process. It also provides a uniform and repeatable surface for bonding
- Peel ply prepared surface quality vary on many **substrate and surface preparation process parameters**. Bond surface quality directly effects the bond integrity. Understanding the effects of these parameters is critical. **Development of reliable and rapid inspection methods** is crucial to ensure the bond process (surface preparation) method is appropriate for a given bond system.
- After an appropriate peel ply surface preparation method is chosen, there are many **other parameters** associated with handling substrates that could potentially change the quality of the bond surfaces. These parameters and their **adverse effect** on the bond integrity needs to be evaluated to provide **Guidance and Develop Protocols** to have a robust bond system.



Goal – **Develop guidelines and protocols to handle peel ply prepared surfaces to obtain a robust bond system**

Peel Ply Surface Preparation Evaluation

- Peel ply removal preparation method provides a repeatable uniformly prepared surface for bonding with, minimum labor.
  - For guideline development, need to understand
    - The effect of different **peel ply materials** and **thicknesses**
    - **Surface contamination** created and ways to reduce it (during application and removal of peel ply and the timeframe of removal)
    - **Rapid inspection methods** to ensure the surface quality of the substrates
    - Peel ply prepared surface exposure to **extreme environments** (hot/wet)
    - Any **adverse effects to the laminate** due to having the peel ply during cure cycle.
    - Effects of peel ply prepared surfaces going through **multiple cure cycles**.

Peel Ply Surface Preparation Evaluation

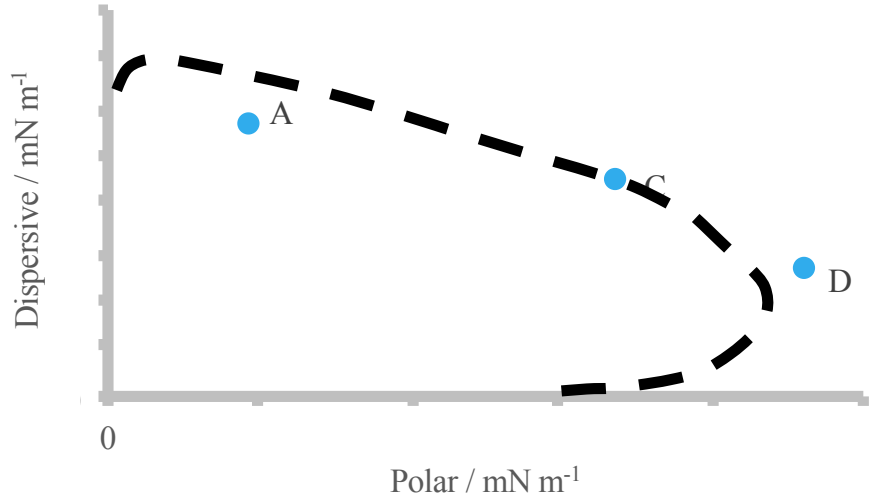
- Types of Peel Ply
  - Polyester (Non Released)
    - Wet (Henkel EA 9895)
    - Dry
      - T1 – 60002 (0.005 – 0.006-inch)
      - T2 – 60005 (0.006 – 0.007 –inch)
  - Nylon (Non Released)
    - Wet (Henkel EA 9896)
    - Dry
      - T1 – 60004 (0.0045 – 0.0055-inch)
      - T2 – 60005 (0.006 – 0.007 –inch)
  - Polyester (Released)
    - Dry
      - T1 – 60001 (0.005 – 0.006-inch) (SRB)
- Carbon Composites Substrates
  - Toray T800/3900-2
- Adhesive Systems
  - Film – FM300-2M
  - Paste – EA 9394
- Cure Cycles
  - FM300-2M – 250F for 2 hrs. at 40 psi pressure + full vacuum
  - EA 9394 – 150F for 1 hr. 6psi vacuum

Peel Ply Surface Preparation Evaluation

- Surface Preparation Details
  - Peel ply removal only
  - Peel ply removal and light sanding (120 grit)
- Exposure to environment (Room temperature ambient and Elevated temperature wet)
  - Remove peel ply immediately after curing
  - Remove peel ply immediately before bonding (30 days)
- Effects of prepared substrates going through multiple cure cycles
  - Co-bond and repair applications
  - Thermal cycle substrates for multiple times to evaluate the effects
- Controlled/non peel ply configuration
  - Carbon epoxy laminates without peel ply
  - Hand Abrasion (120 grit)
  - No surface preparation (MEK wipe only)

Peel Ply Surface Preparation Evaluation
Methods of Bond Surface Quality Assessment

- Surface Characterization
  - Surface roughness measurements
  - Contact angle measurements
  - Scanning electron microscopic (SEM) inspection for surface details
  - X-ray photoelectron spectroscopy (XPS) to detect surface contamination
- Wettability Envelope Development



Peel Ply Surface Preparation Evaluation
Methods of Mechanical and Physical Property Evaluation

- Fiber Volume Fraction Quantification
  - Due to the resin absorption in peel plies, fiber volume fraction is affected
  - Flatwise tensile testing to quantify the effect.
- Mechanical Property Assessment
  - ASTM D1002/D3165 type Single lap shear to determine the shear strength
  - ASTM D5528 to determine the fracture toughness properties
- Peel Ply Prepared surfaces going through multiple cure cycles
  - Measure the degree of cure for repeated cure cycle – simulation of core bond and repair applications.

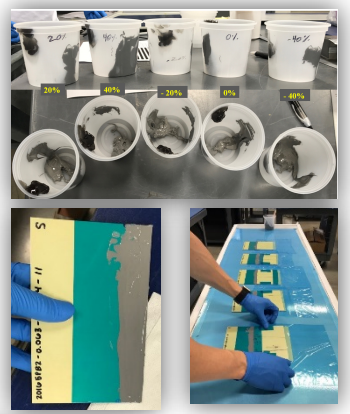
Summary of Activities

- Current Activities
  - Task 1 – Substrate and adhesive compatibility
  - Task 2 – Use of peel ply for composite substrate preparation
- Completed Activities
  - Effects of mix-ratio in two part paste adhesives
  - Evaluation of assembly time in paste adhesives
    - Amine blush effects
  - Fluid sensitivity of adhesive
  - Efficient adhesive screening method testing.

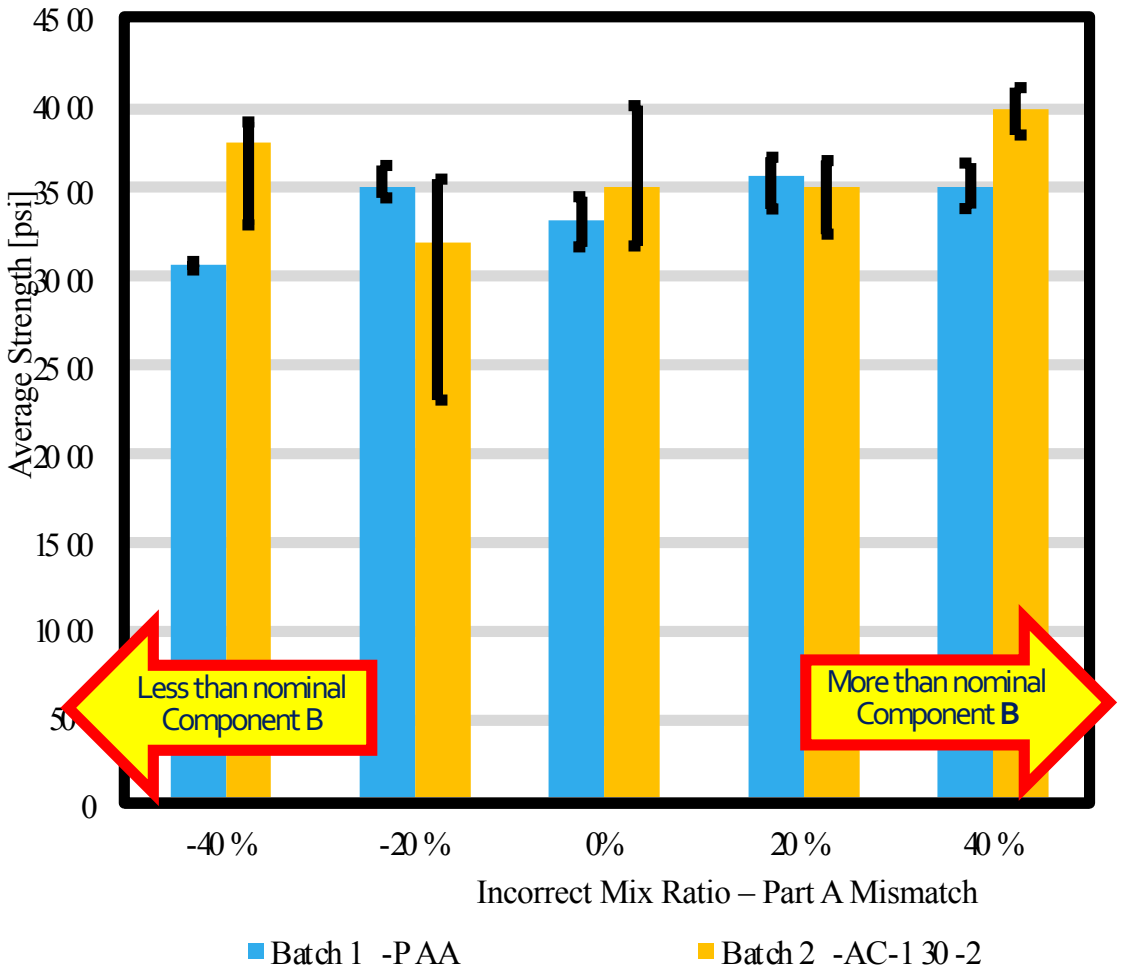
Effects of incorrect mix-ratio in two part paste adhesives

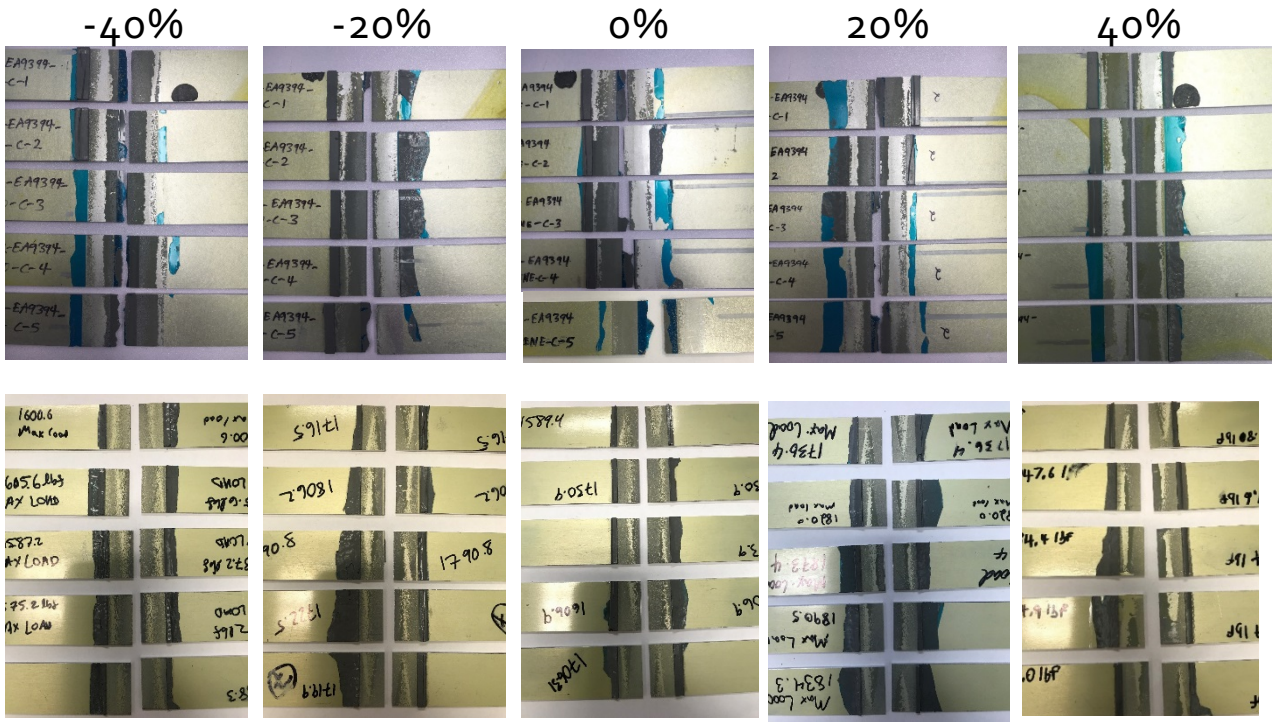
- Two part adhesive for smaller quantities are available in cartridge form. (Mix ratio is not a concern) For applications that require larger quantities, common method is to obtain them in separate containers and manually mix it. It is important to evaluate the sensitivity of mix ratio in these applications
- Experimental Approach – used PAA+BR127 and Abrasion + AC120-2 prepared aluminum and carbon composite substrates and fabricated panels with different mix ratios for Part A and part B. Test methods evaluated are D1002 – single lap shear, mode I fracture toughness, and floating roller peel specimens (selected incorrect mix ratios).
- EA 9394 was used for the study with Part A mix ratio error ranging from -40% to +40%

Adhesive Identification	Adherend Material	Percent Mismatch	Adhesive Quantity A [g]	Adhesive Quantity B [g]	0.005" Glass Bead Weight [g]
EA9394	Aluminum 0.063"	-40%	30.000	3.060	0.00331
		-20%	30.000	4.080	0.00341
		0%	30.000	5.100	0.00351
		20%	30.000	6.120	0.00361
		40%	30.000	7.140	0.00371



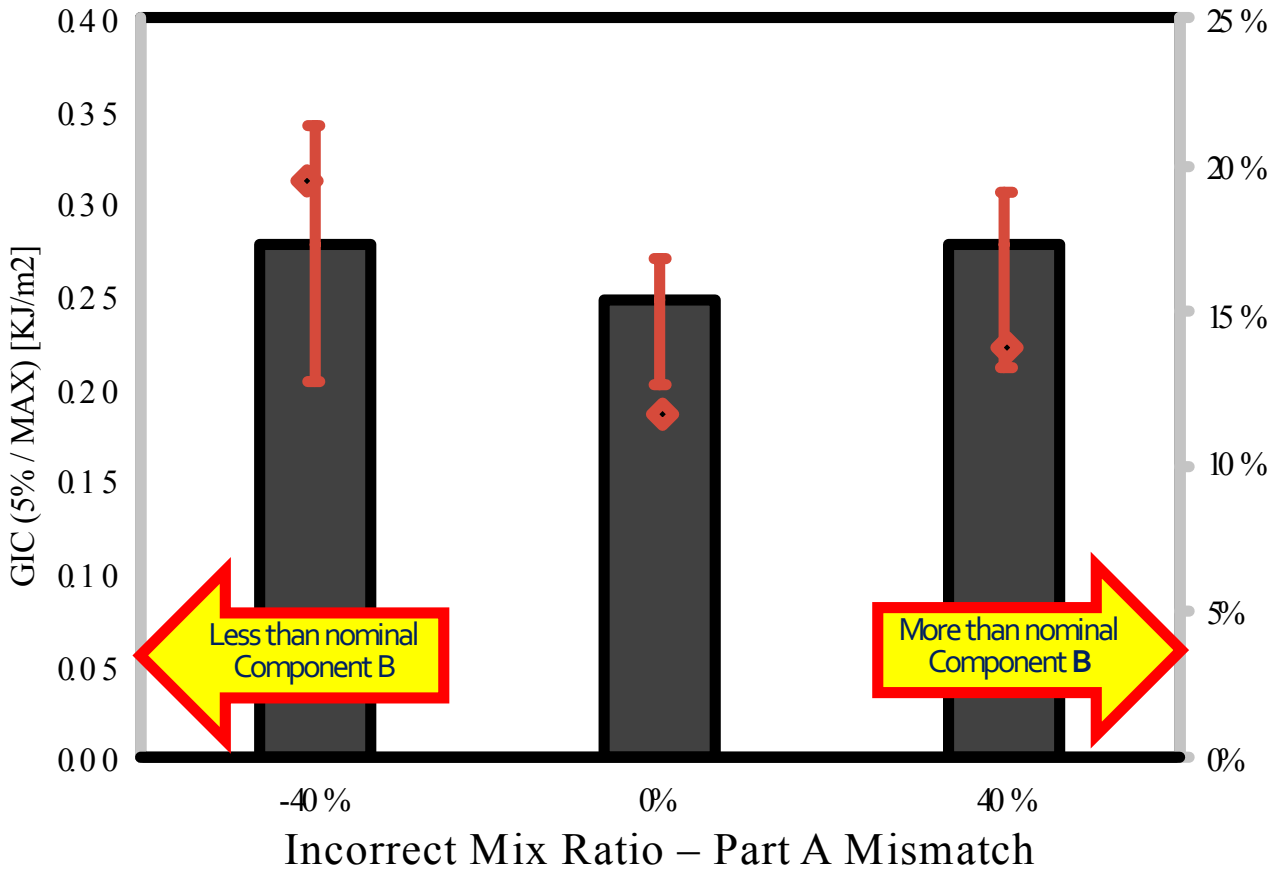
Effects of incorrect mix-ratio in two part paste adhesives
Test Results – Single Lap Shear – ASTM D1002

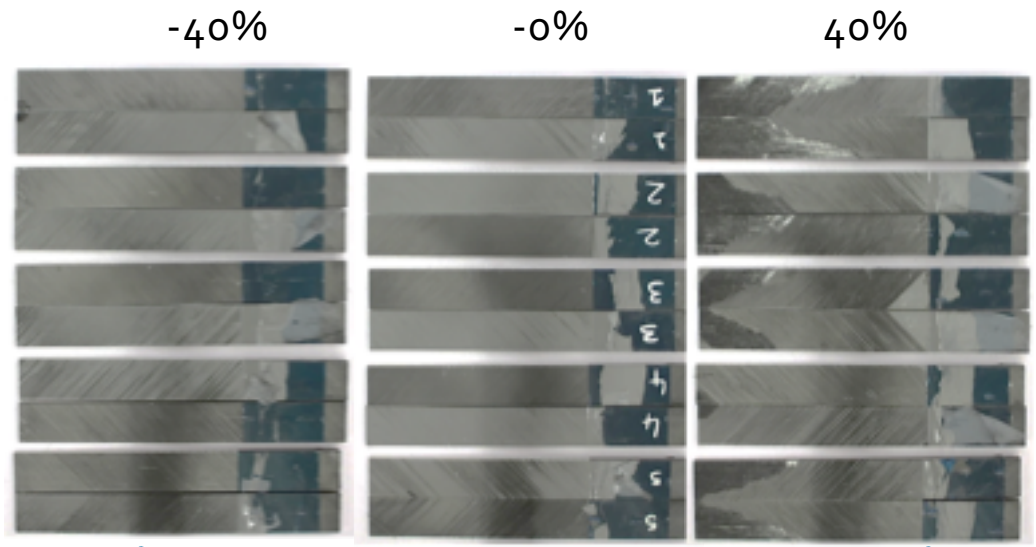




Adhesive/Cohesive Failures

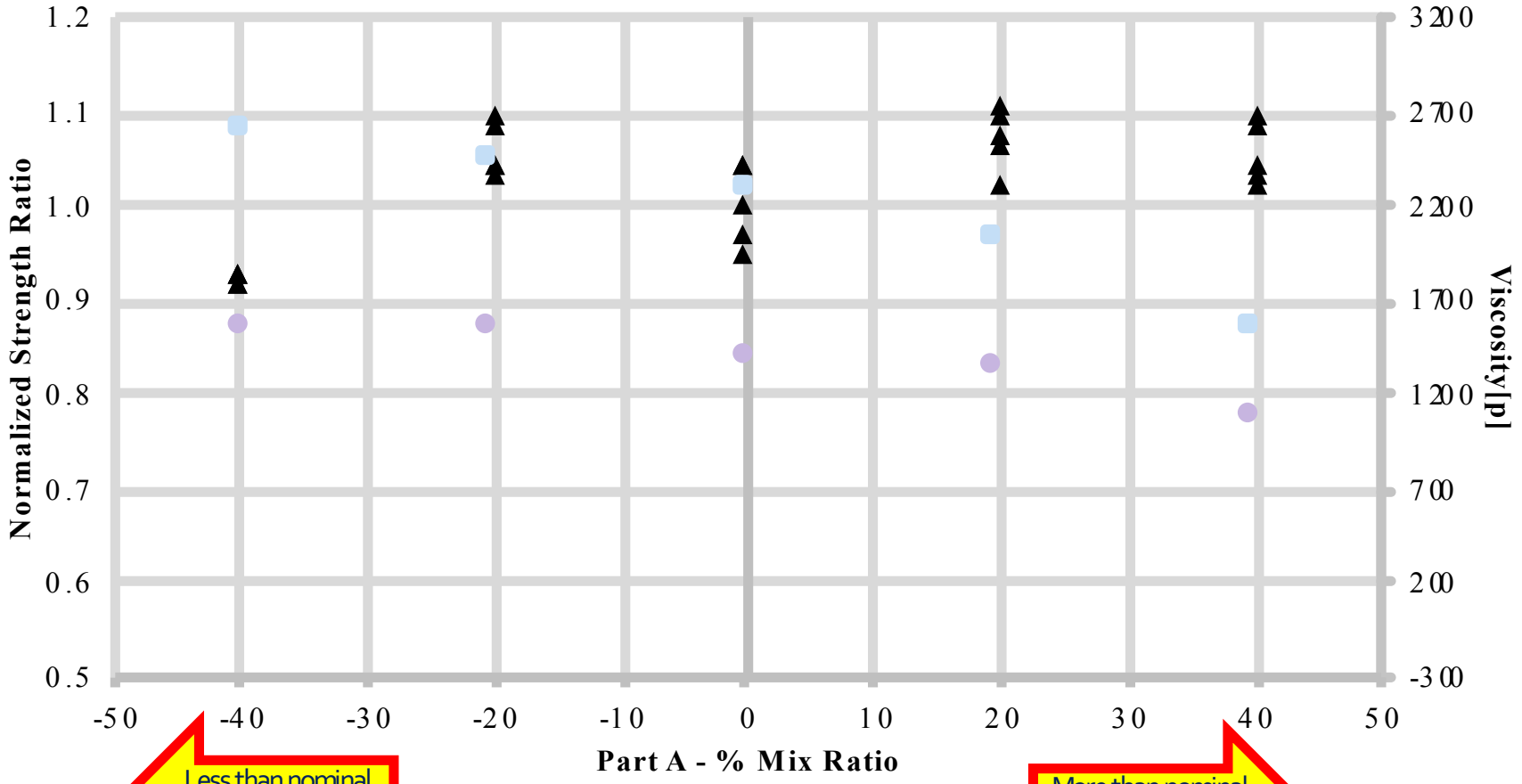
Effects of incorrect mix-ratio in two part paste adhesives
Test Results – Mode I – ASTM D5528





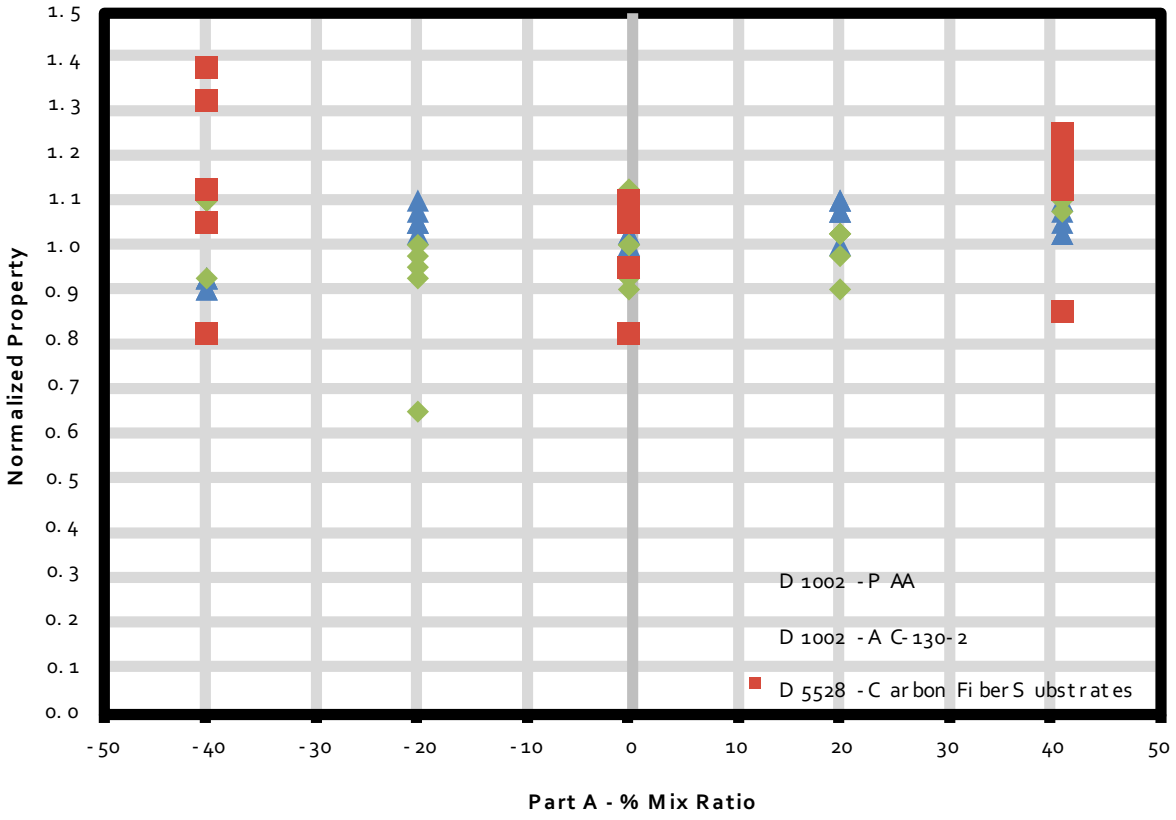
First Ply/Adhesive Failures

Effects of incorrect mix-ratio in two part paste adhesives
Test Results – Viscosity Response



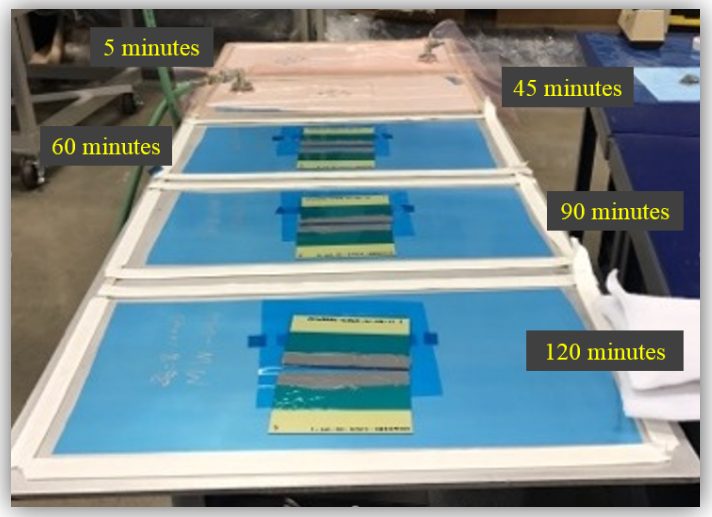
Effects of incorrect mix-ratio in two part paste adhesives
Test results – Summary

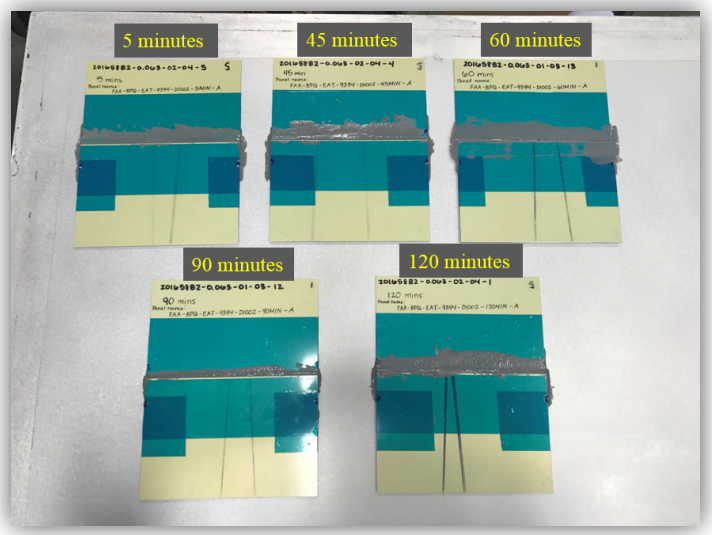
- EA9394 adhesive system showed a ~±15% change in the mechanical properties of single lap shear and fracture toughness at the extreme mix ratios between -40% & +40%
- Repeatability of the experiment was validated with a second data set. Data correlates will with the original testing.
- No change in the failure modes was seen between the extreme ends of the experimental procedure.
- Static response of the properties are desirable. However, understanding of the mix ratio effect on fatigue properties needs to be investigated.



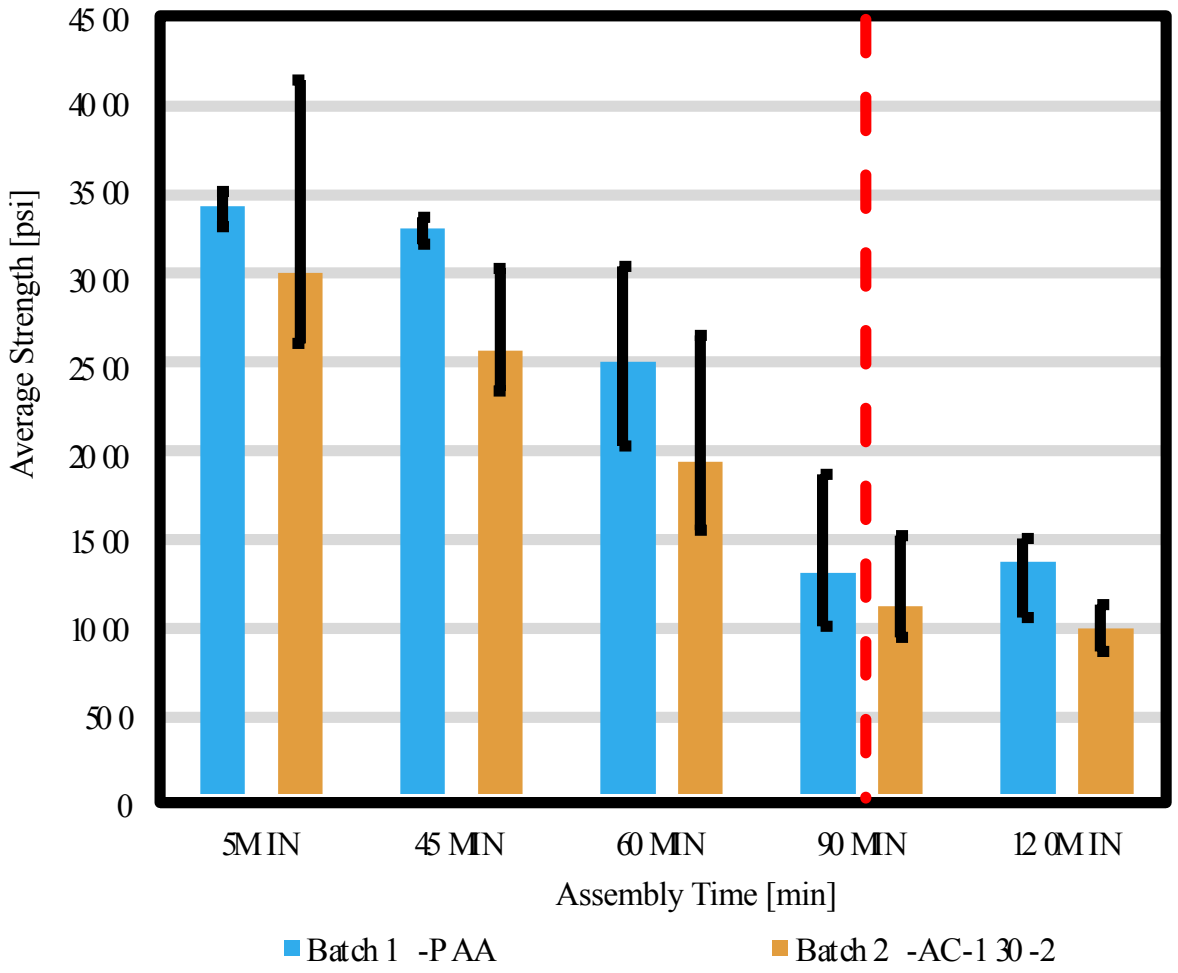
Evaluation of assembly time in paste adhesives

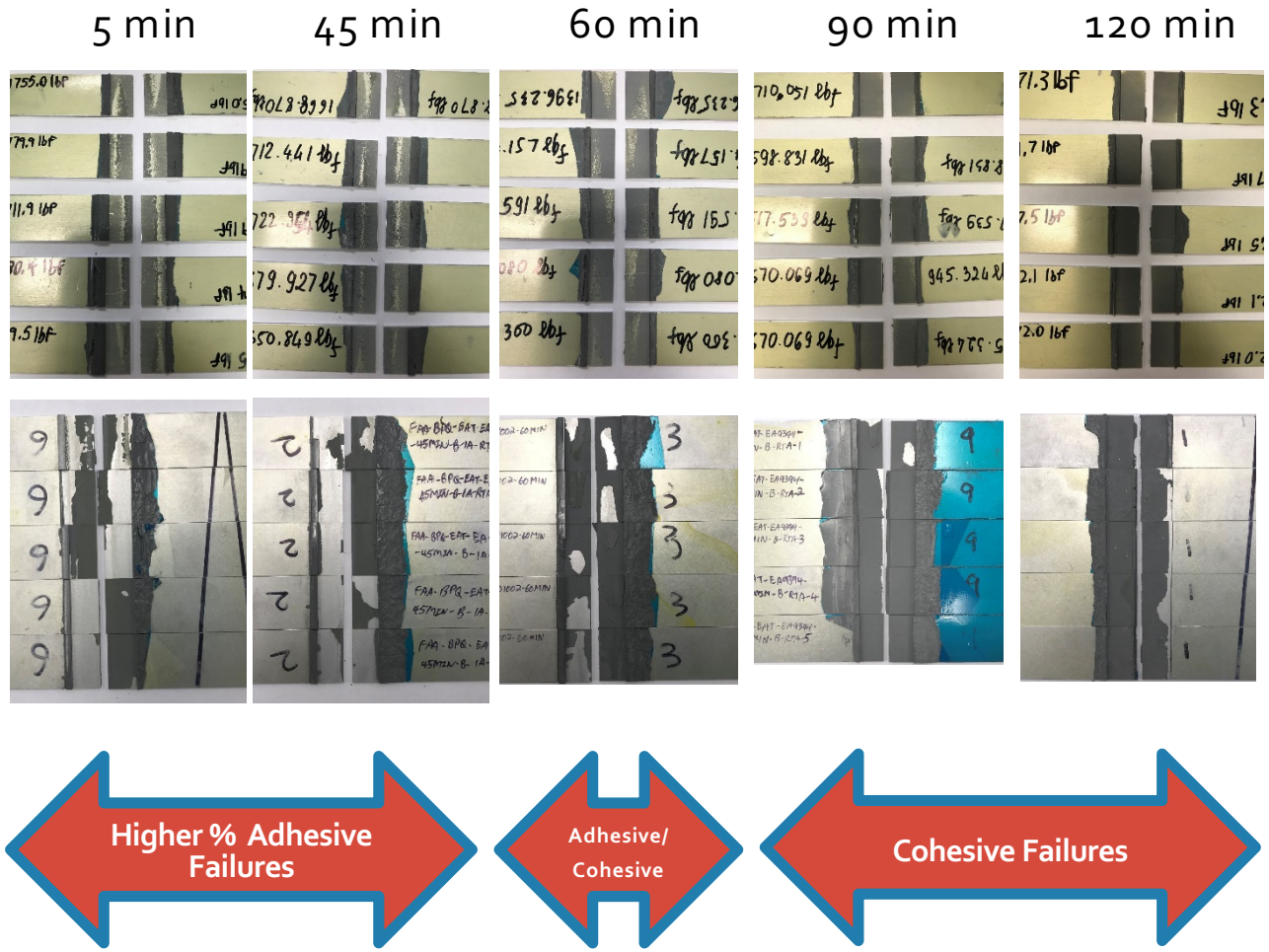
- Manufacturer provided pot life is to be used as a material specification. In bonding applications, assembly time is defined as the time it takes to mix, apply adhesive and mate the two parts together. Depending on the bond area and the complexity (contour) of the structure, this could be a critical parameter.
- Experimental Approach – used PAA+BR127 and Abrasion + AC120-2 prepared aluminum and carbon composite substrates and fabricated panels with different assembly times. Test methods evaluated are D1002 – single lap shear, mode I fracture toughness, and floating roller peel specimens (selected incorrect mix ratios).
- Assembly time for EA 9394 was varied from 0, 5, 45, 60, 90, and 120 minutes



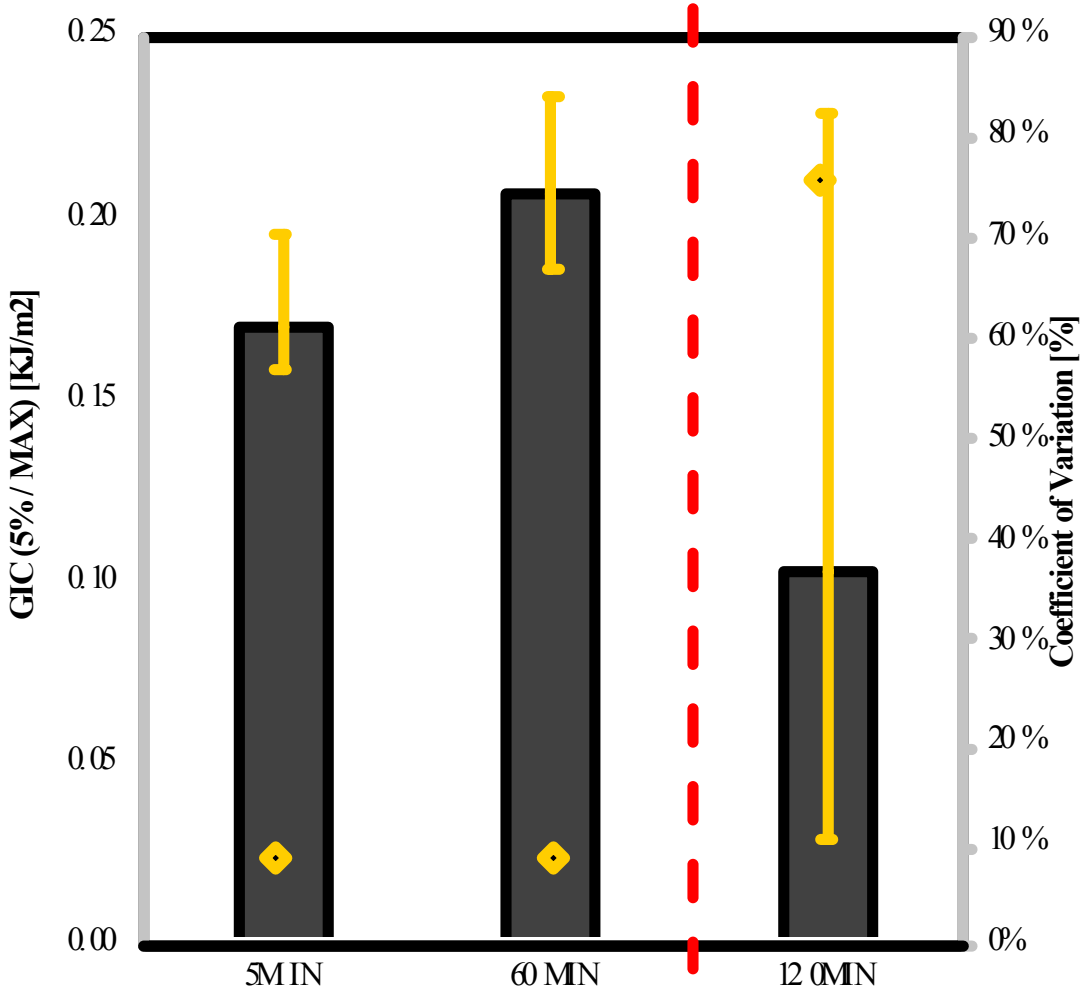


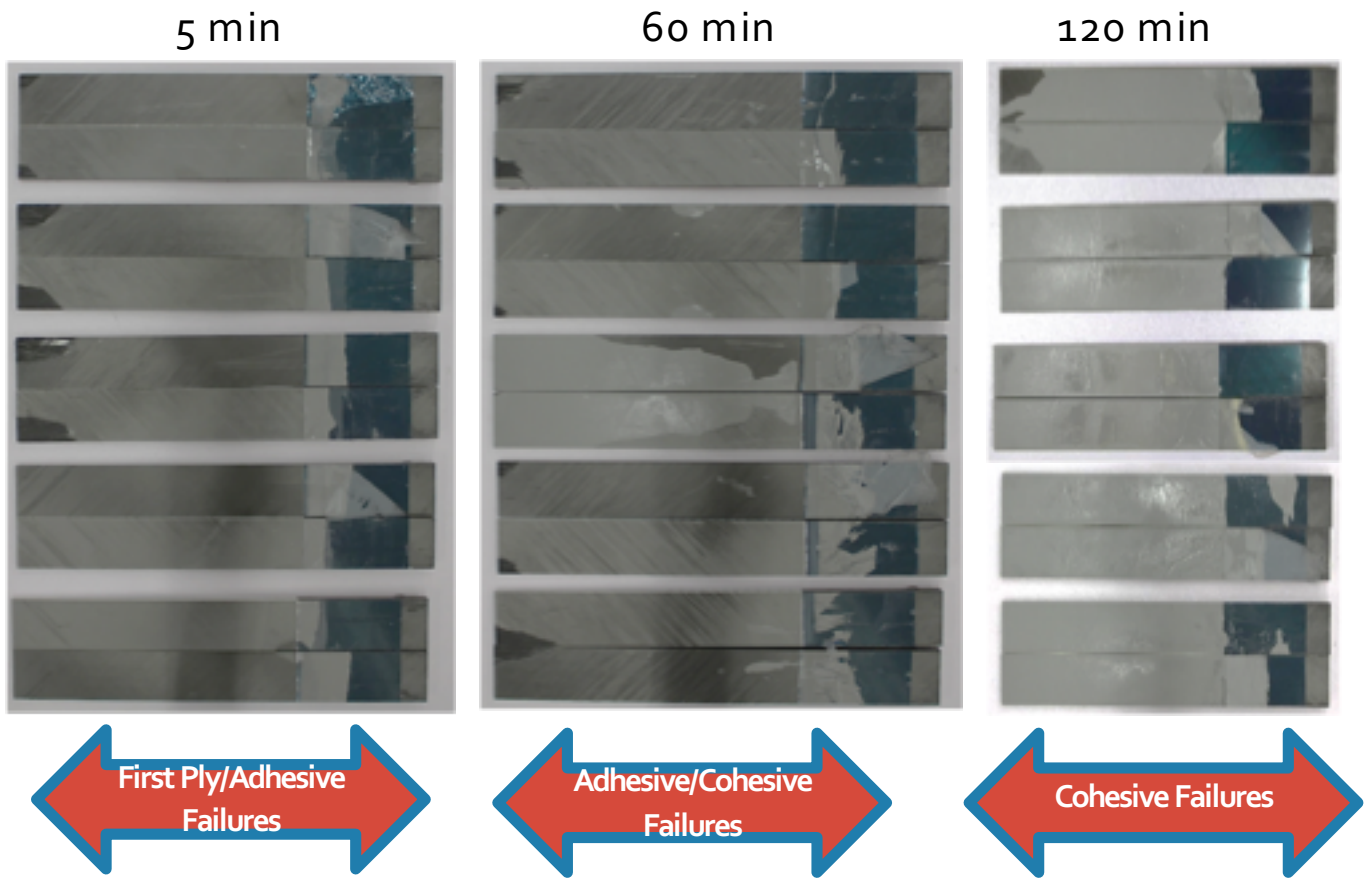
Evaluation of assembly time in paste adhesives
Test Results – Single Lap Shear – ASTM D1002



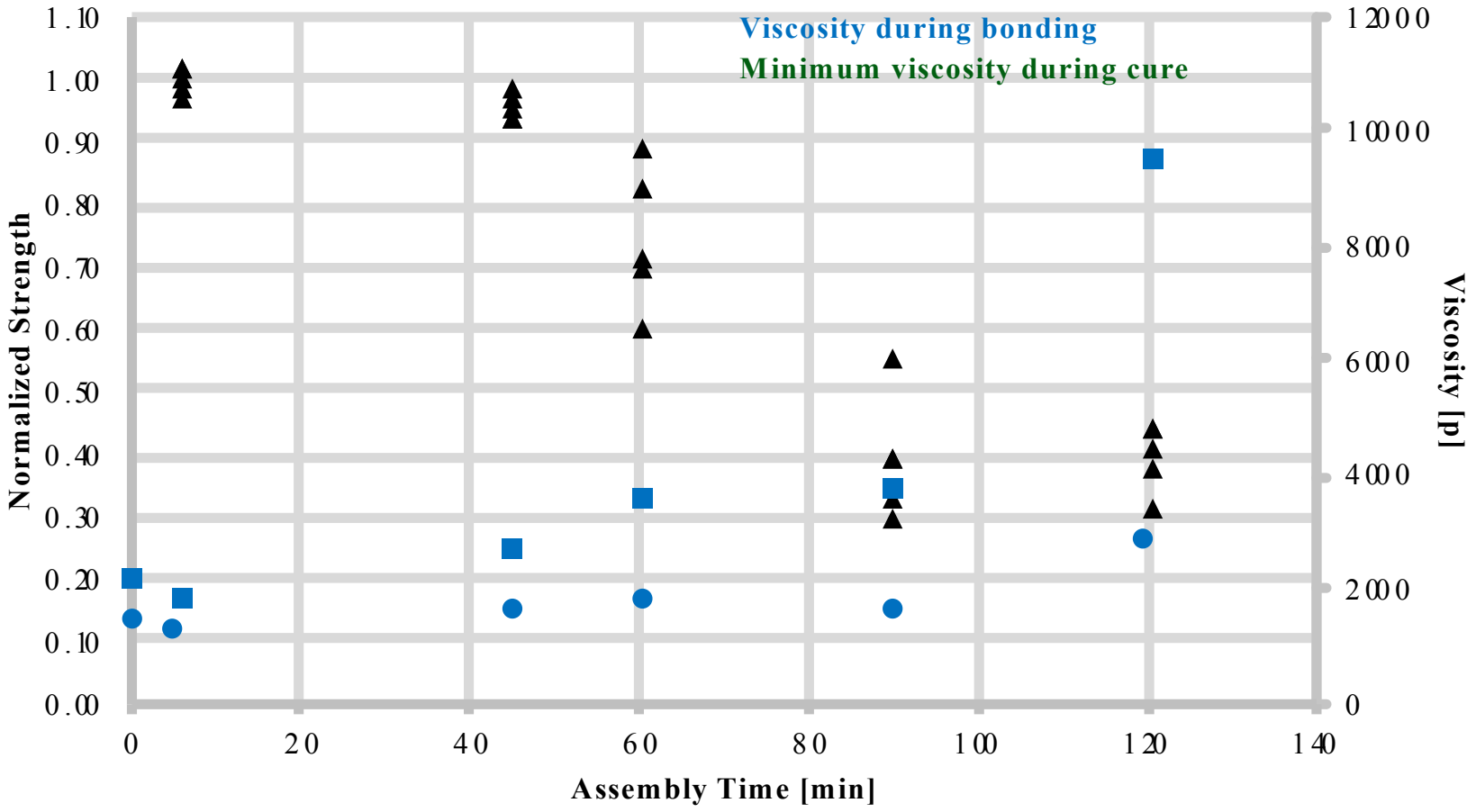


Evaluation of assembly time in paste adhesives
Test Results – Mode I – ASTM D5528



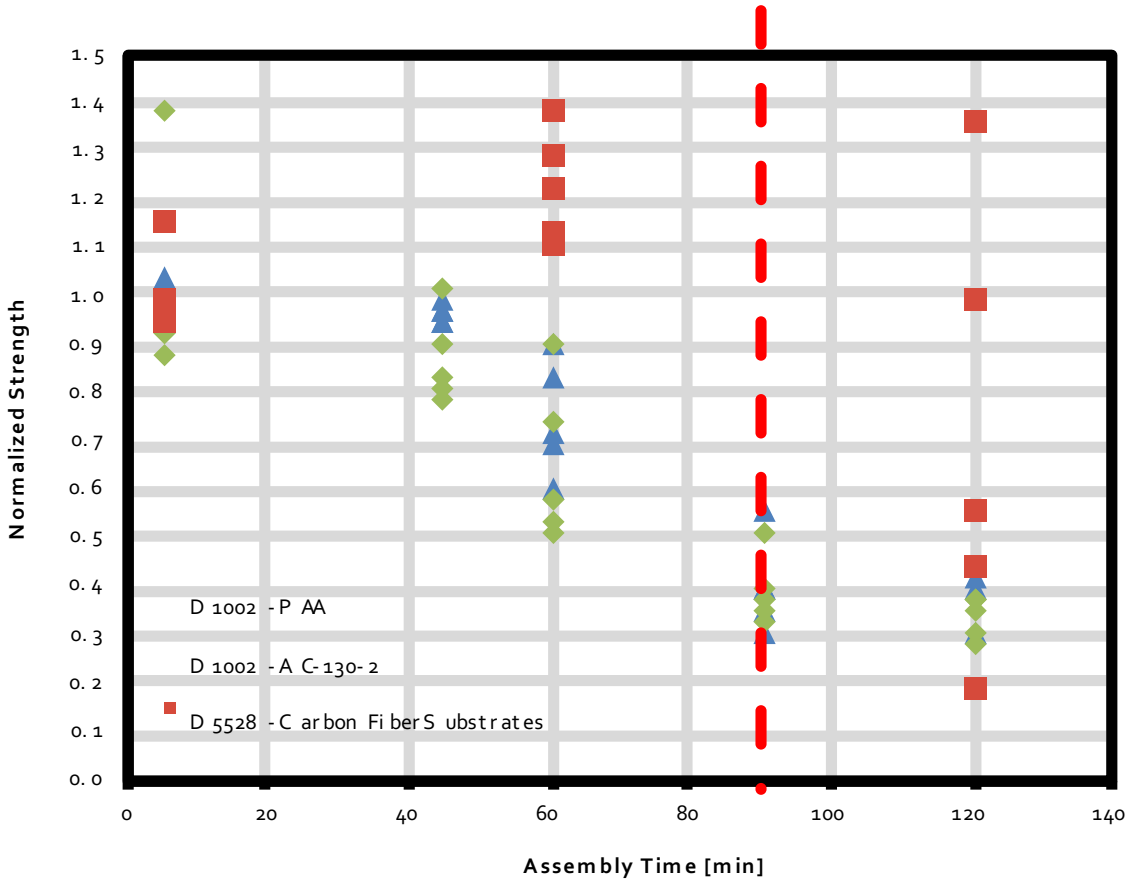


Evaluation of assembly time in paste adhesives
Test Results – Viscosity Response



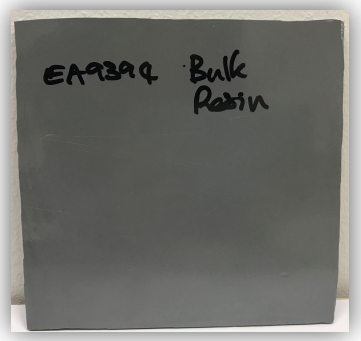
Evaluation of assembly time in paste adhesives
Test results – Summary

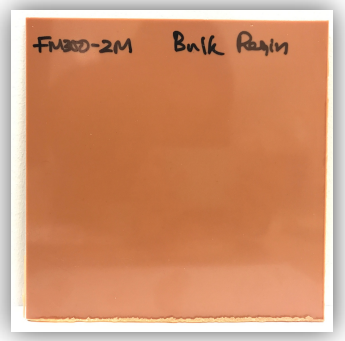
- EA9394 adhesive system showed very significant effect for longer assembly times.
- Single lap shear strength reduced by ~ 20% when assembly time was 60 minutes. This started dropping to ~50% for 90 minutes (pot life)
- Mode I fracture toughness data showed a large scatter in test data for increased assembly times.
- Mode I data showed an increase in the properties for 60 minutes assembly and rapidly dropped when the assembly time was increased to 120min.
- Failure modes throughout all the failure modes indicated cohesive/adhesive failures until 60 minute assembly time and changed to cohesive after 60 minute mark.
- Static response of the properties are desirable. However, understanding of the mix ratio effect on fatigue properties needs to be investigated.

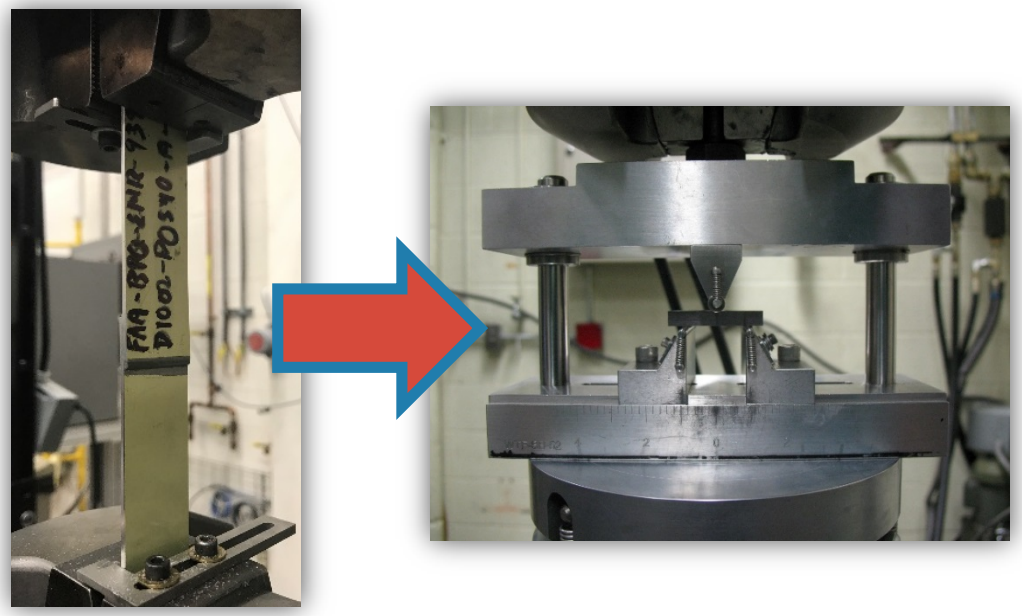


Fluid sensitivity of adhesive

- Current method used to evaluate the fluid sensitivity of adhesives is the D1002 single lap shear specimen configurations.
- D1002 Lap Shear specimen configurations requires treated substrate materials which involves numerous steps form preparation to storage.
- The adhesive area exposed to fluids is minimum. (Adhesive Thickness)
- A relatively simpler (test method + specimen geometry) bulk adhesive specimens were fabricated using the adhesive systems and simpler test configuration (3-pt bending) was used to evaluate the effects and compared with the current D1002 method.
- EA 9394 and FM300-2m material systems were used for the evaluation.
- Fluids used in this study
  - Skydrol LD-4 (SAE AS1241, Type IV, Class 1) – 90 days
  - MEK washing fluid. ASTM D740 – 90 minutes
  - 145F/85% Relative Humidity 1000hrs – Controlled Condition

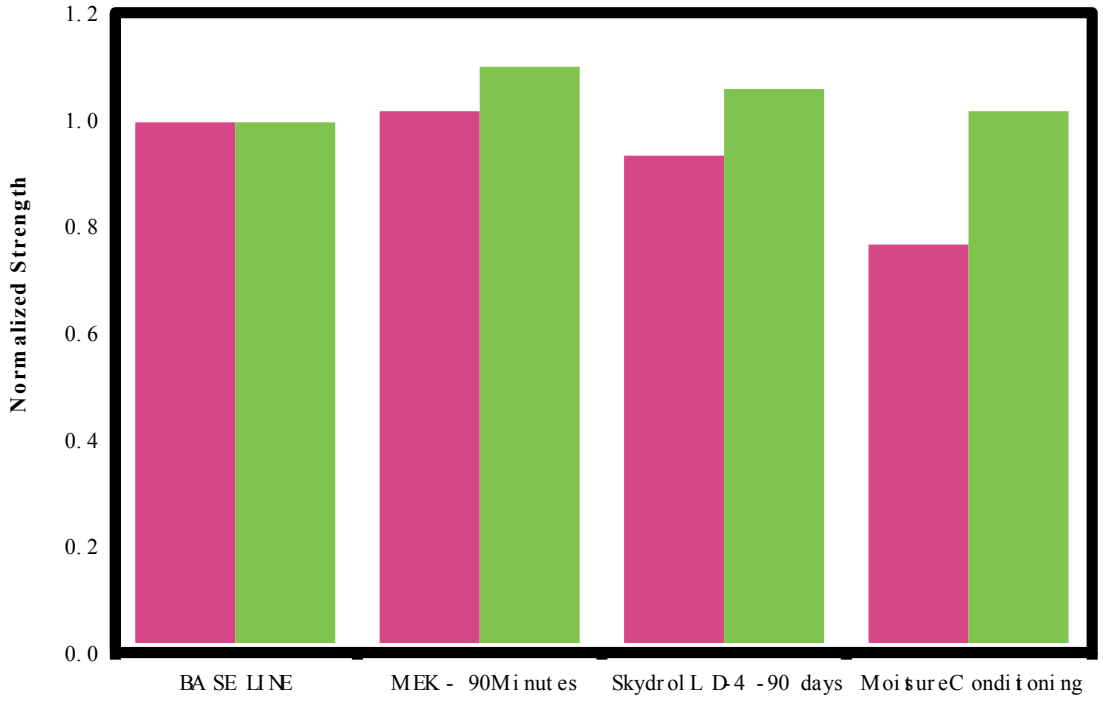


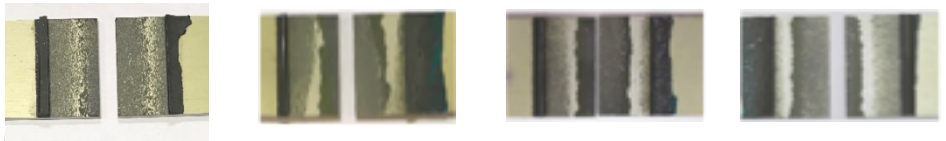




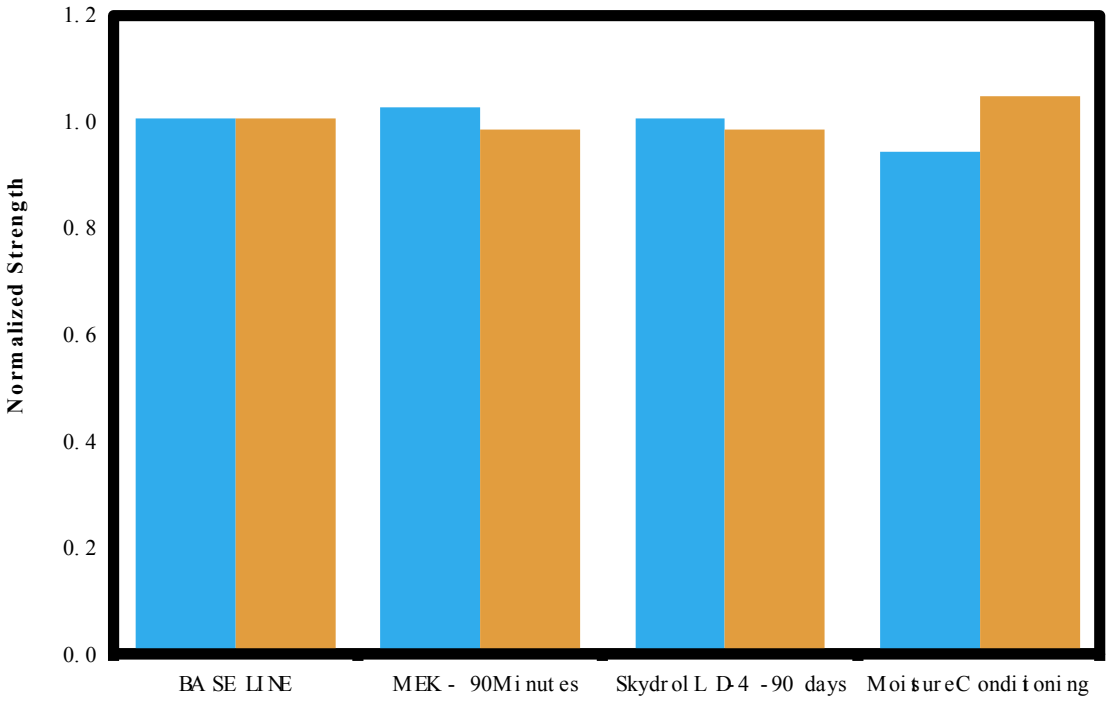
Fluid sensitivity of adhesive Test Results

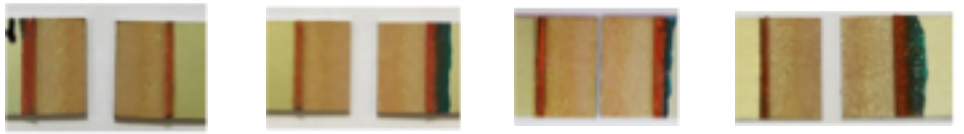
EA9394





FM300-2M

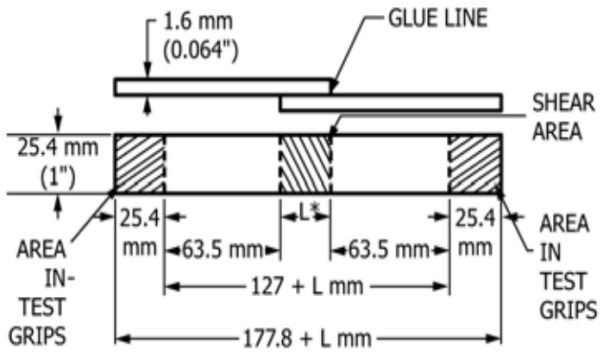




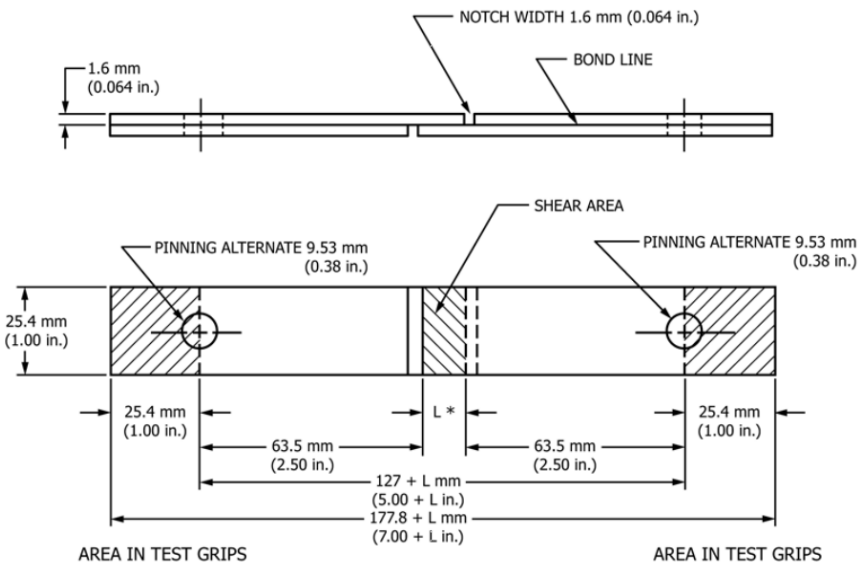
Adhesive Screening Test Methods

- Currently ASTM D1002 test method is being used to perform screening test/receiving inspections of adhesive material. ASTM D1002 requires a specialized substrates – treated chemical treatment/preparation method.
- As an alternate to this test method/substrate, Epoxyglass G10 substrates has been evaluated to be used in this type of screening testing.
- Different substrate thicknesses has been evaluated as well as a added new test method.
  - ASTM D1002 – Al substrates – 0.063-in thick (PAA+BR127)
  - ASTM D1002 – Epoxyglass G10 substrates – 0.063-in thick (Abrasion) – Thickness matched
  - ASTM D1002 – Epoxyglass G10 substrates – 0.093-in thick (Abrasion) – EI matched
  - ASTM D3165 – Epoxyglass G10 substrates – 0.25-in thick (Abrasion) - Standard
- Adhesive systems evaluated
  - FM300-2M and EA9394

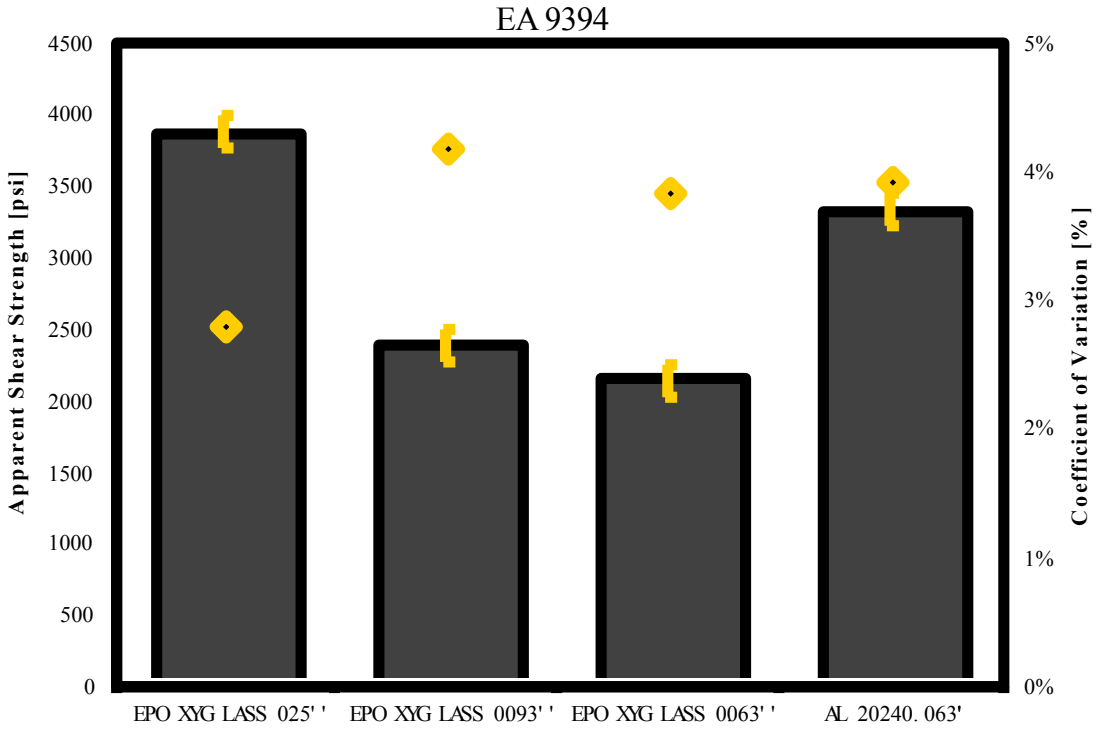
ASTM D1002

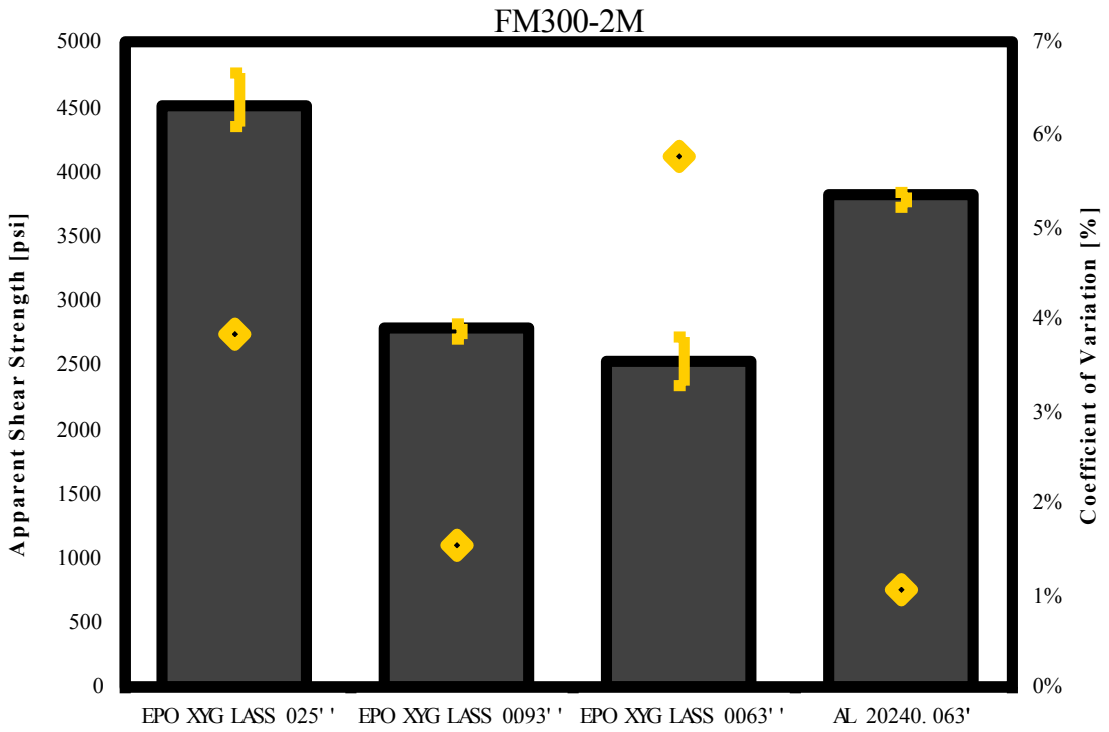


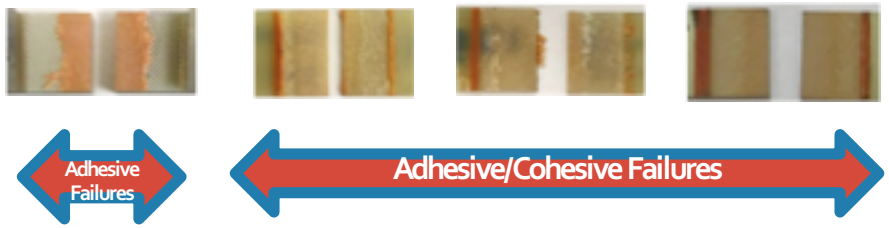
ASTM D3165



Adhesive Screening Test Methods
Test Results







Adhesive Screening Test Methods Summary

- In a receiving inspection/screening tests, it is usually a Pass/Fail criteria
- Epoxyglass substrates can be used for screening/receiving inspection tests. Baseline tests needs to be performed for the identical specimen configurations.
- Higher variation is seen in the bondline thickness for 0.25-inch thick epoxyglass substrates. Bonding process/bondline control mechanism needs to be revisited to get the required bondline thicknesses.

Looking Forward

- **Future Works**
  - Generate bond process protocols for
    - Selecting compatible substrate and adhesive combinations for a robust bond structure
    - Provide guidelines on how to select and use peel ply for composite substrate preparation
- **Benefit to Aviation**
  - Generate bond process protocols
    - Provide guidance on the critical parameters in the bond process and how to mechanically test them to generate protocols

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  - Royal Lovingfoss (NIAR)
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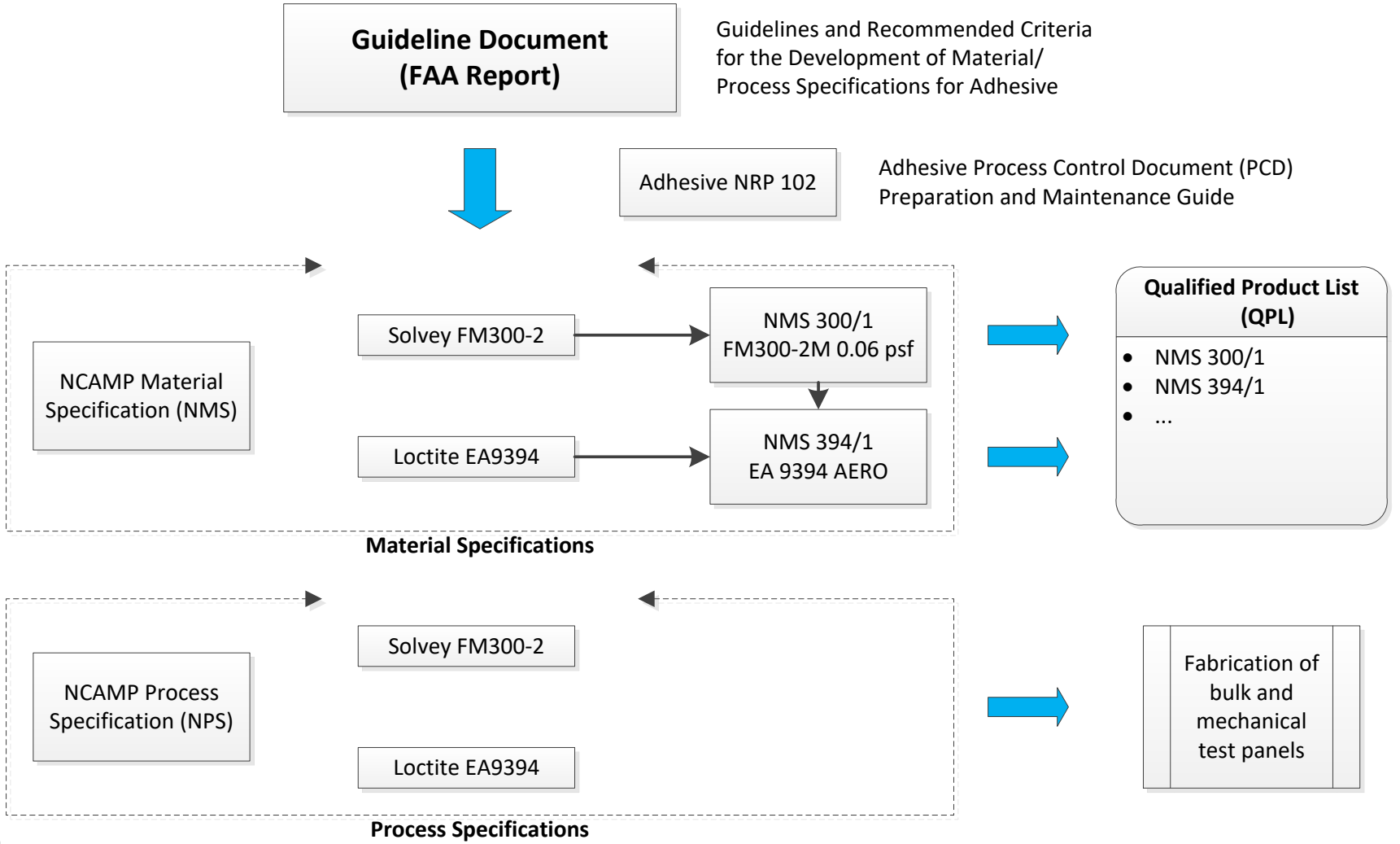
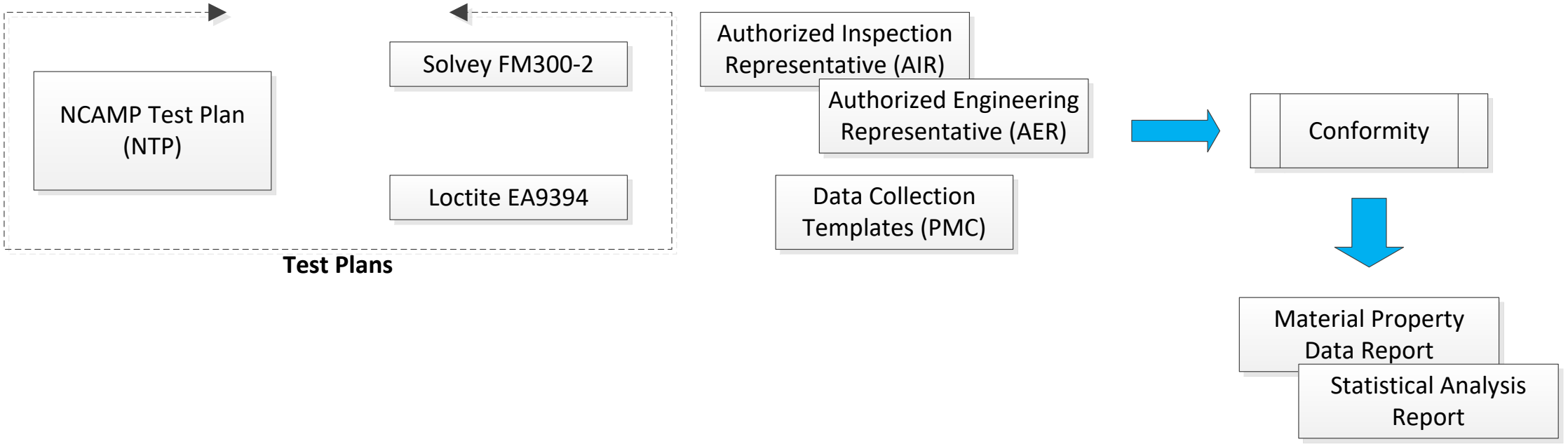
Questions and Comments ???

Development of NCAMP Specifications



Development of NCAMP Test Plans



Road Map - Adhesive Qualification Guidance

Guidelines → Specs (MS/PS) → Test Plans → Procedures

Testing → Data Analysis → Data Reports → Maintaining Data

Data Review → Submit Data to CMH-17 → Yellow Pages → CMH-17