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Adhesive Bond Process Qualification Protocols Development & Development of Roadmap for Bonded Structure Certification

Waruna Seneviratne, John Tomblin, and Upul Palliyaguru

2019Technical Review - (05/22/2019)

NIAR
Wichita State University
National Institute for Aviation Research

Adhesive Bond Process Qualification Protocols Development & Development of Roadmap for Bonded Structure Certification

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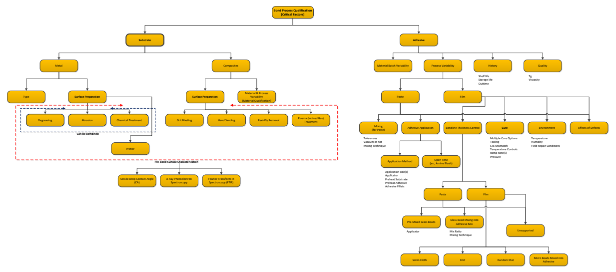
  - *Upul Palliyaguru and Anushi Amaranayake*
- FAA Technical Monitor
  - Ahmet Oztekin
- Other FAA/CMH-17 Personnel Involved
  - Larry Ilcewicz, *PhD, Cindy Ashforth, and* Curtis Davies,
- DoD & Industry Participation
  - AFRL, Boeing, Bell Helicopter, Henkel, Honda Aircraft Co., Lockheed Martin, MMM,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

Adhesive Bond Process Qualification Protocols Development (Background)

- Aircraft companies tend to use bonded joins in their primary structure due to various time and cost savings. However, qualification of the bond process and certification of the bonded structure requires extensive amount of substantiation work.
- Due to the complexity and numerous variables seen in a bond system, locking on to a these parameters needs extensive exploration of all possible variations in the bond process. After locking onto this processes, effective and efficient methods for quality assurance needs to be implemented to qualify the bonding process.
- After the bond process qualification tasks are completed, bonded structure needs to be certified per the requirements of the safety agencies.



The Primary goal of this research program is to develop a road map for qualification activities of a bond system and support development of certification road map for bonded structures per the safety requirements through substantiation.

Adhesive Bond Process Qualification Protocols Development (Road Map)

Design and Preliminary Bond System Assessment	Design and Preliminary Bond System Assessment	Bond Process Qualification and Protocol Generation	Structural Certification of Bonded Structure & Maintenance

Objective

Manufacture a bonded wing structure

Preliminary Design Requirements/Knowledgebase of the Bonded Structure

1. General Size
2. Mechanical property requirement for Bonded Joints
3. Environmental Envelope
4. Manufacturing Requirements
5. Analysis of critical bonded joint types seen in the structure
6. Bondline thickness requirements

Preliminary Selection and Screening of Substrate and Adhesive Materials

1. Material allowable – (Material databases)
2. Adhesive & Substrate compatibility assessment & Wettability assessment
3. Selection of surface preparation methodology
4. Adhesive processing parameters of a representative design.
5. Conduct basic adhesive test methods at room temperature to validate the parameters

Bond Process Qualification Plan and Protocol Development

1. Finalized Bond System
2. Based on the parameters, compose a test matrix to ensure quality assurance of surface preparation and processing parameters
3. Generation of quality assurance methodologies

Structural Certification of Bonded Structure & Maintenance

1. Screening of bond system
2. Long term durability
3. Substrate & adhesive characterization
4. Bonded joint characterization
5. Durability & environmental scatter
6. Damage Tolerance and Crack growth

Maintenance

1. Inspection methodology development
2. Inspection methodology for bond strength degradation.
3. Identification of inspection level and frequency.

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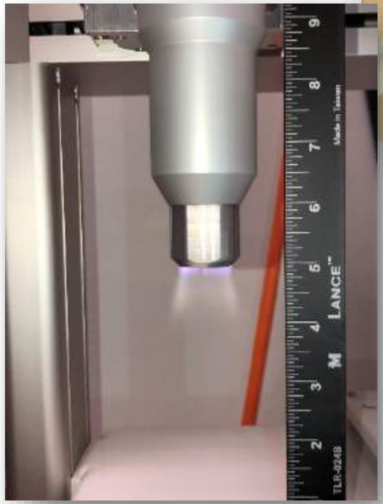
Maintenance

1. **Inspection methodology development**
2. **Inspection methodology for bond strength degradation.**
3. **Identification of inspection level and frequency.**

Overview of the Presentation

- Preliminary screening and down-selection of adhesive-substrate combinations
- Critical parameters in the surface preparation
  - Surface preparation methodology
  - Quality assurance and handling of prepared substrates
- Critical parameters in the adhesive application and cure process
  - Adhesive handling guidelines
  - Mixing and application
  - Bondline thickness control
- Bond process qualification protocols generation to asses the effect of varying the parameters



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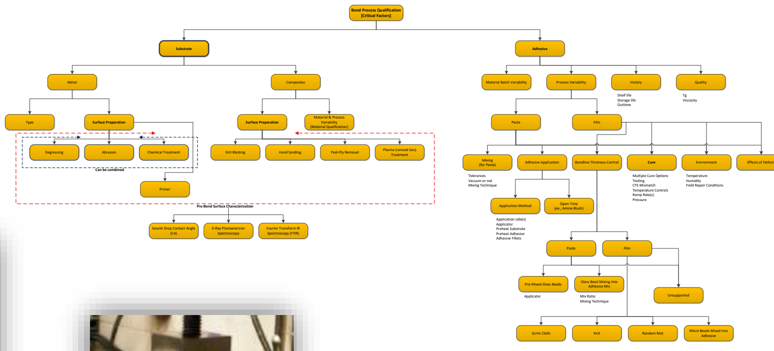
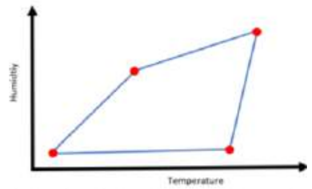
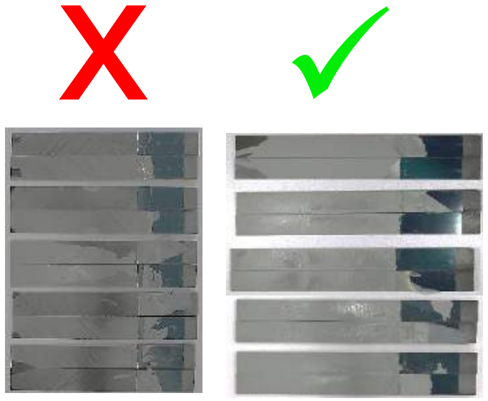
Preliminary Selection and Screening of Various Substrate and Adhesive Materials



- ✓ Selection of preliminary candidates for the adhesive and substrate materials
- Bond system parameter down-selection
  - Surface preparation and adhesive processing
    - Phase I – Facility and equipment limitations
      - Preliminary design dimensions – a general idea of the size
      - Facility processing equipment – Curing and pressure application
    - Phase II – Material performance limitations and incompatibility issues
      - Operational environmental envelope
      - Physical - wettability of substrates and adhesive
      - Thermal – CTE and Tg mismatch
      - Mechanical – poor static and durability performance – failure mode based assessment

Preliminary Screening Activities





Bond Process Qualification Protocol Generation
Quality Assurance of Surface Preparation Methodologies

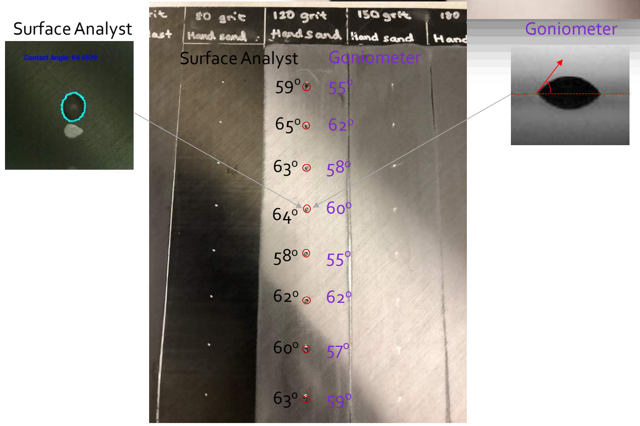
- Pre-surface preparation checklist
  - Quality control and process specification of substrates (cured)
    - Ex. Mold release, surface finish (bag/tool)
- Quality control of equipment/tools used in surface preparation
  - Material specifications
    - Ex. Sand paper/sanding disks, peel ply, chemicals
  - Operational settings of equipment
    - Ex. – Sander types, speed, force, Plasma generator parameters
- Quality control and training of technicians involved in the process
  - Quantification and assessment of variability in the hand sanding process
- Evaluation of surface preparation to bonding time limitation
  - Effect of environmental exposure duration on surface free energy
- Quality check of the prepared substrates to ensure the integrity of the bond system.
  - Development of surface preparation standards and quantification of the effects

Common Surface Preparation Methods (Metallic & Composite substrates)

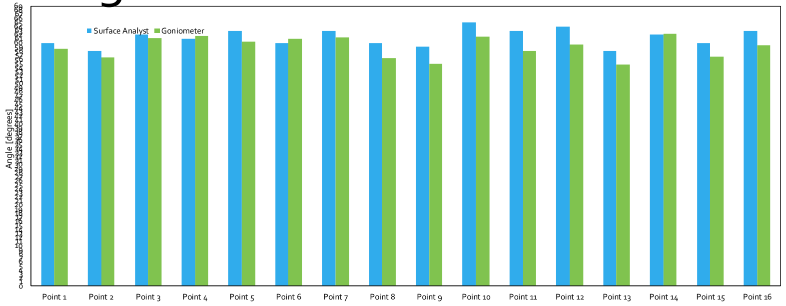
- Abrasion
  - Hand Sanding
  - Grit Blasting
- Peel Ply
- Atmospheric Plasma Treatment (ATP)
- Degreasing
- Chemical Treatments
- Corona Discharge
- Laser Ablation

Bond Process Qualification Protocol Generation
Quality Assurance Standard Development

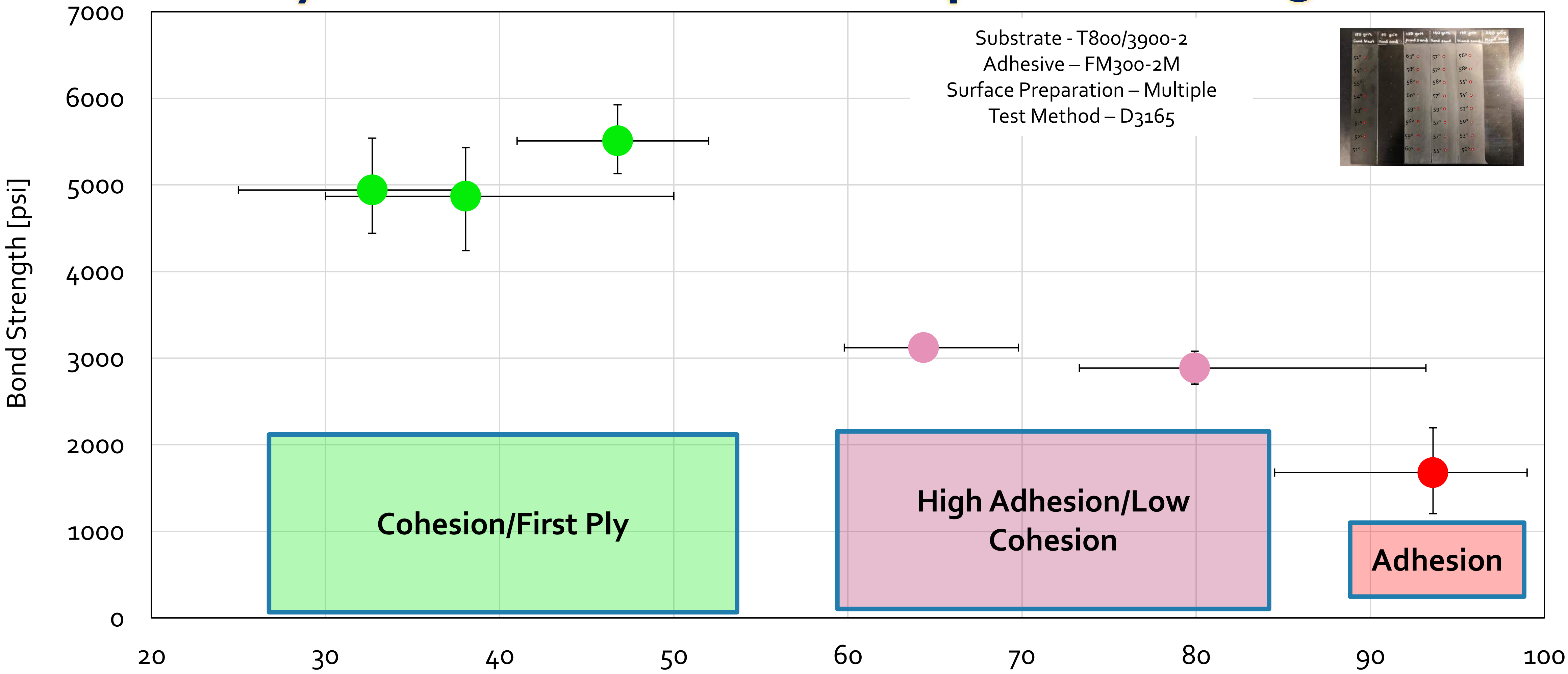
- Surface Preparation
  - Goal – Increase the surface free energy -> better wettability -> good bonds
  - Method of verification -> Water contact angle measurement
  - Quality check -> Water contact angle measurement comparison to a known standard
  - Equipment used – Surface Analysts – BTG Labs
  - Contact angle measurements validated with Goniometer results.
- Surface preparation quality assurance standard
  - Utilizing different abrasion methods (pressures/grit size) – obtain a range of different surface free energies (contact angles)
  - Fabricate bonded joint specimen and evaluate the bond strength



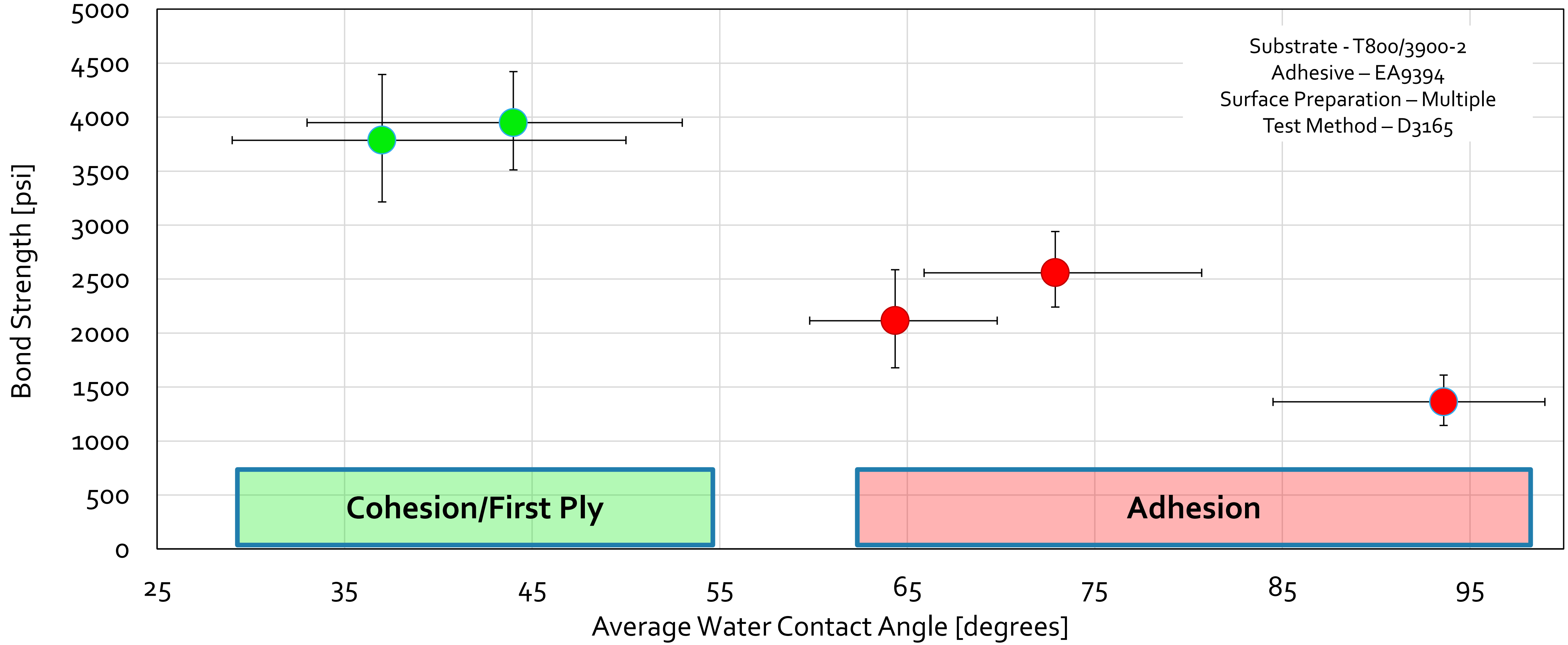
± 2 ° variation

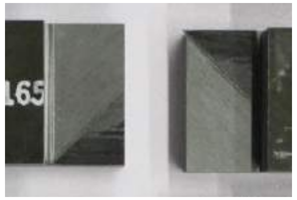


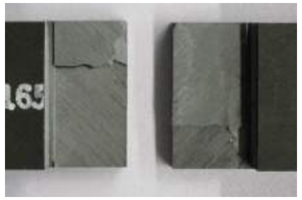
Quality Assurance of Surface Preparation – FM300-2M

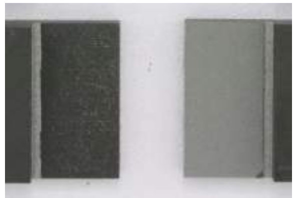
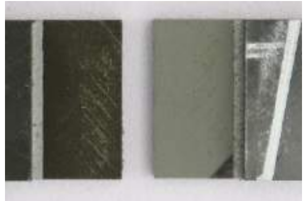
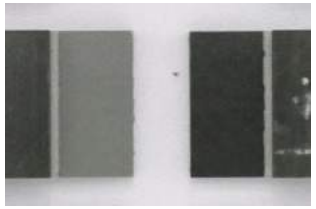


Quality Assurance of Surface Preparation – EA9394







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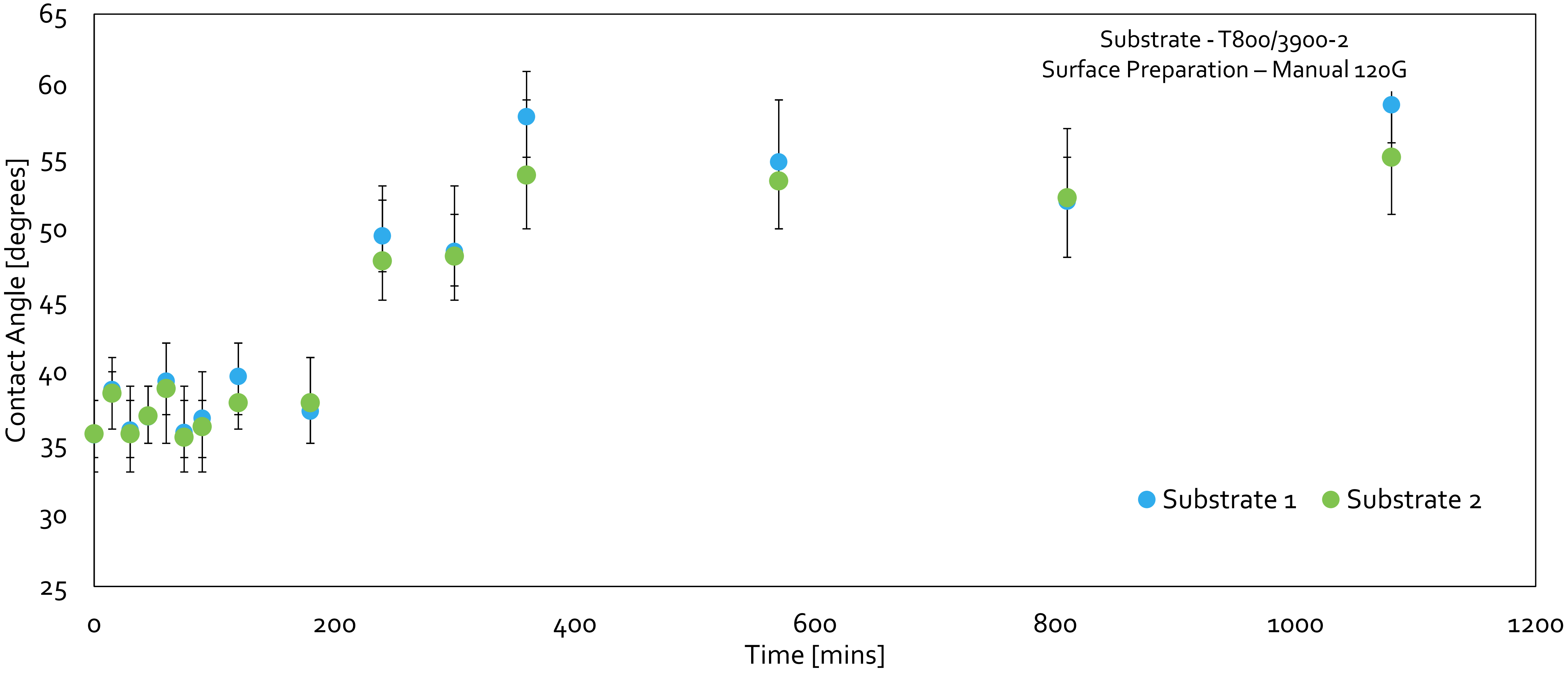
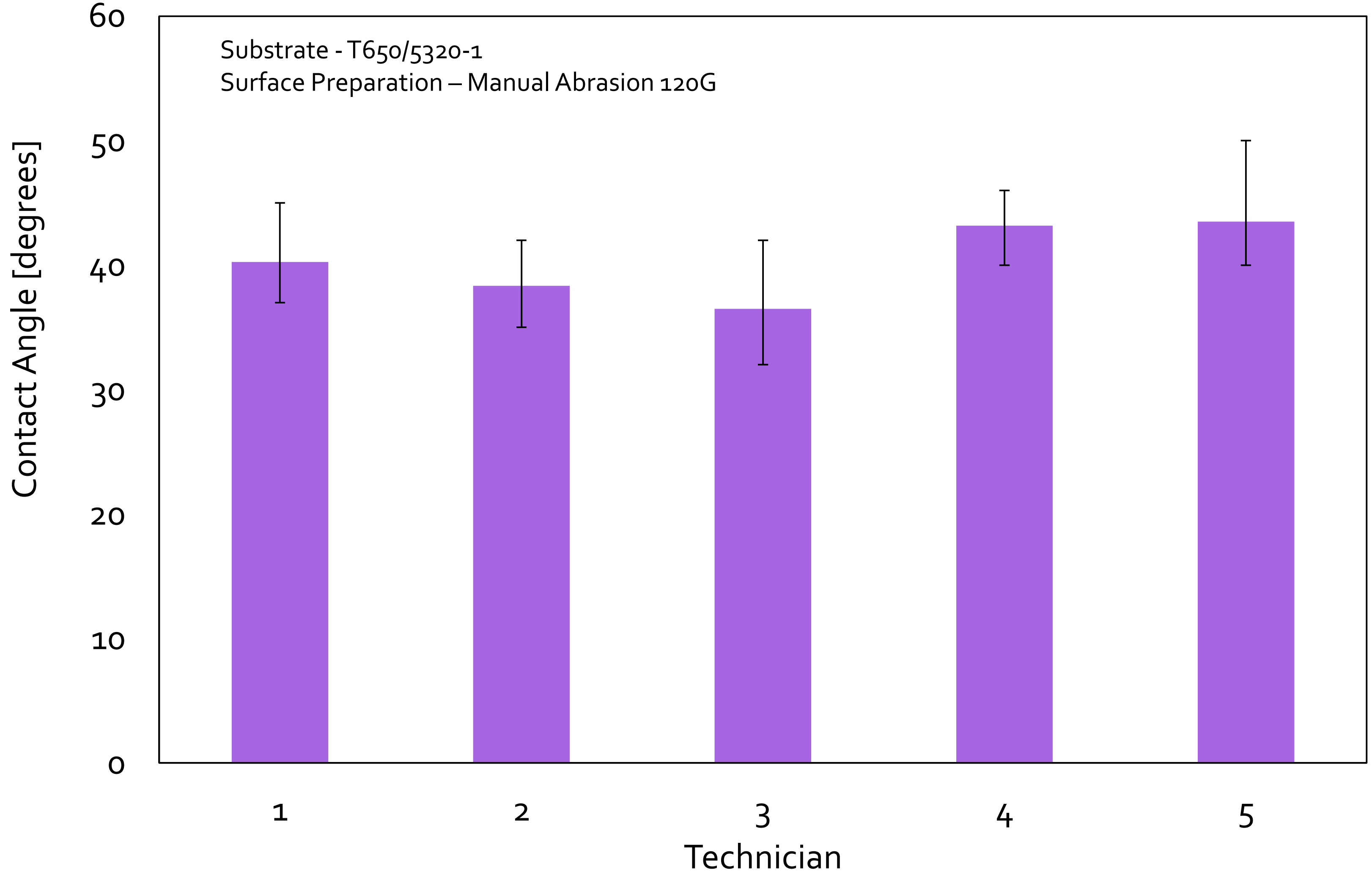
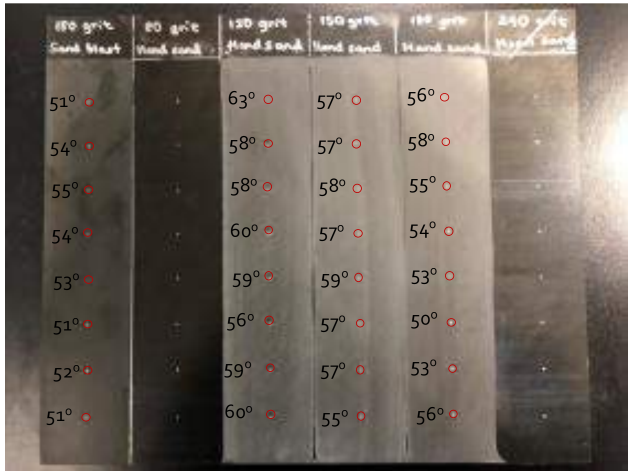
Environmental Exposure Effects of Prepared Substrates



Surface Preparation - Hand Abrasion Technician Process Variation





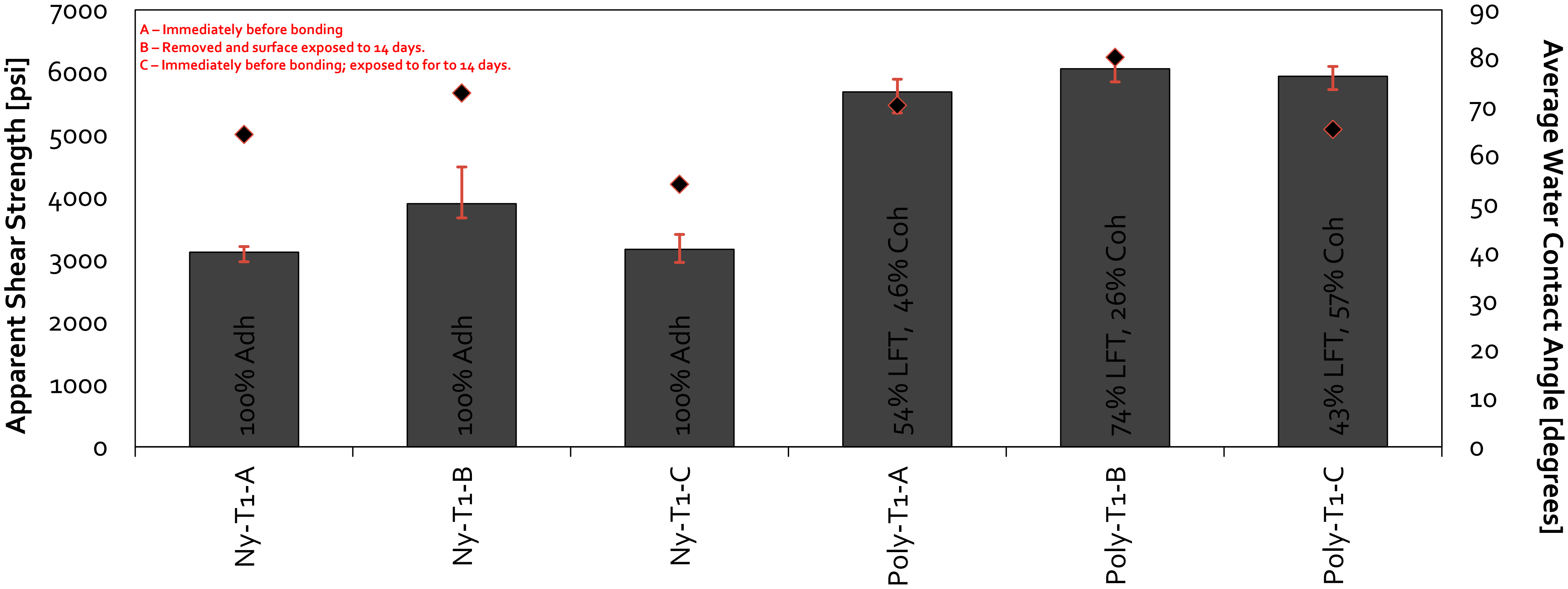
Surface Preparation - Peel Ply Removal

- Initial assessment of peel plies
  - Commonly used Nylon and Polyester peel ply was used for the study.
- Peel ply usage studies were performed to identify critical parameters
  - Peel ply removal time frame and exposure duration
    - A – Immediately before bonding
    - B – Removed and surface exposed to 14 days.
    - C – Immediately before bonding; exposed to for to 14 days.
    - Mode I and Single Lap Shear Properties
  - Post cure effects on peel ply prepared surfaces – Multiple Cure Cycles (MMC)
    - FM300-2M – T800/3900-2 (Substrate and Adhesive combination)
      - Cure Cycles
        - Baseline Initial Cure – 350F for 2hrs with 85 psi pressure (Substrate Cure)
        - MCC1 – 350F for 2 hrs.
        - MCC2 – 350F for 2hrs (X2)
    - Degree of Cure and Fiber Volume Fraction
    - Mode I and Single Lap Shear Properties (in progress)

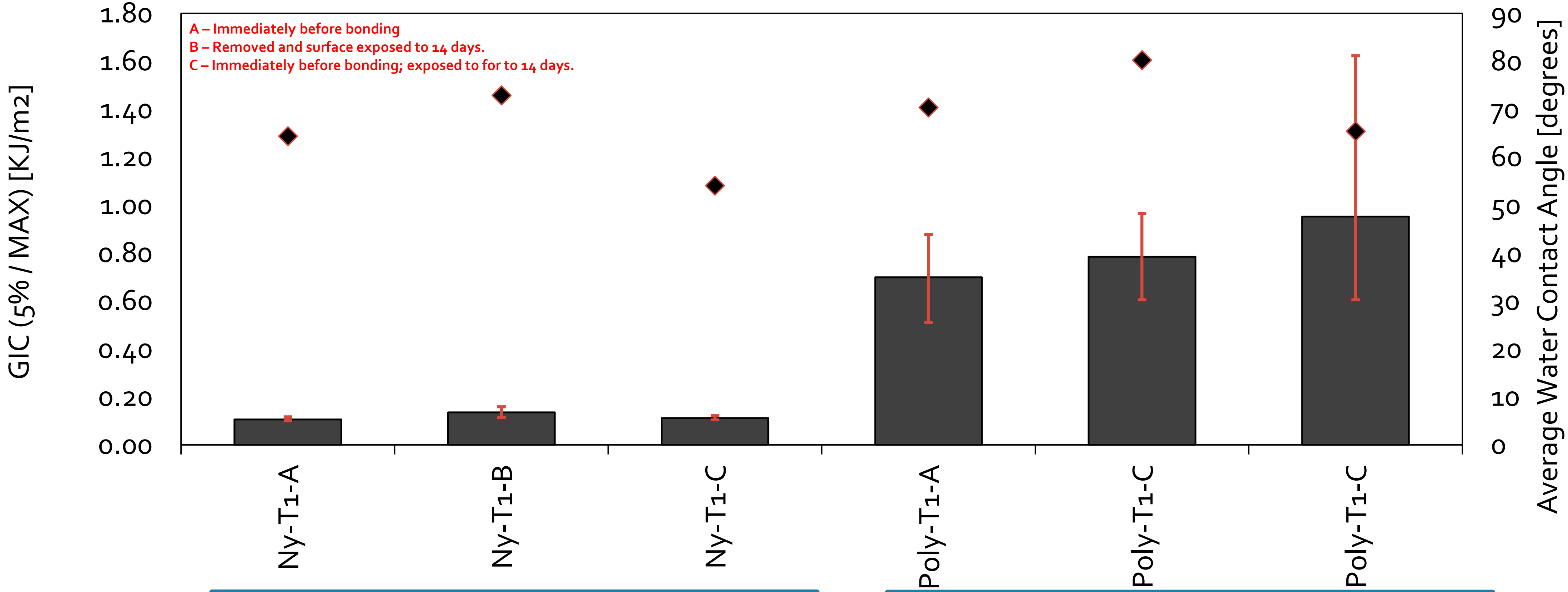
Material	Code	Style	Finish	Thickness [in]	Description
Nylon	40000	56180	60	0.0075 – 0.0085	Natural
	41661	56137	60	0.0065 – 0.0075	Natural
	51789	52006	60	0.0045 – 0.0055	Natural
	52008	56115	60	0.004 – 0.005	Natural
Polyester	60001	60001	60	0.005 – 0.006	Natural
	60002	56030	60	0.005 – 0.006	Natural
	60004	56111	60	0.0045 – 0.0055	Natural
	60005	56210	60	0.006 – 0.007	Natural
	60005	56210	65	0.006 – 0.007	Very Low Porosity

MEK	MEK wipe only			
120G	Hand sanding with 120 grit			
Ny	Nylon peel ply	**T1**	**A**	Peel ply removal immediately before bonding
			B	Peel ply removed, surface exposed for 14 days
			C	Peel ply intact, substrate exposed for 14 days
Poly	Polyester peel ply		**PP-MCC1**	Peel ply intact, one post cure thermal cycle
			PPR-MCC1	Peel ply removed, one post cure thermal cycle
			PP-MCC2	Peel ply intact, two post cure thermal cycles
			PPR-MCC2	Peel ply removed, two post cure thermal cycles

Evaluation of Peel Ply Removal and Exposure
D3165 - Single Lap Shear – FM300-2M



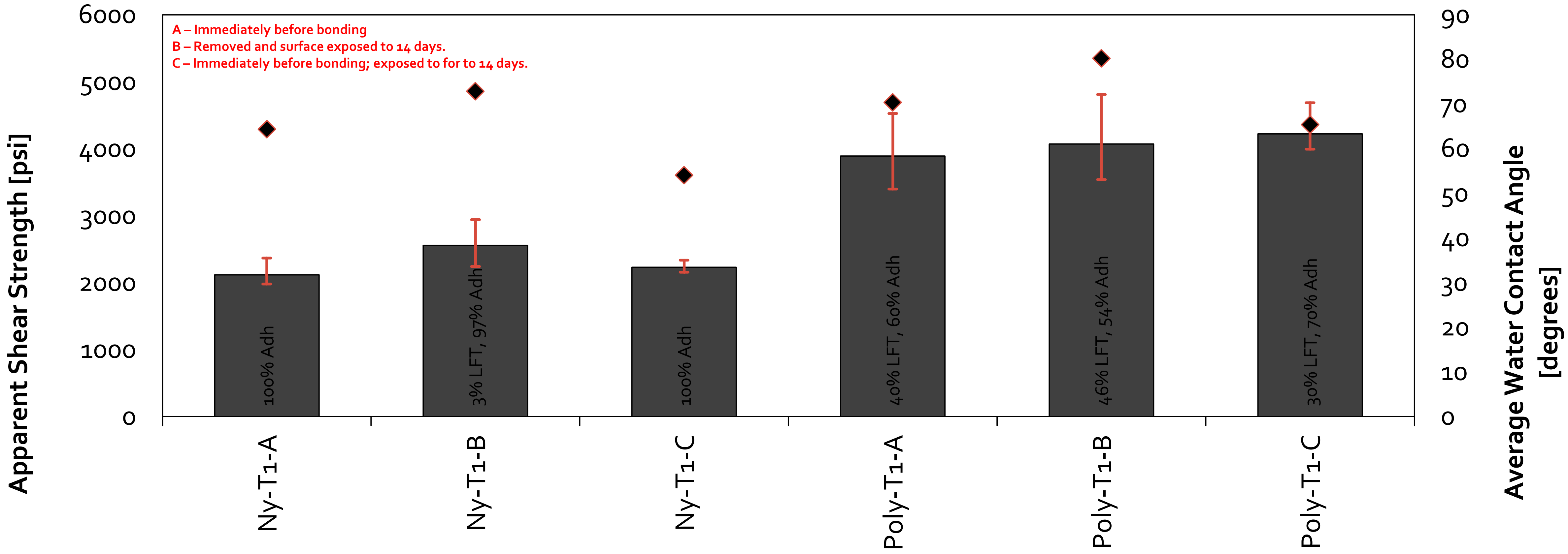
Evaluation of Peel Ply Removal and Exposure
D5528 - Mode I - FM300-2M

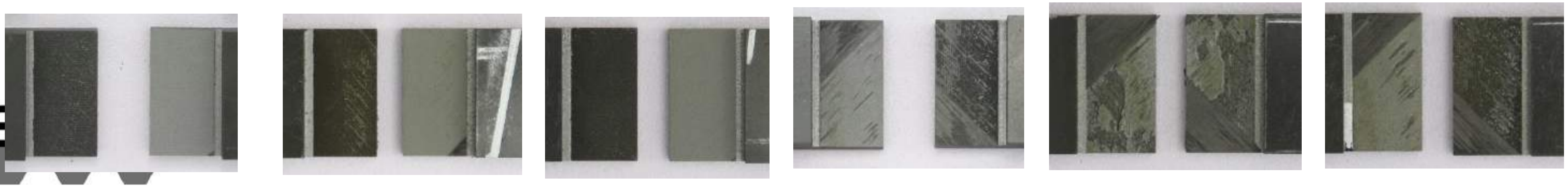


Adhesion Failure

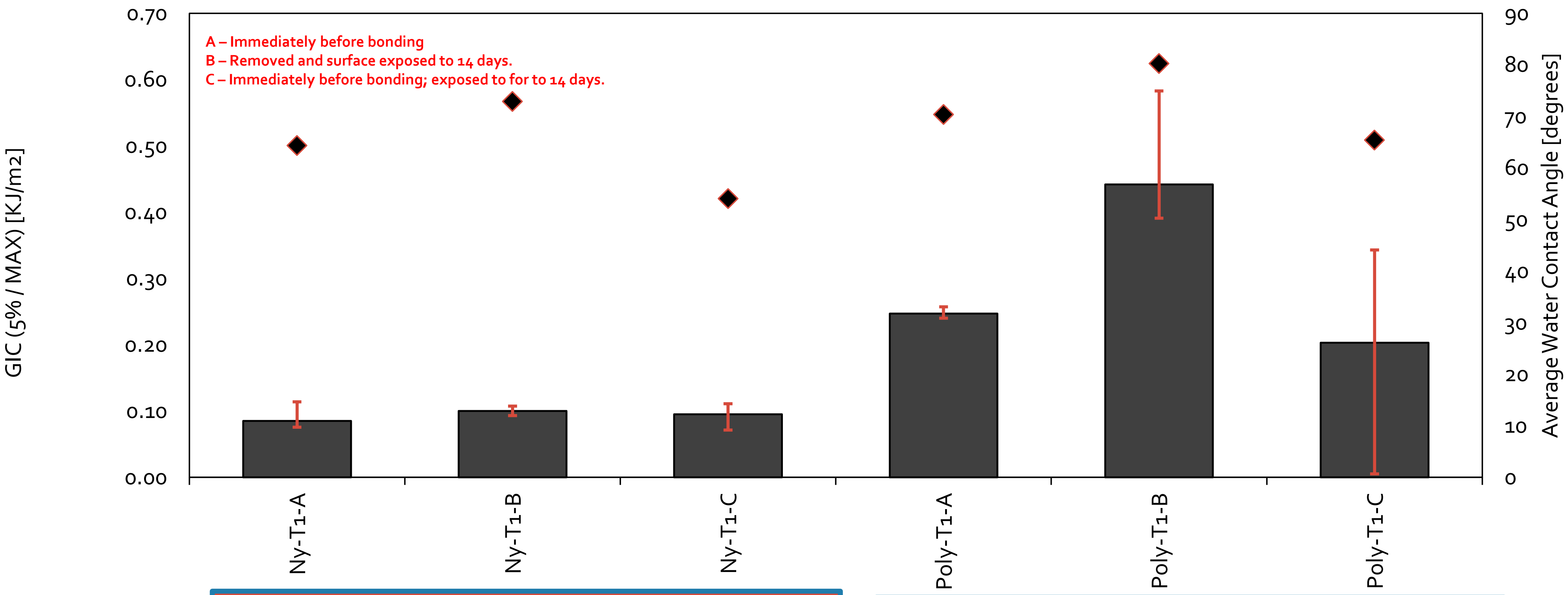
Cohesion/First Ply Failure

Evaluation of Peel Ply Removal and Exposure
D3165 - Single Lap Shear – EA 9394





Evaluation of Peel Ply Removal and Exposure
D5528 - Mode I – EA9394



Adhesion Failure

Cohesion Failure

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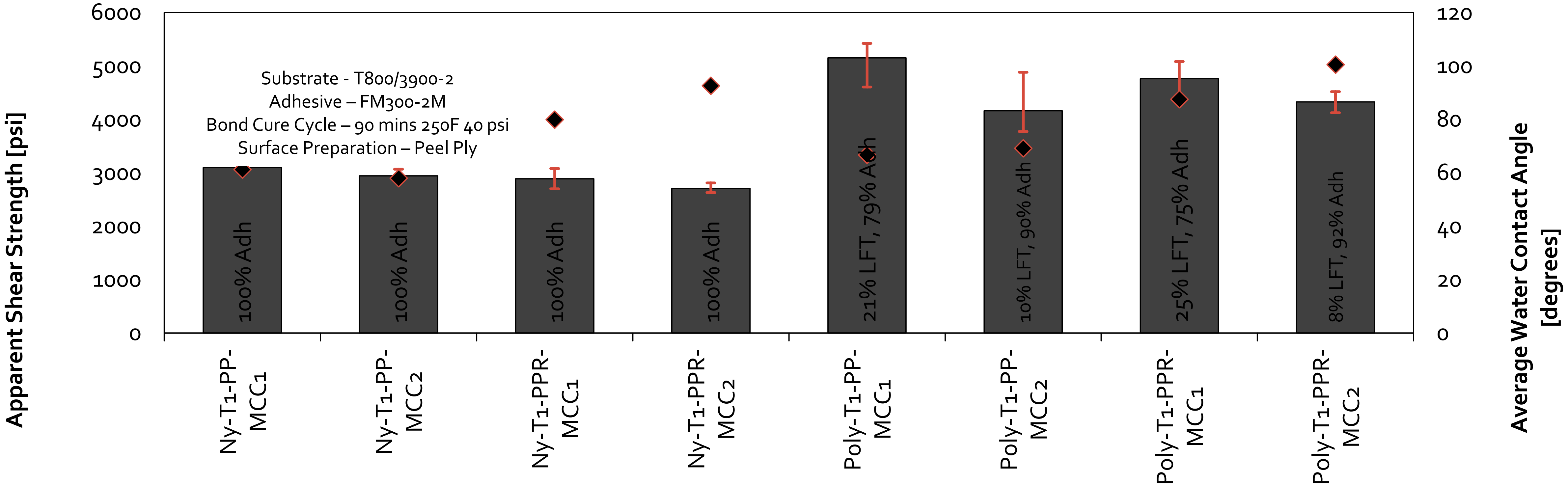
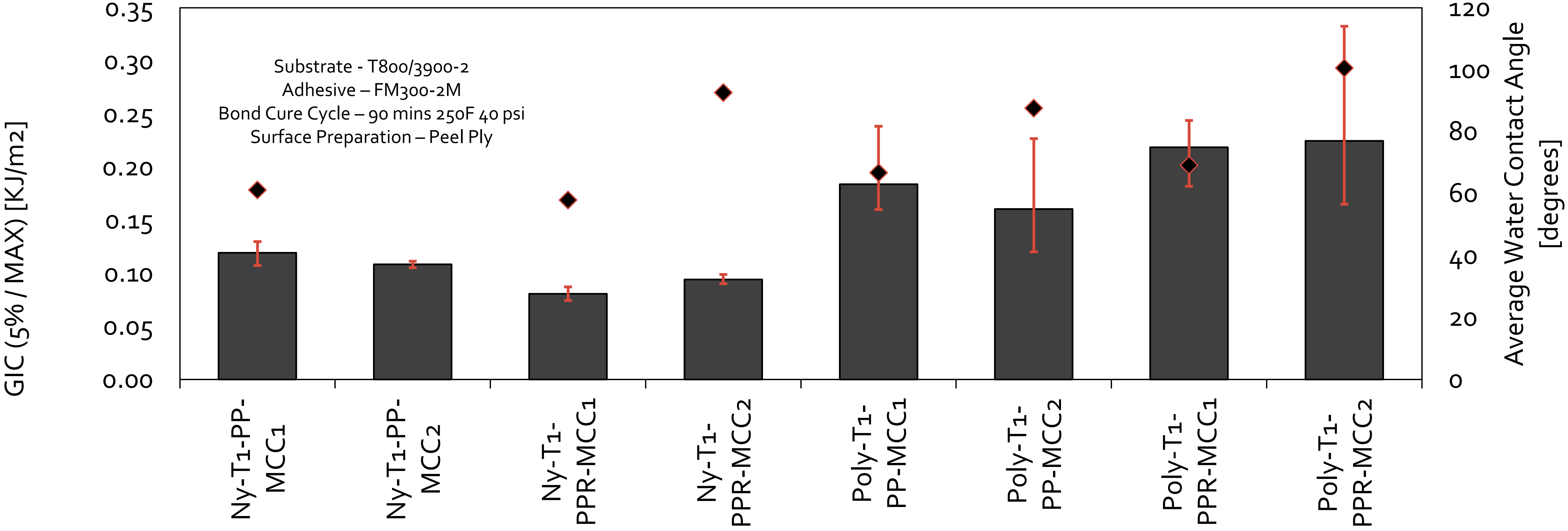
Evaluation of Peel Ply Removal – Multiple Cure Cycles
FM300-2M - D3165 Single Lap Shear



Adhesion Failure

Low Cohesion/First Ply – High Adhesion

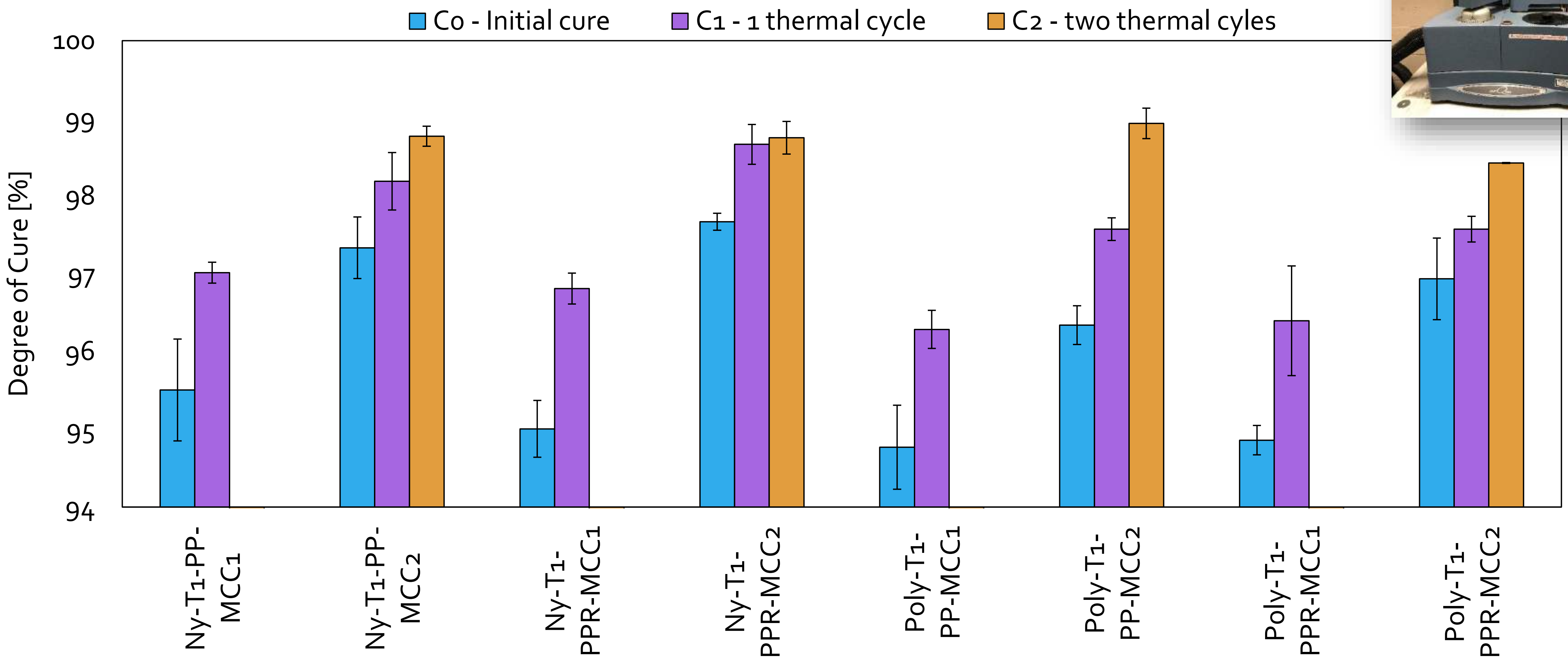
Evaluation of Peel Ply Removal – Multiple Cure Cycles
FM300-2M – D5528 Mode I



Adhesion Failure

Low Cohesion/First Ply – High Adhesion

Evaluation of Peel Ply Removal – Multiple Cure Cycles Degree of Cure



Evaluation of Mixing Method
Hand Mixing vs. Speed Mixer

- Hand mixing
  - Materials weighed into cup and mixed for 5 minutes. Mixture is then transferred to second cup and mix for an additional 5-10 minutes or until the consistency of the adhesive has changed to become smoother and easier to mix.
- Speed Mixer
  - Materials weighed into FlackTek compatible cup and placed inside machine with holder. An appropriate recipe (depending on weight) is chosen and the machine is run.

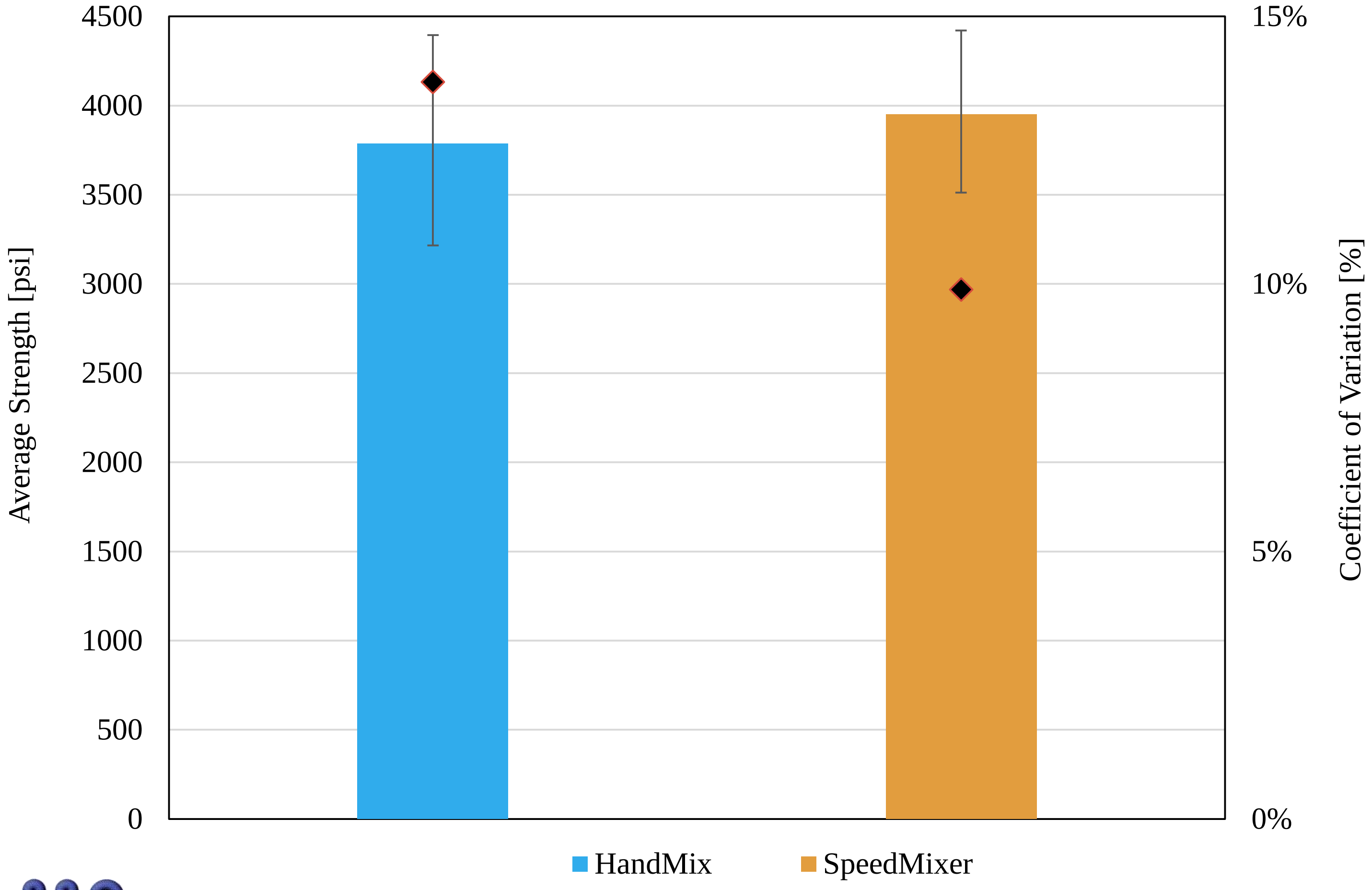
Hand mixing

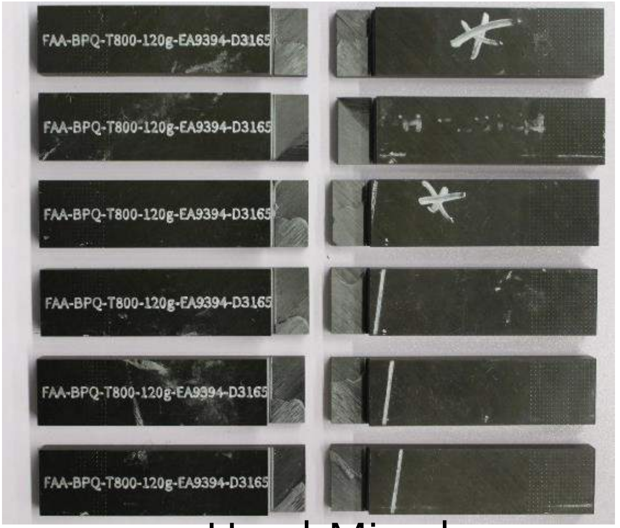
Speed Mixer Flacktek DAC 600.1FVZ

	Zone		
	A	B	C
RPM	1000	1600	2000
Time (secs)	60	40	90

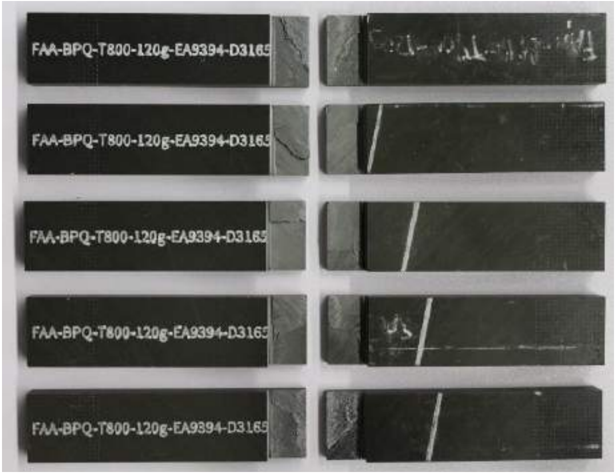
Recipe for 125g of adhesive

Evaluation of Mixing Method
Hand Mixing vs. Speed Mixer





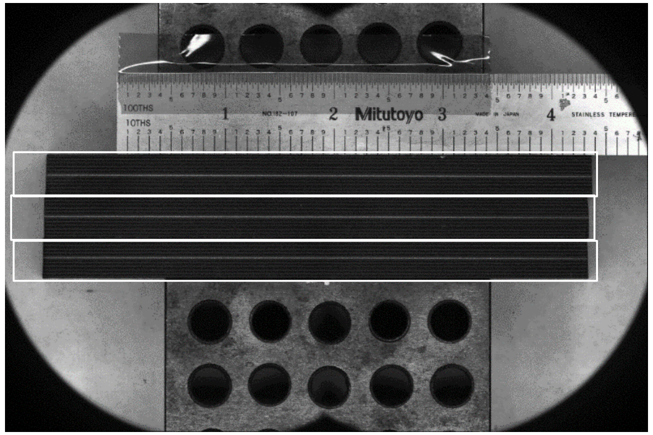
Hand-Mixed



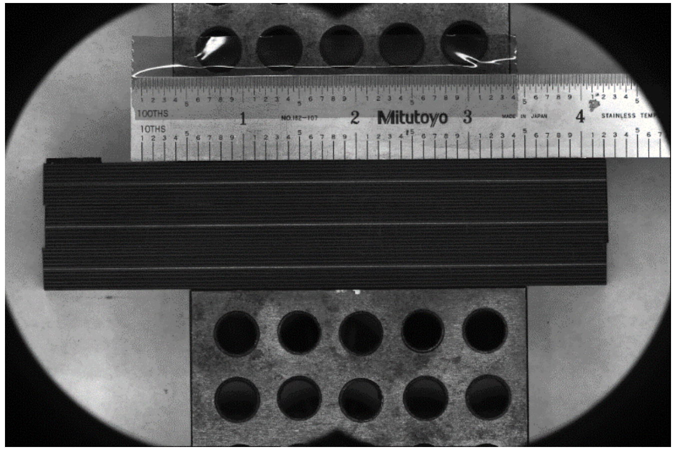
Machine Mixed

Bondline Control Mechanism

- Bondline control mechanisms available
  - Glass beads
  - Tracer Wires
- Evaluate the mix percentage for optimal bondline control
  - 0.0059-inch GB mixed at 0.05% and 0.1% by weight
  - 0.01-inch GB mixed at 0.05% and 0.1% by weight
- Effects of cure/pressure application
  - In Progress
- Effects on the mechanical properties
  - In Progress

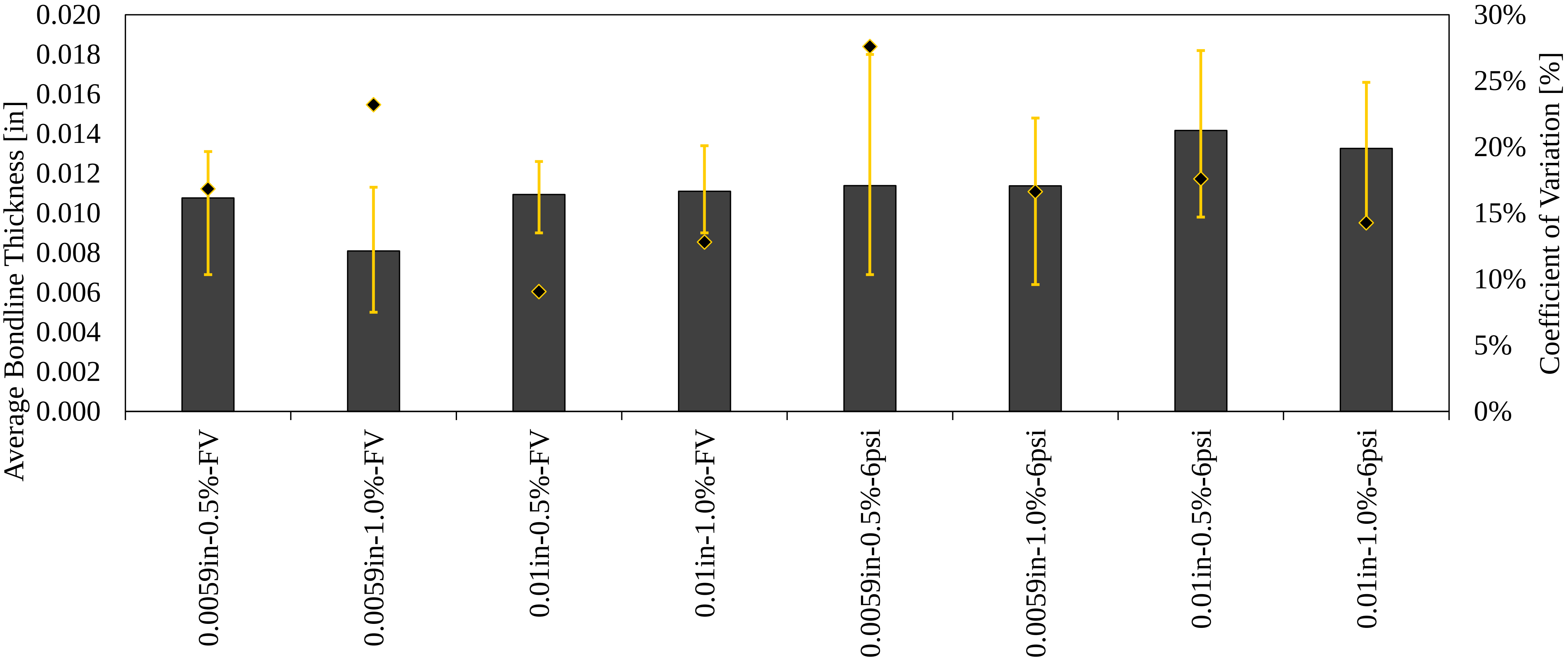


0.01GB-0.5PCT-6PSI-X (Side 1)



0.01GB-1.0PCT-6PSI-X (Side 2)

Bondline Control Mechanism



Summary and Conclusion

- Quality assurance through water contact angle measurements for substrate provide reliable data to ensure the substrate preparation is acceptable
- Surface preparation to bonding time assessment provide the state change substrates go through when exposed to environment. This can be used to fine tune the bond process.
- Technicians variability assessment is critical in understanding the sensitivity of some of the manual labor critical activities. Understanding the variability is critical to ensure proper training is provided.
- Exposure duration and configuration in peel ply removal technique show variation in the bond performance. Investigations are currently underway to evaluate the reason for the PP removed exposed substrate showed slightly higher properties.
- Polyester peel ply showed a change in the strength and failure mode when exposed to thermal cycles.

Looking Forward/Future Work

- **Future Works**
  - Generate bond process protocols for
    - Selecting compatible substrate and adhesive combinations for a robust bond structure
    - Provide guidance on protocol development for cure process related activities
    - Look into other surface preparation methods and look into critical parameters
- **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 to ensure the integrity of the final bonded product

Summary

Evaluation of Peel Ply Removal and related parameters

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Road Map

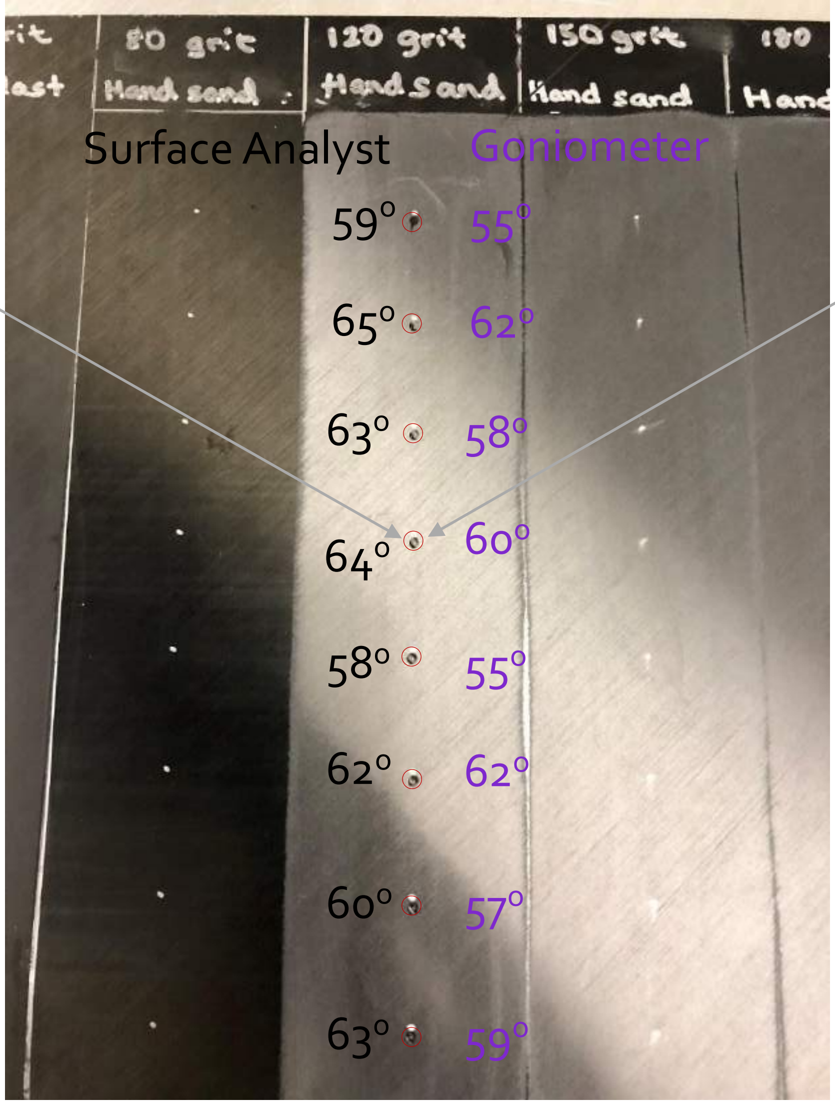
Material Databases Adhesive & Substrates → Preliminary Screening of Materials and Processes → Development of Bond Process Qualification Protocols → Certification of Bonded Structure

- Adhesive and Substrate material database
  - Ex – NCAMP material qualification databases (substrates, adhesive, etc.)
- Evaluate **physical, thermal and mechanical** properties
- Evaluate the operational **environmental envelope.**
- Preliminary material **processing parameters**.

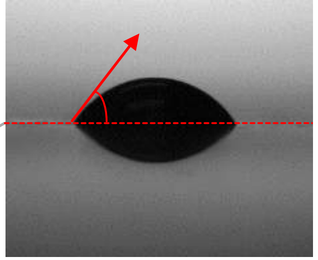
- Selection of Bond System Parameters
  - Substrate ✓
  - Adhesive ✓
  - Surface Preparation Methodology
  - Adhesive Processing

Goniometer vs. Surface Analysis Comparison

Surface Analyst

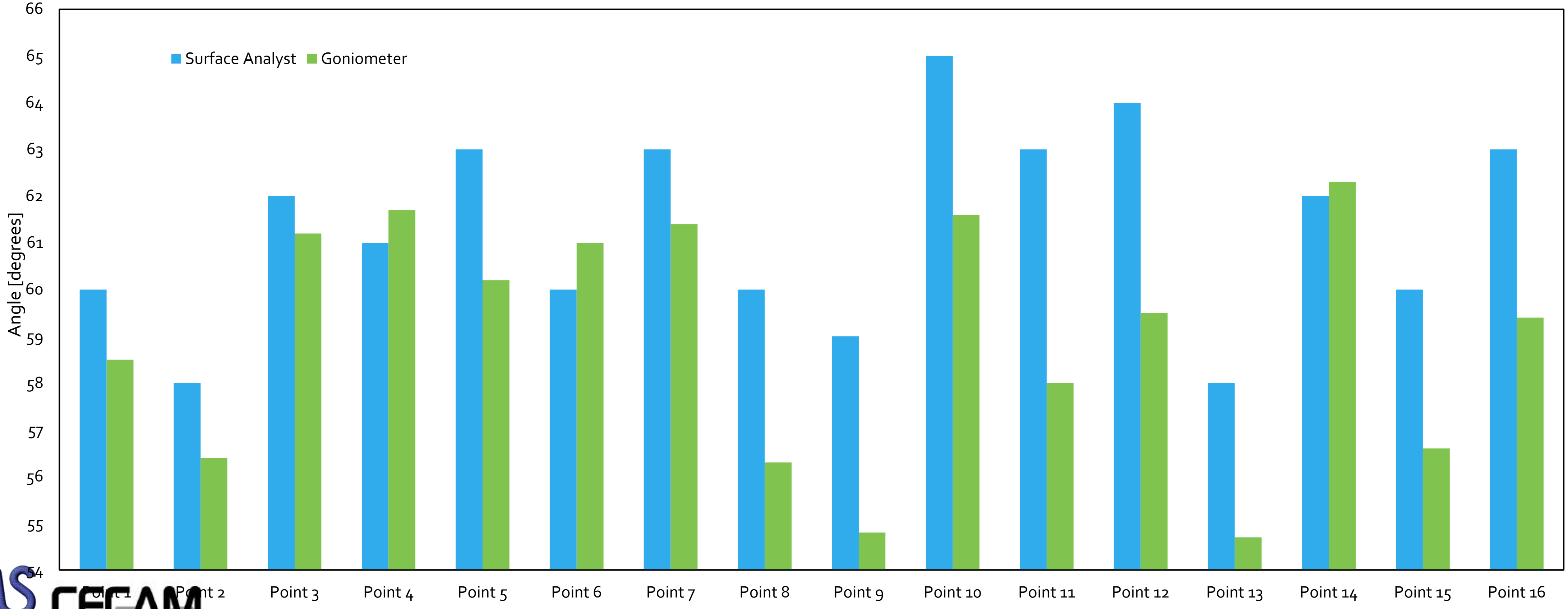


Goniometer

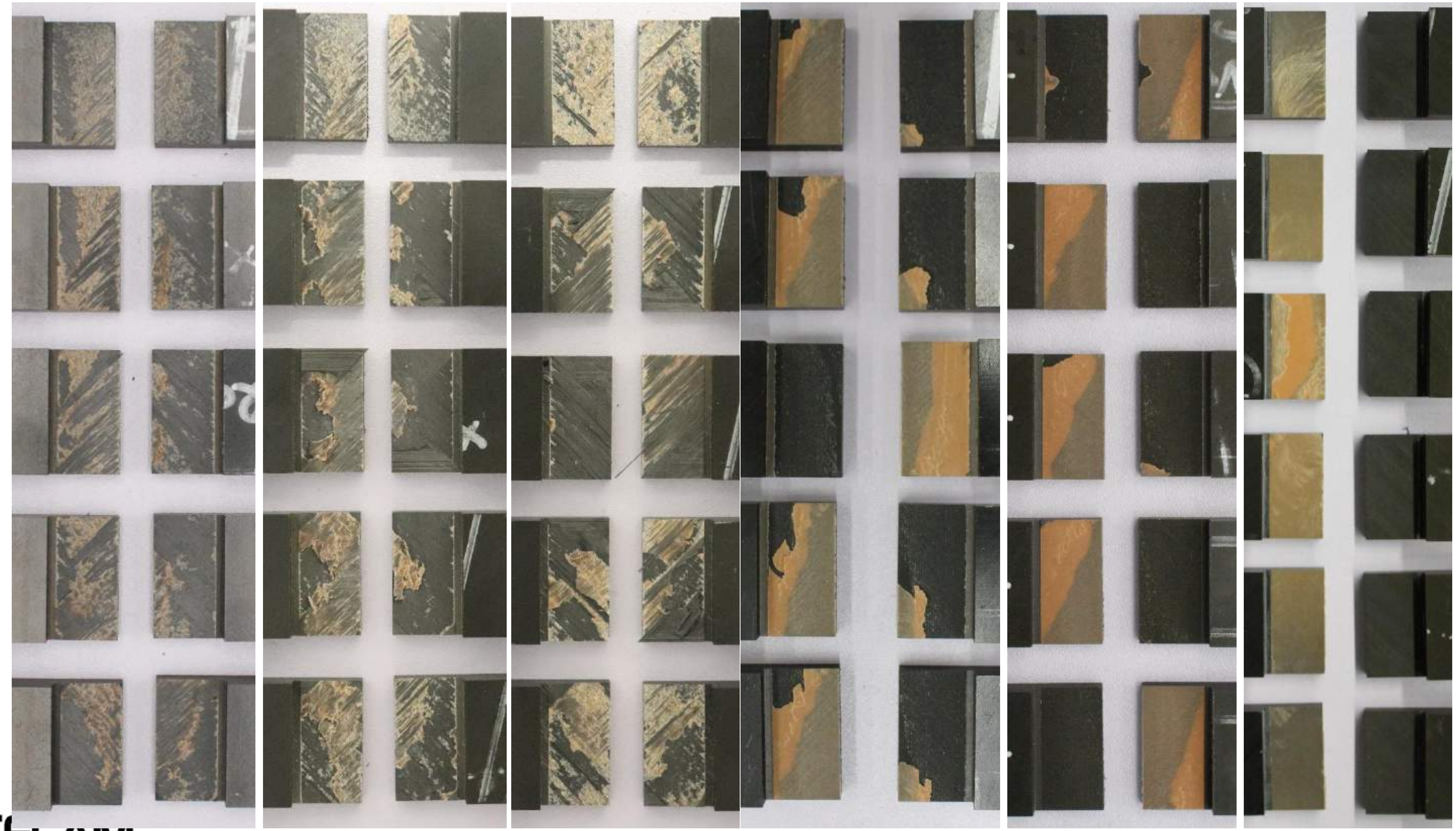


Goniometer vs. Surface Analysis Comparison

- Average difference of 2 degrees between the SA and goniometer measurements, with a maximum difference of 4 degrees.



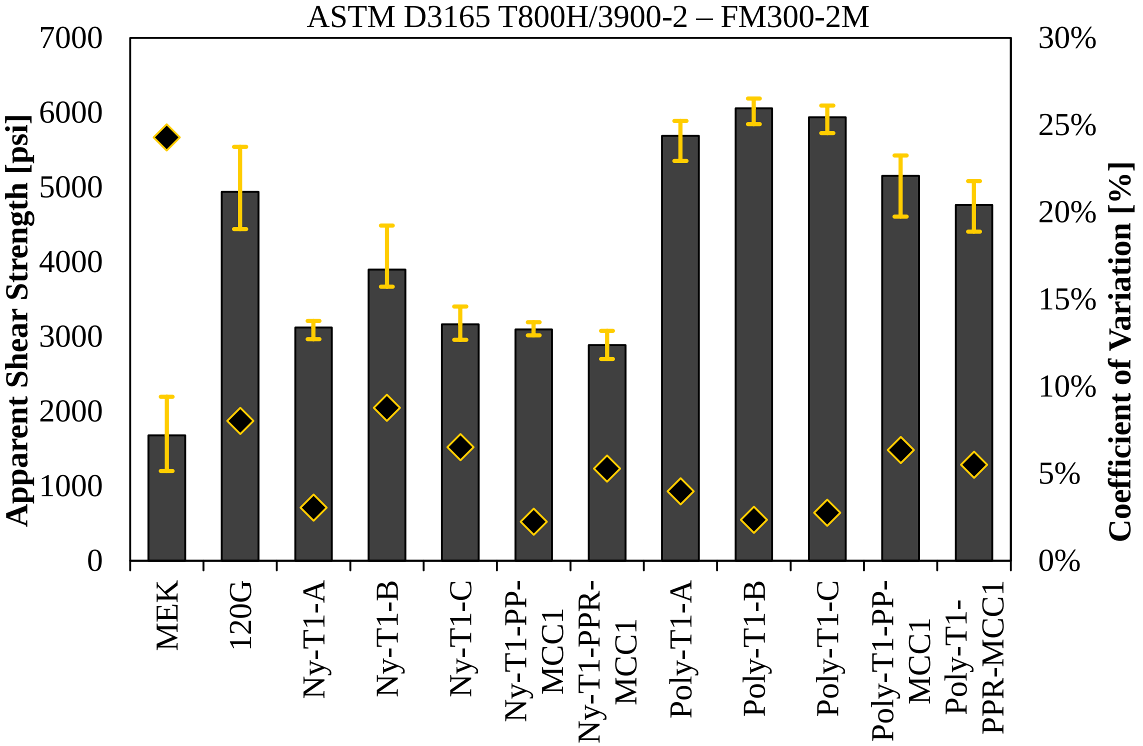
Contact Angle vs. Bond Strength



33 degrees | 38 degrees | 47 degrees | 64 degrees | 80 degrees | 94 degrees

Peel Ply study – Single Lap Shear

- Apparent average shear strength for composite D3165 substrates bonded with FM300-2M



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120G	Hand sanding with 120 grit			
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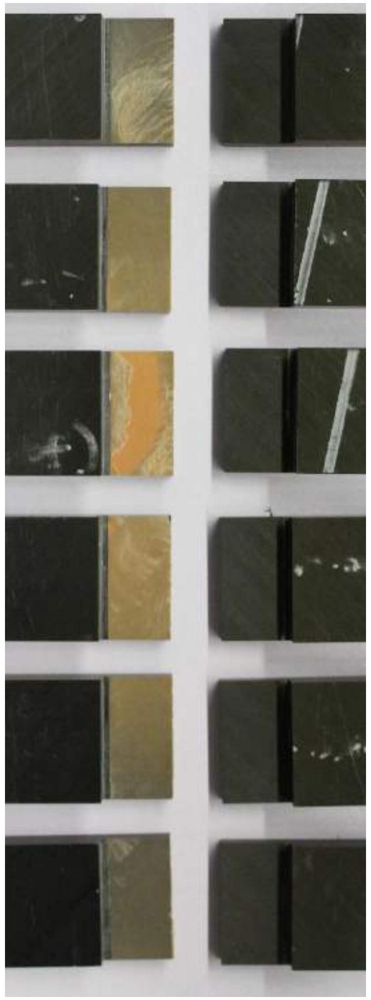
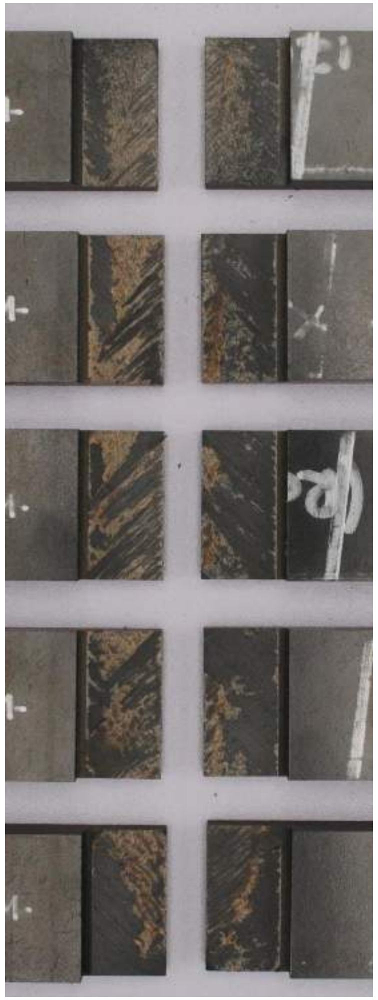
			PPR-MCC2	Peel ply removed, two post cure thermal cycles

D3165 Failure Modes - FM300-2M



MEK



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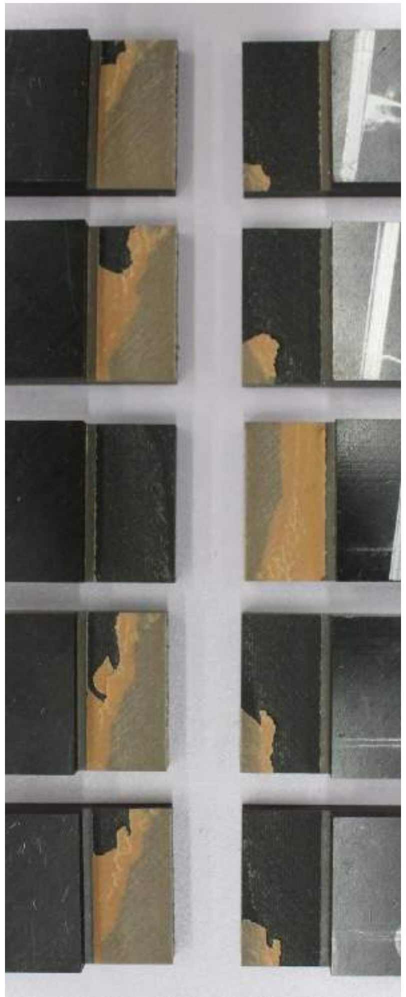
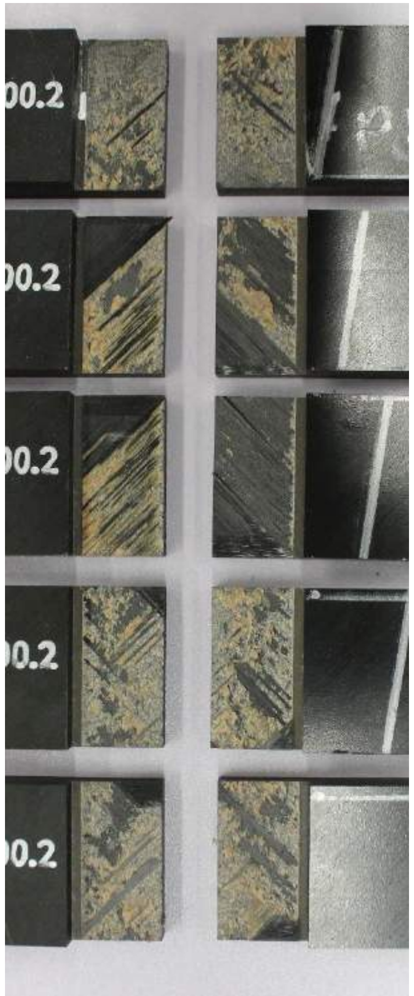
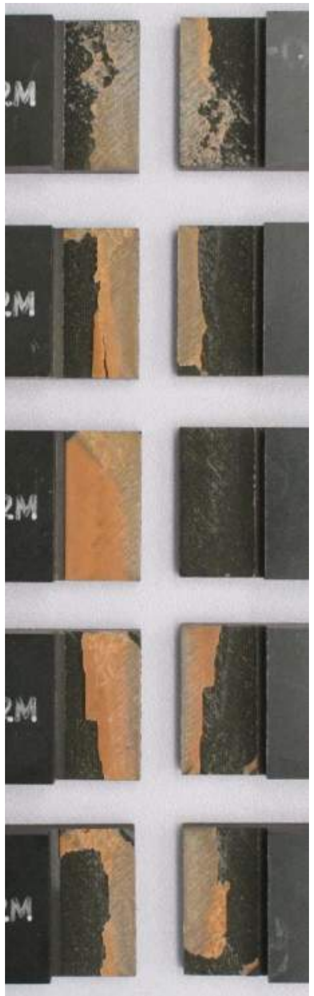


Ny-T1-A



Poly-T1-A

D3165 Failure Modes - FM300-2M



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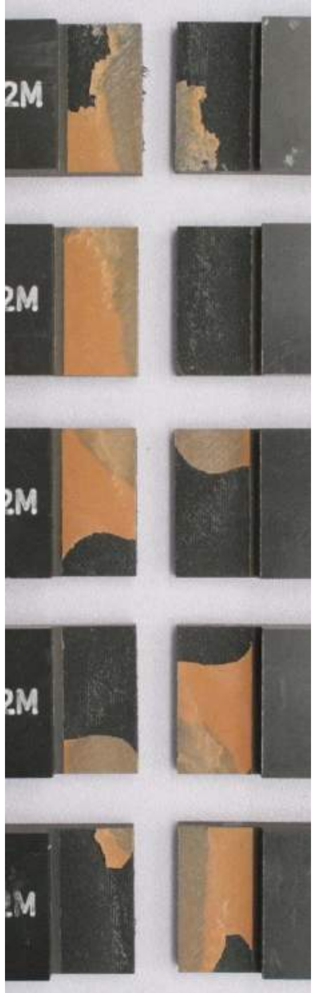

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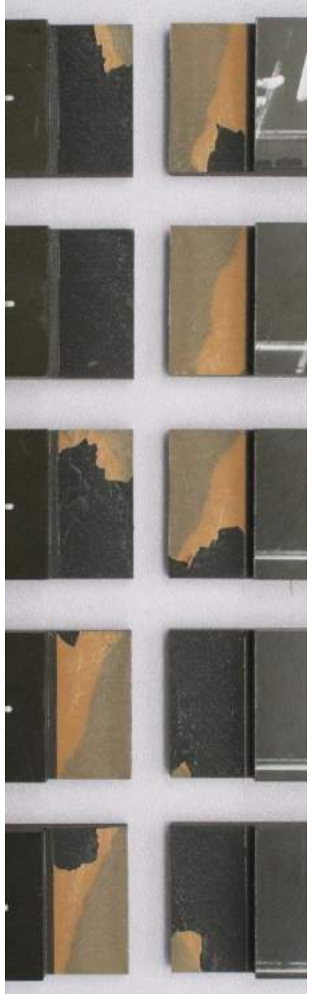
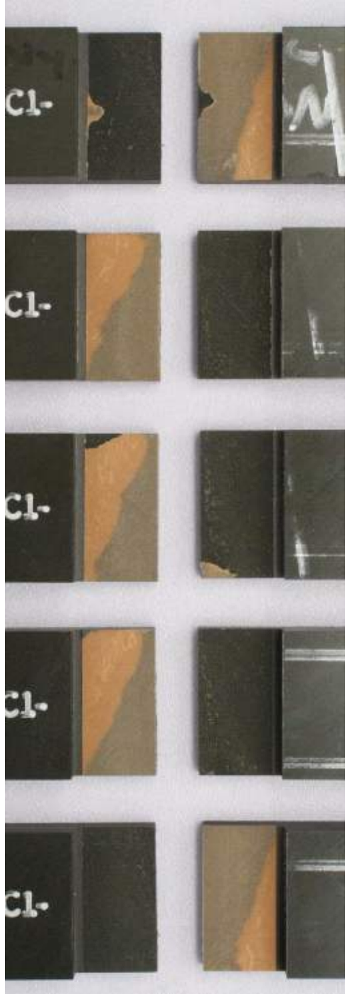
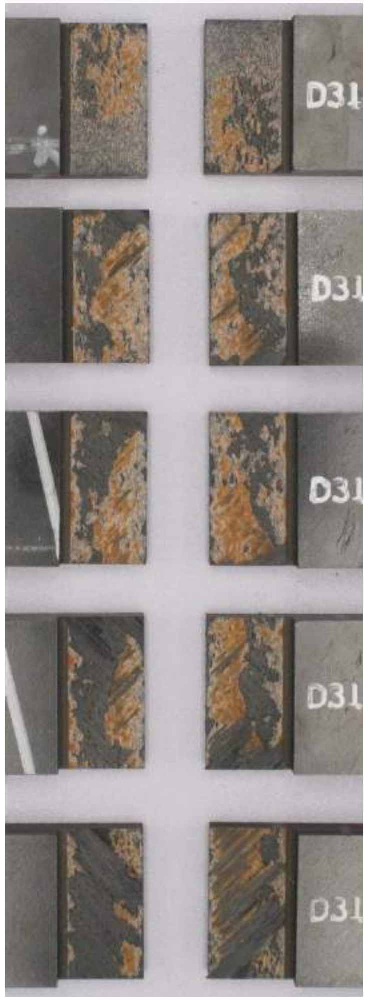


Ny-T1-PP-MCC1



Ny-T1-PPR-MCC1

D3165 Failure Modes - FM300-2M



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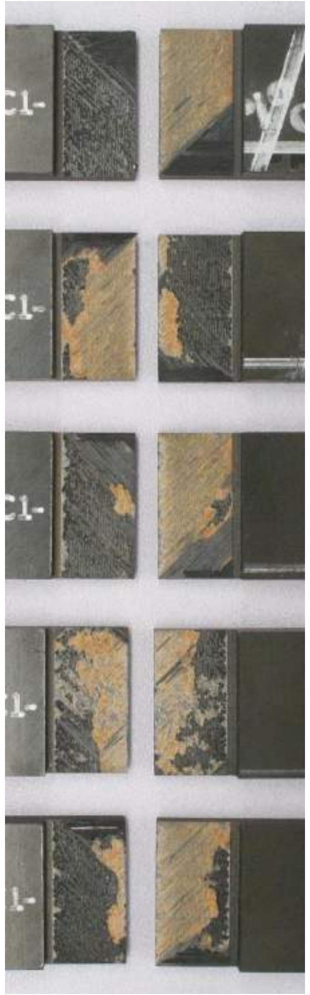
Poly-T1-C



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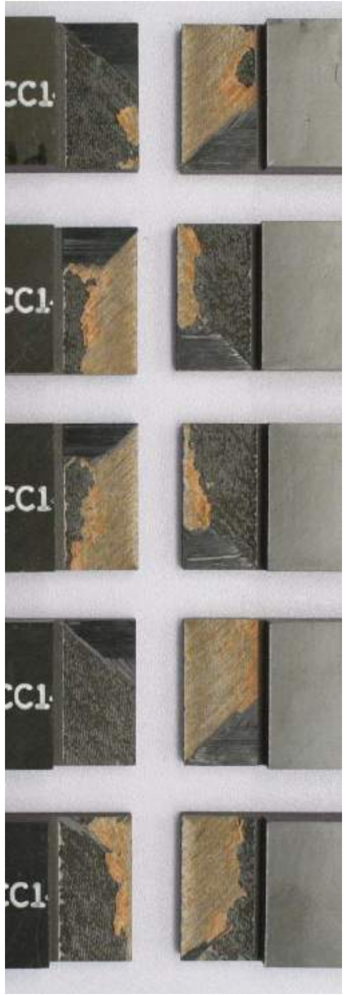
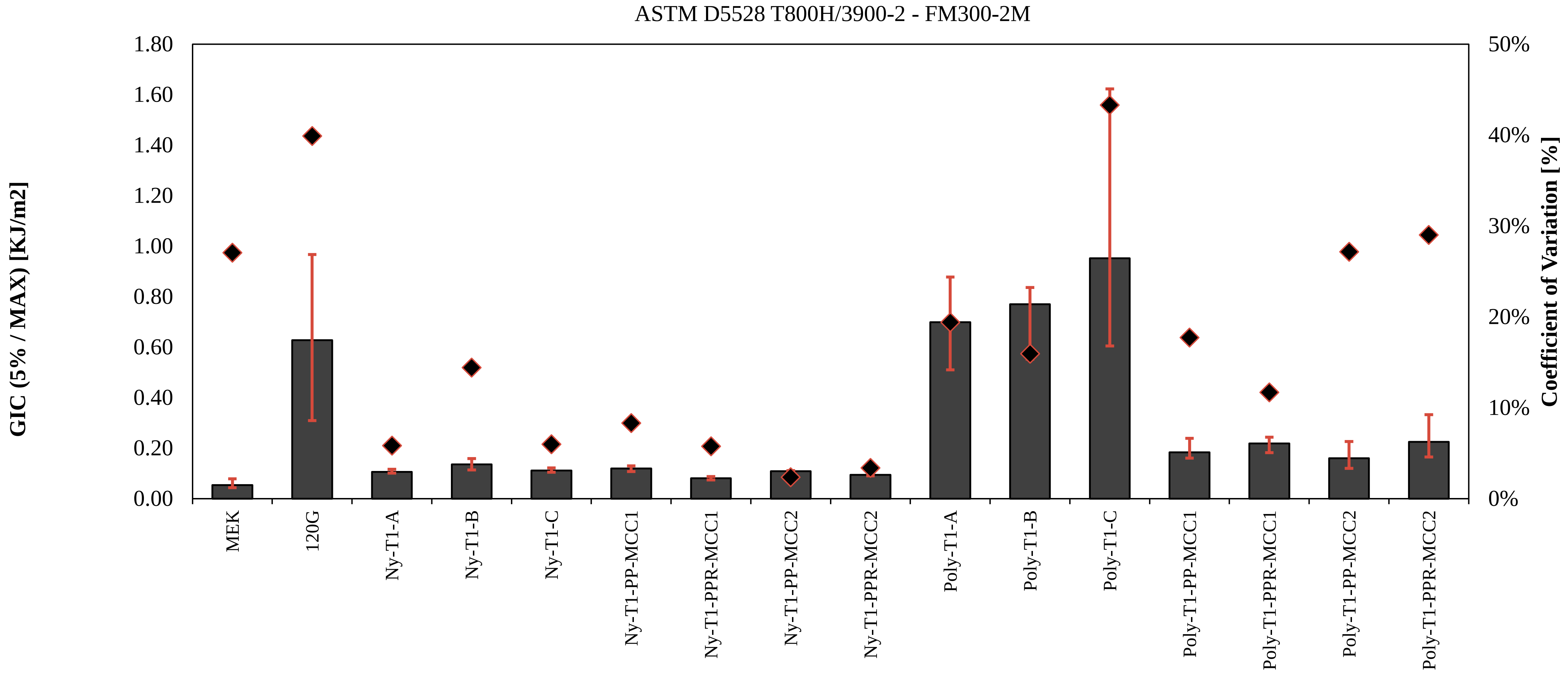
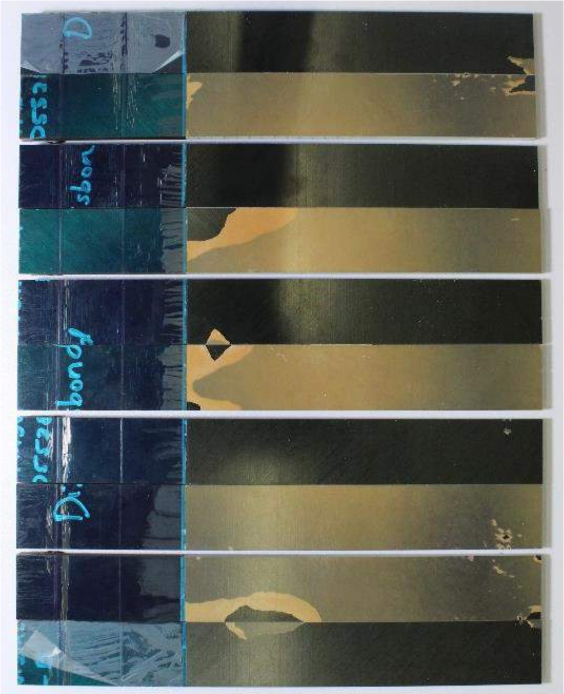


Poly-T1-PPR-MCC1

Peel Ply Mode I Failure Mode Pictures



D5528 Post Test - FM300-2M

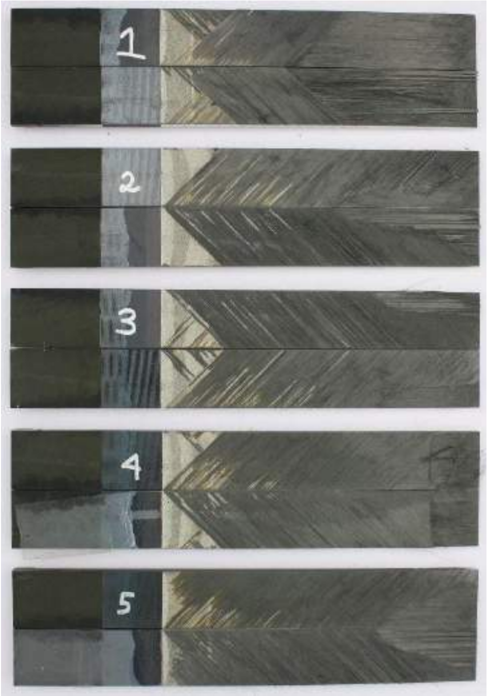


MEK

120G

D5528 Post Test - FM300-2M

Poly-T1-A



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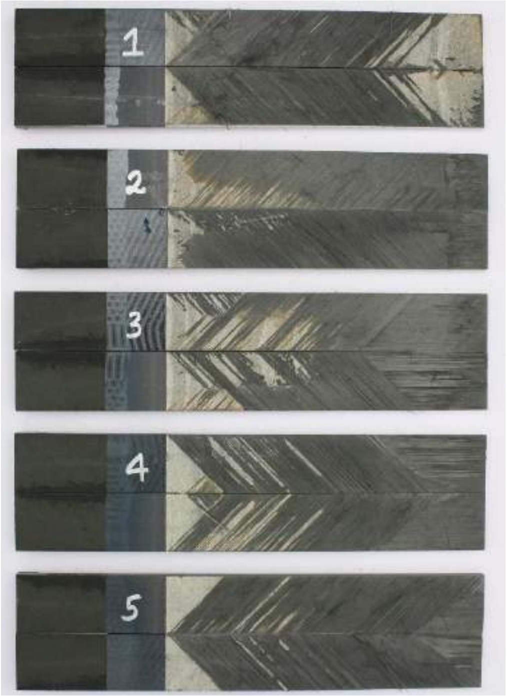

Poly-T1-C

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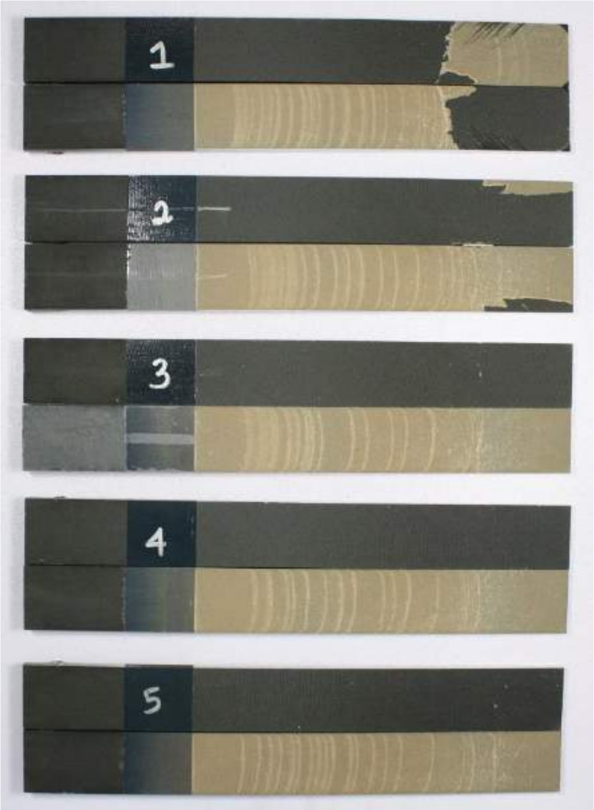
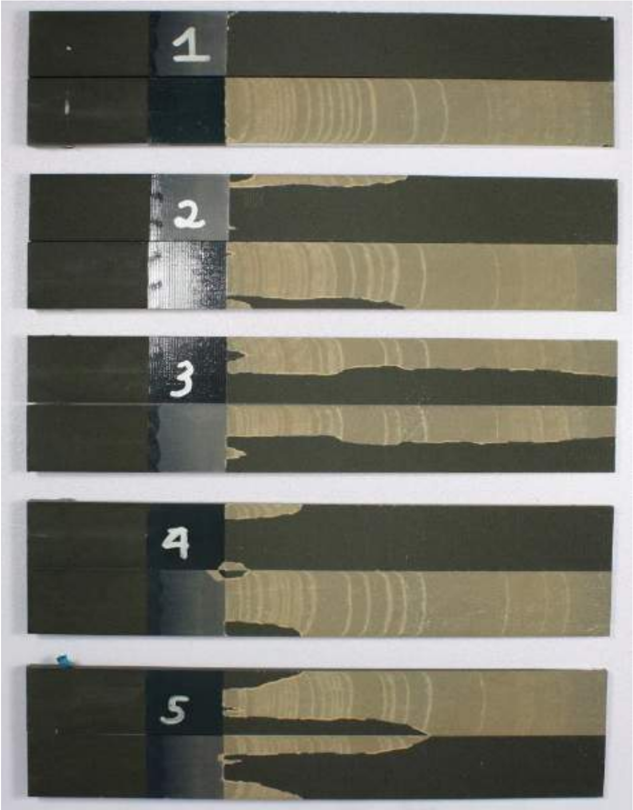
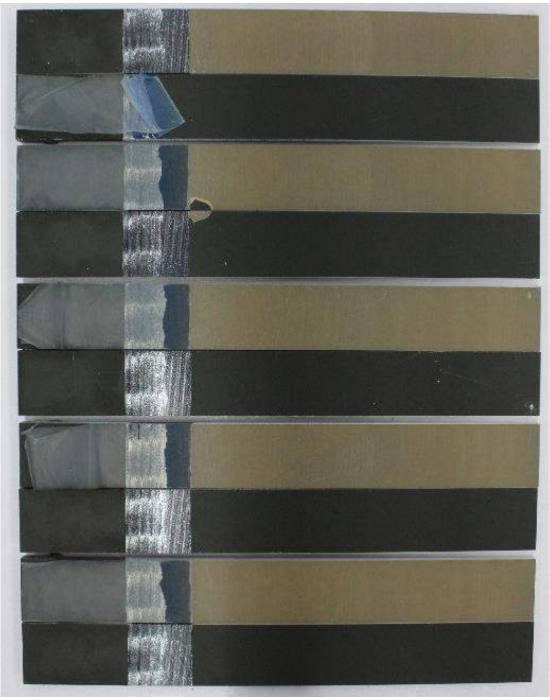


Poly-T1-PP-MCC1



Poly-T1-PPR-MCC1

D5528 Post Test - FM300-2M



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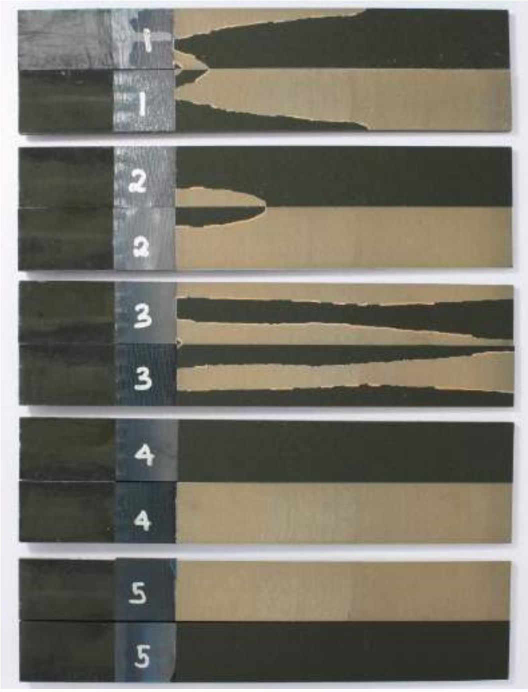

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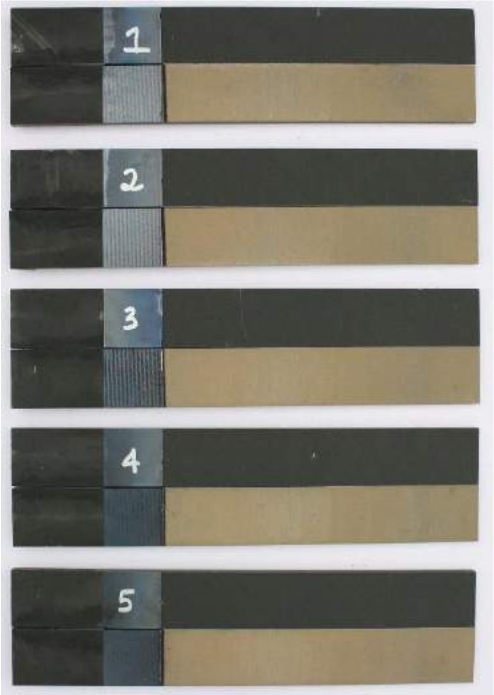
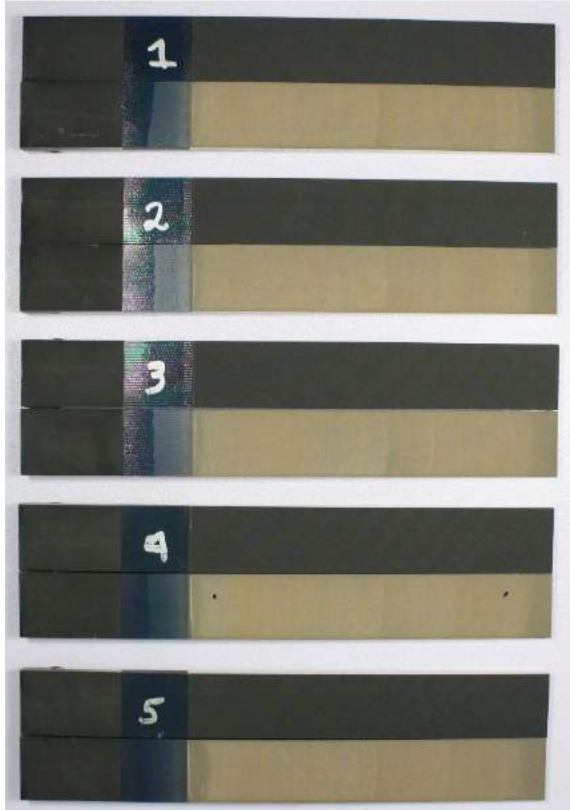
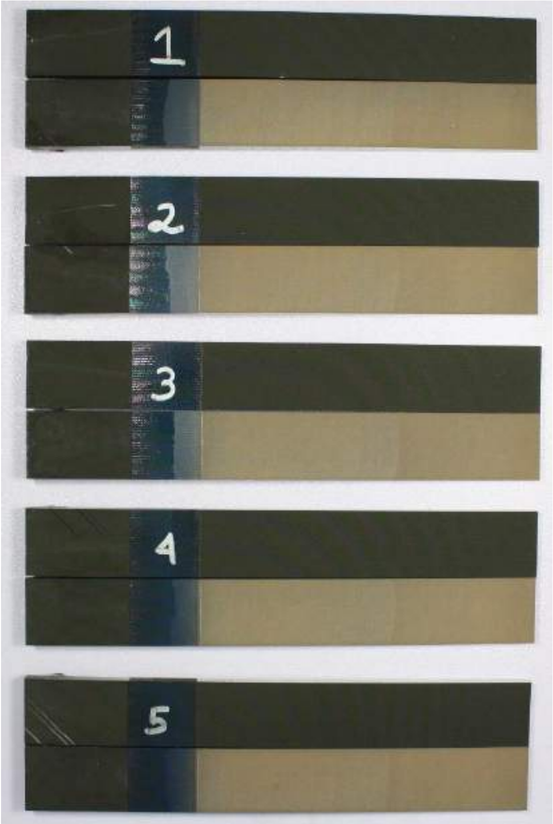


Ny-T1-C

D5528 Post Test - FM300-2M



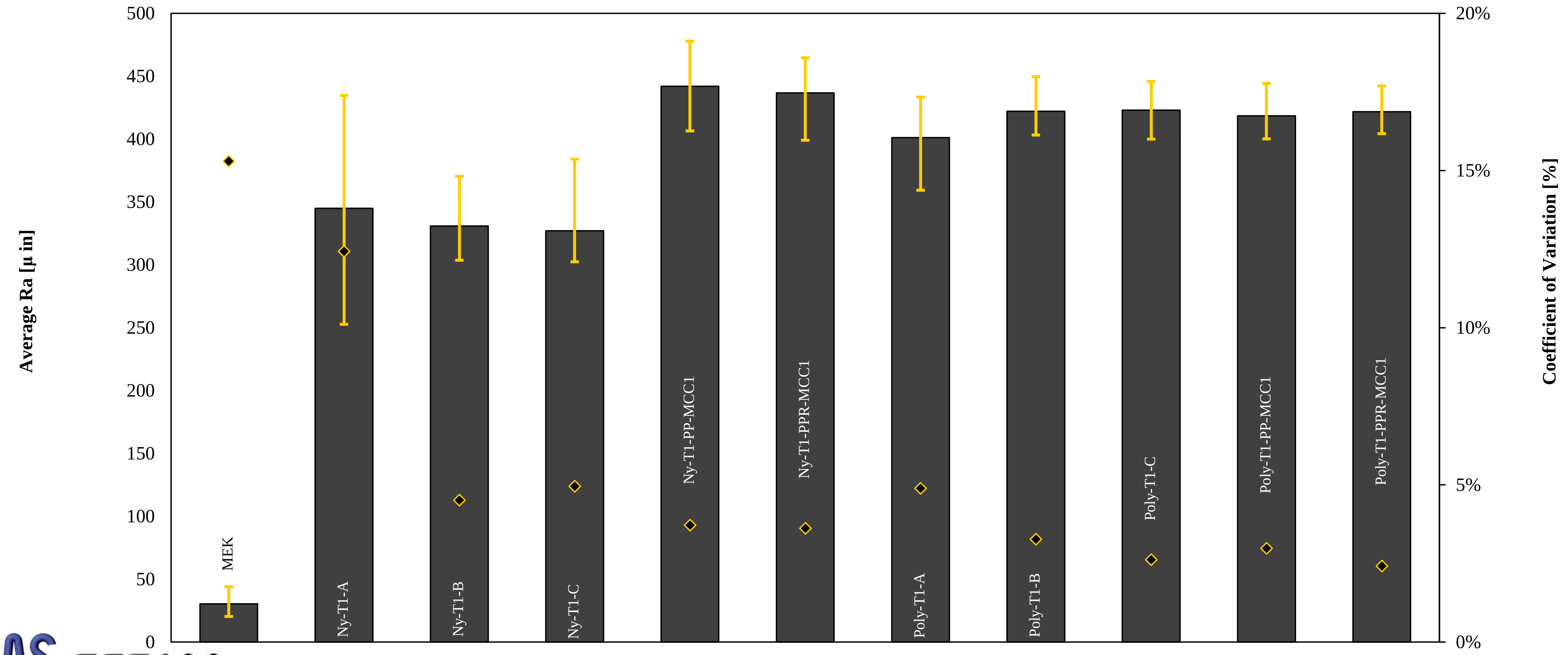
Ny-T1-PP-MCC1



Ny-T1-PPR-MCC1

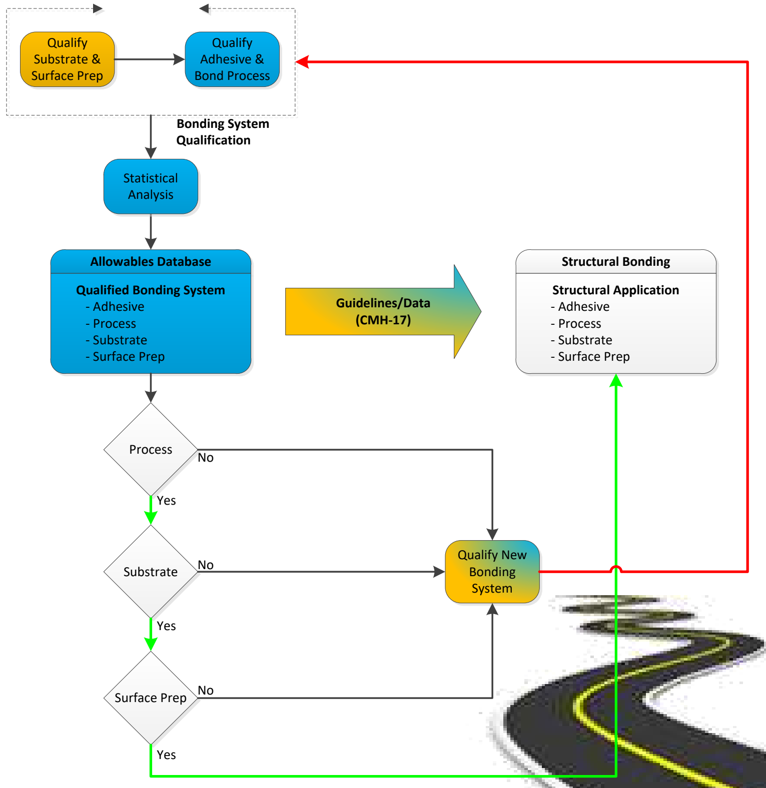
Peel Ply – Surface Roughness

- All measurements were taken in the 0° direction.

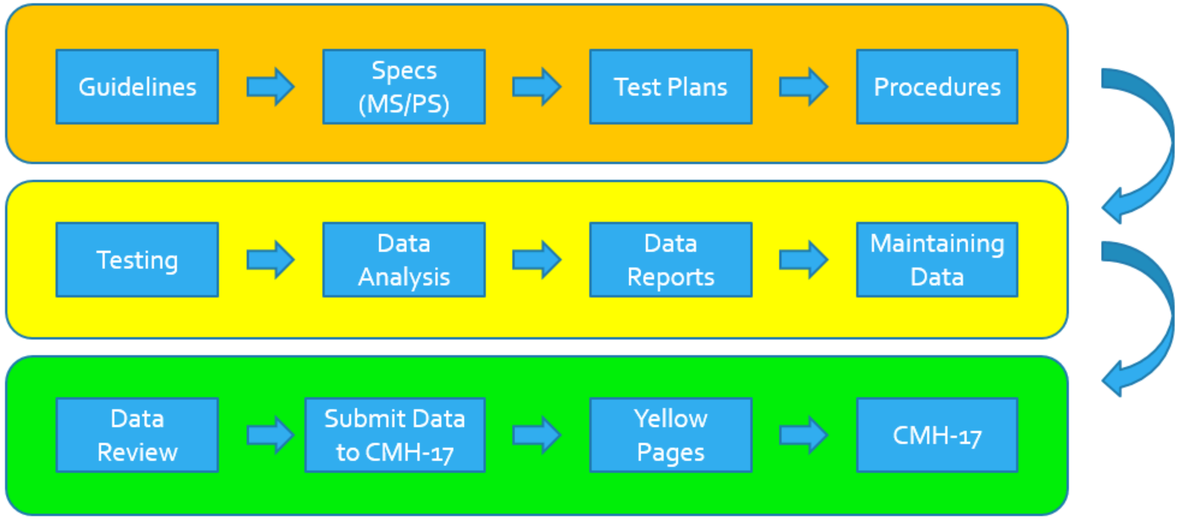


Road Map - Adhesive Qualification Guidance

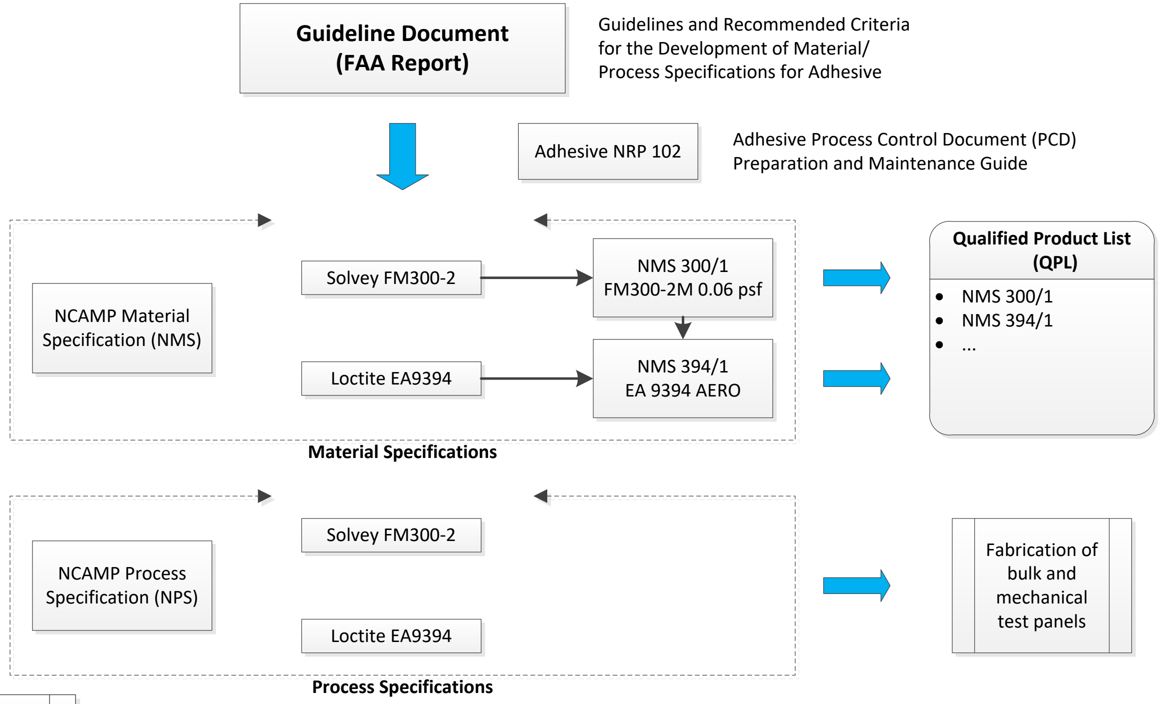
- Adhesive Characterization Qualification (ACQ)
  - Develop test matrices
    - Bulk physical, chemical, and mechanical test matrices
    - Adhesive (joint) mechanical tests
    - Fluid sensitivity
    - Equivalency tests
  - Develop databases
    - Select adhesive bond systems
    - Inclusion to CMH-17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



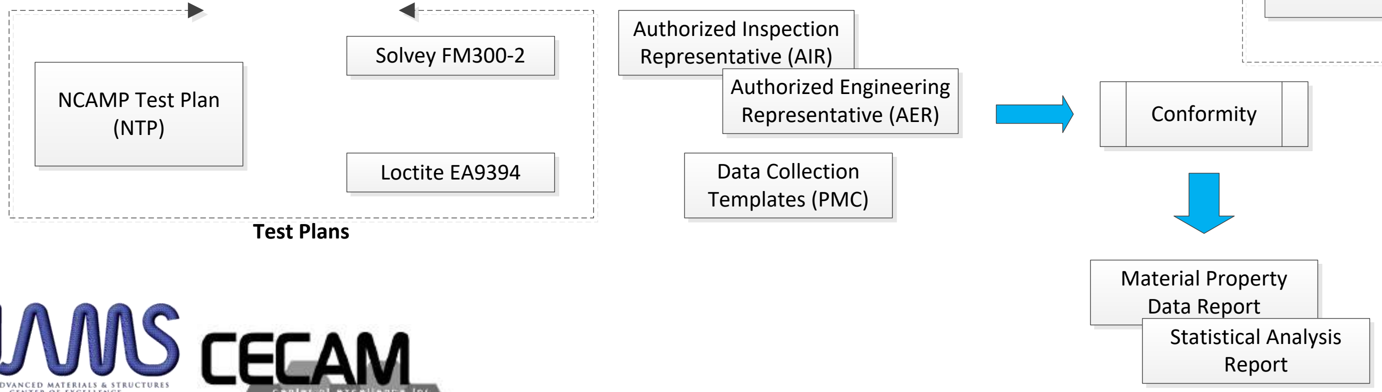
Road Map - Adhesive Qualification Guidance



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

Current Status

EA 9394

Property	Test Method	Test Environment	EA9394 Batch A C1	Batch A C2	Batch B C1	Batch B C2	Batch C C1	Batch C 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 C1	Batch A C2	Batch B C1	Batch B C2	Batch C C1	Batch C 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					

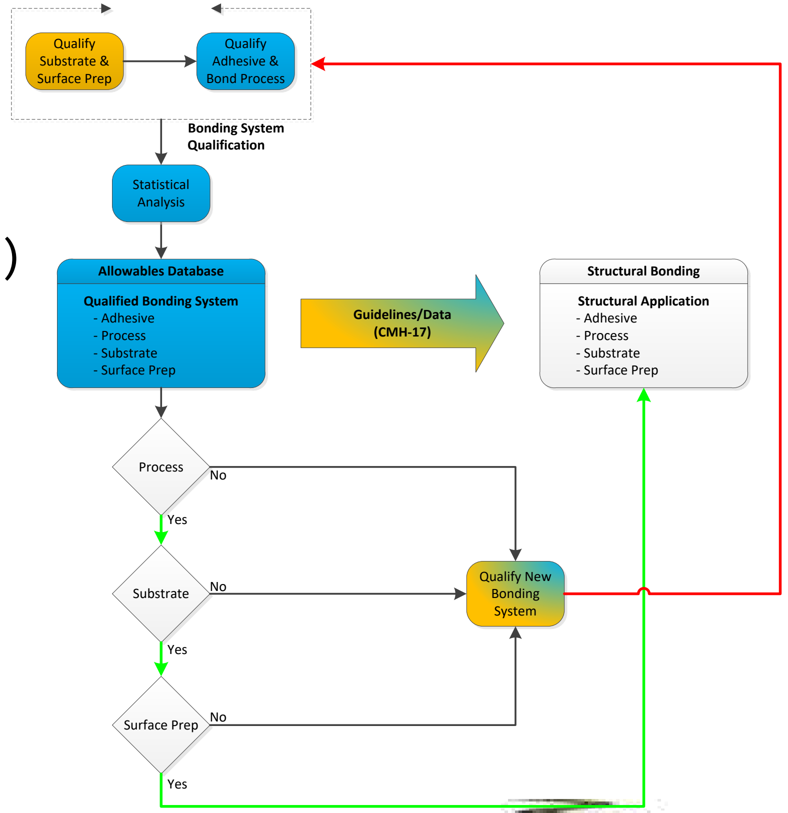
- Waiting on Adhesive Batch
- Specimen/Panel Bonding in Progress
- Specimen Machining in Progress
- Conditioning in Progress
- Testing in Progress
- Testing Complete

Look Forward

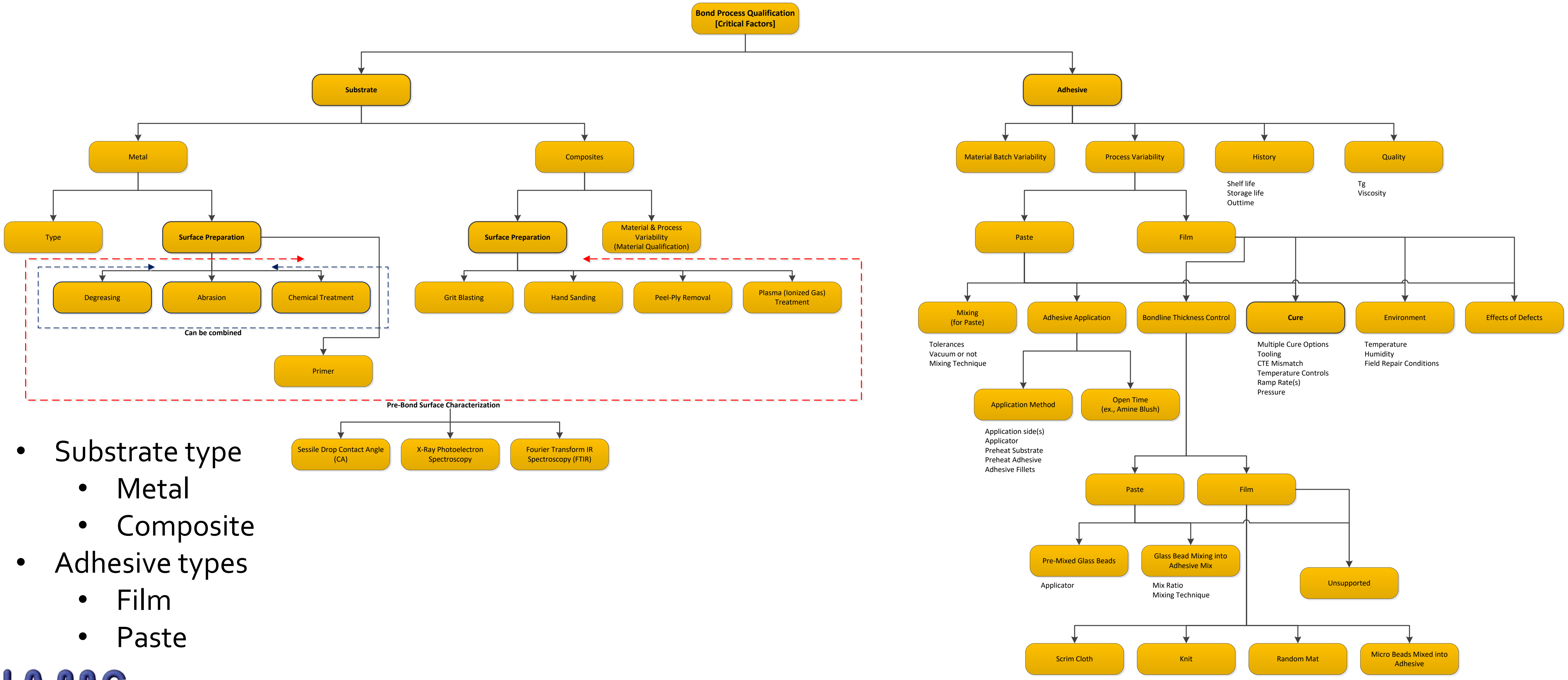
- **Future Activities**
  - Generate the B-Basis allowable for EA9394 and FM300-2M material systems
  - Focus on performing equivalency on adhesive materials.
  - Analyze failure modes for different test environments and report them accordingly
- **Benefit to the Aviation Community**
  - Guidance on 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

Substrate

1. Composite
2. Metal

Adhesive System

1. Paste
2. Film

Substrate Surface Preparation

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

Bonding Process/Curing

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

Substrate

1. Composite
2. Metal

Adhesive System

1. Paste
2. Film

Substrate Surface Preparation

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

Bonding Process/Curing

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 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

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

Compatibility

Mechanical

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

Physical

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

Thermal

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

Task 1 - Substrate and Adhesive Compatibility

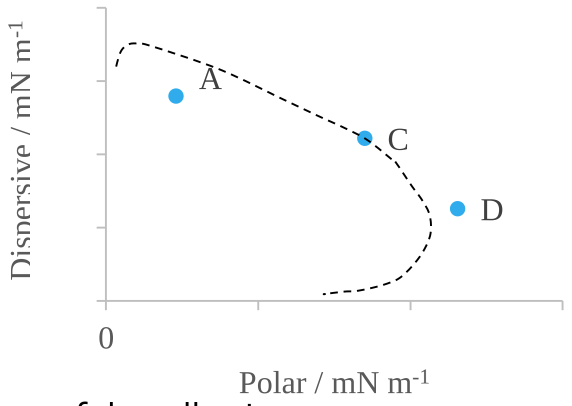
- Substrates
  - Carbon Fiber Composites
    - UNI – T800/3900-2
    - PW - T300/3900-2
  - Glass Fiber Composites
    - Fabric – Epoxyglass G10
  - Metallic
    - AL 2024-T3
    - Ti – Grade 2

- 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)

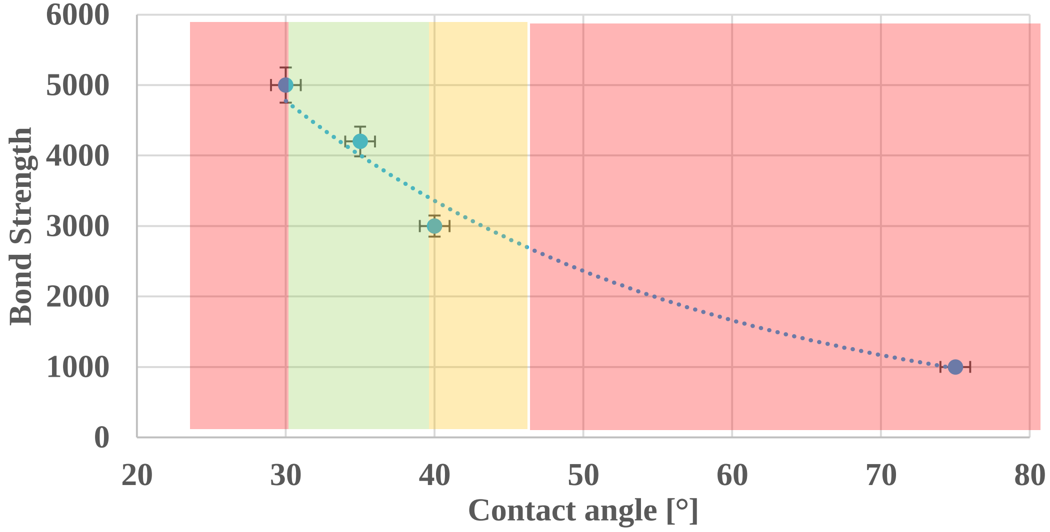
Task 1 - Substrate and Adhesive Compatibility Assessment
Physical Compatibility

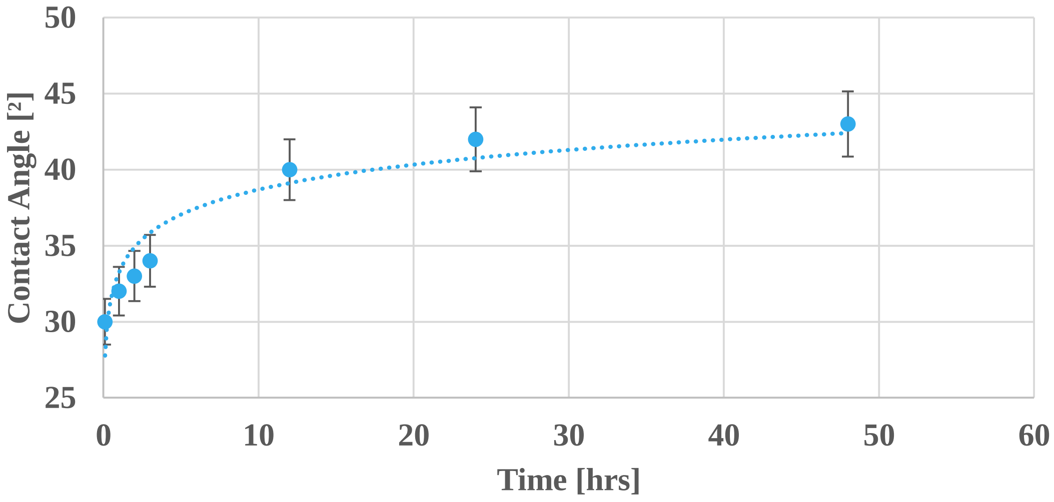
- Objective
  - **Generate guidelines to ensure the surface preparation + substrates are physically compatible for bonding.**
- Physical Compatibility
  - Surface morphology related tests for substrates
    - Surface roughness
    - Contact angle
    - Surface Energy
  - Wettability envelope development
    - 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

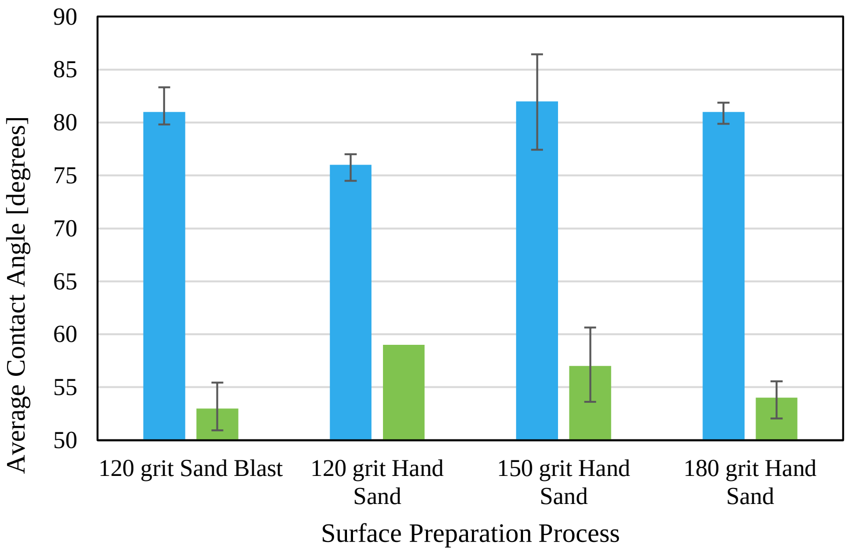
- Develop the relationship between the
  - Contact angle of substrates vs. bond quality.
  - Elapsed time vs contact angle
- Use in-situ surface energy measurement techniques (BTG-Labs - Surface Analysts) to assess the substrate characterizations
- Repeat the process for a given adhesive for different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

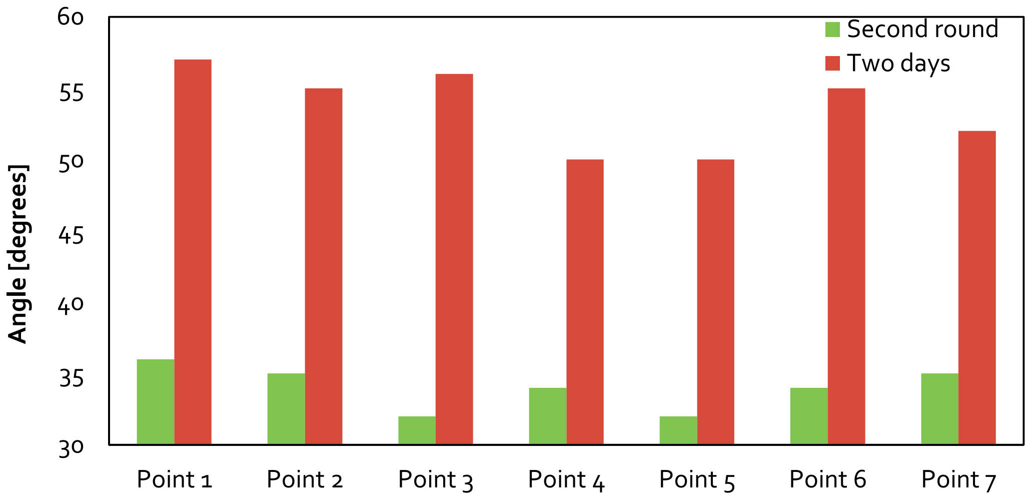




Task 1 - Substrate and Adhesive Compatibility Assessment
Physical Compatibility – Current Status

- 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

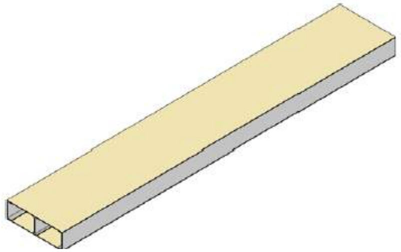
- Objective
  - **Generate guidelines to ensure the thermal properties of substrates and adhesives are compatible for bonding and during service life.**
- Thermal Compatibility
  - CTE mismatch
    - Between substrates and adhesives cured at elevated temperatures
    - CTE mismatch in bonded structures 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)

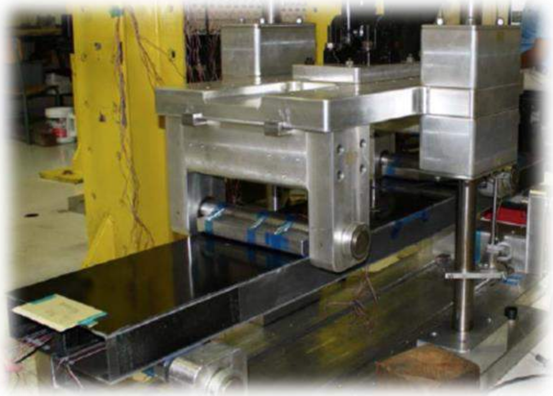
In - Progress

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





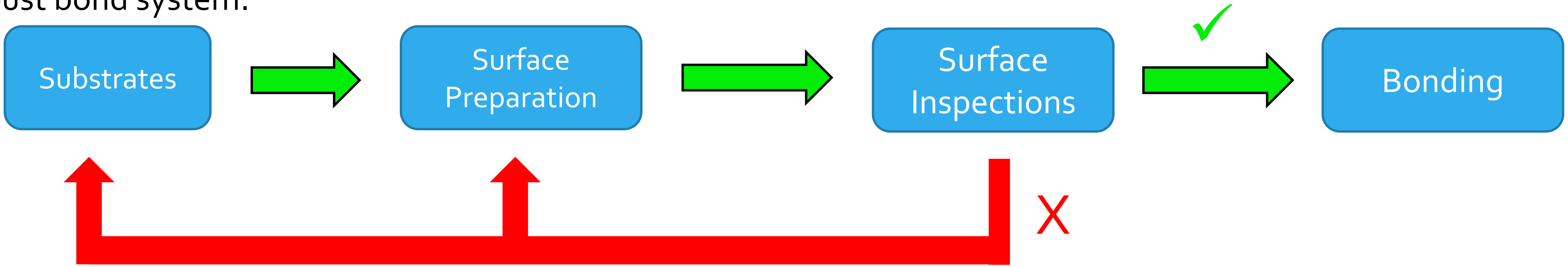
Task 1 - Substrate and Adhesive Compatibility Additional Tasks

- 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)

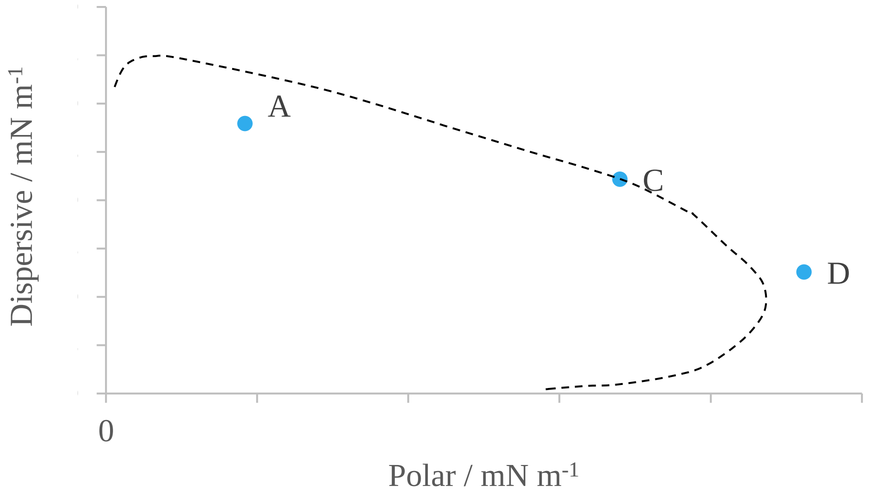
- Carbon Composites Substrates
  - Toray T800/3900-2
- Adhesive Systems
  - Film – FM300-2M
  - Paste – EA 9394
- Cure Cycles
  - FM300-2M – 250F for 2 hrs. at 40 psi pressure + full vacuum
  - EA 9394 – 150F for 1 hr. 6psi vacuum

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.

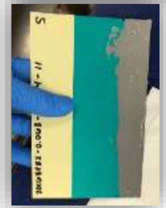
Summary of Activities

- 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%

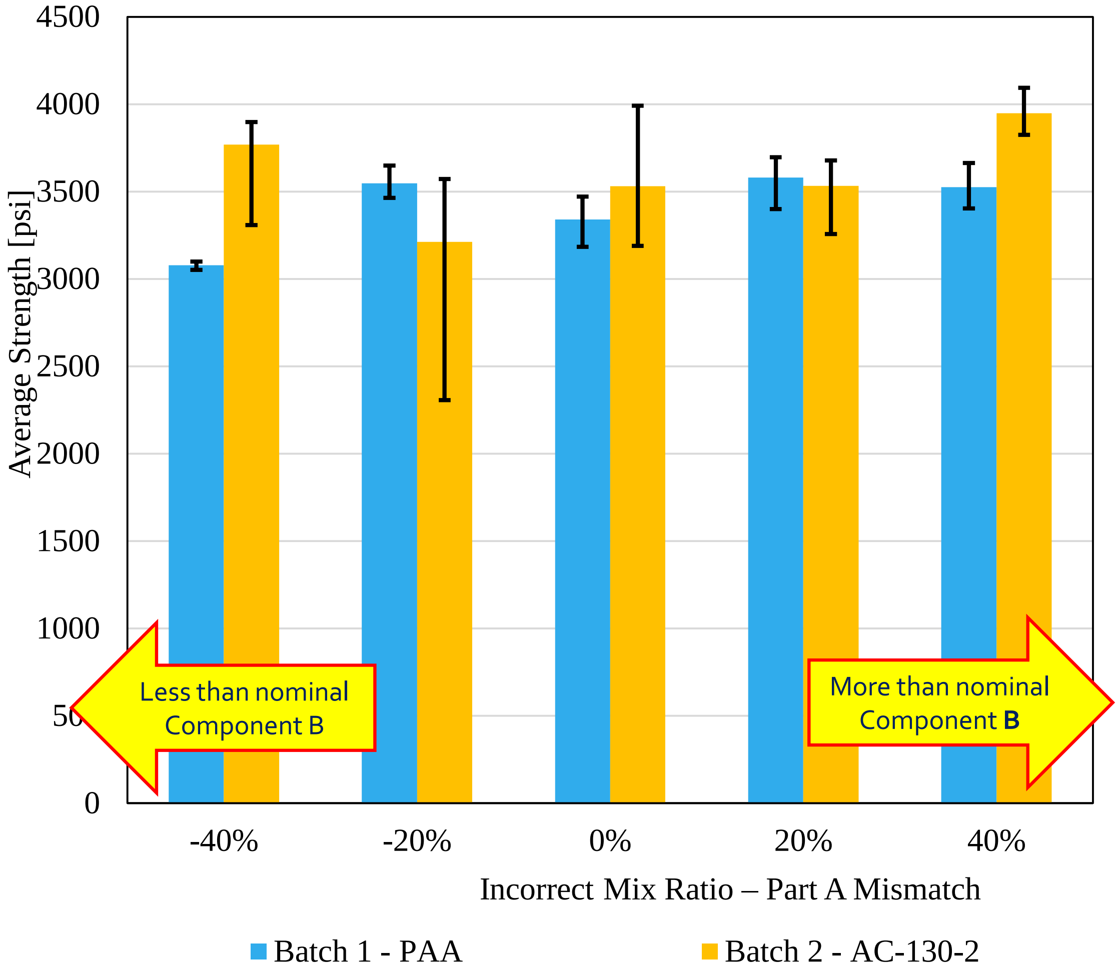
Adhesive Identification	Adherend Material	Percent Mismatch	Adhesive Quantity A [g]	Adhesive Quantity B [g]	0.005" Glass Bead Weight [g]
EA9394	Aluminum 0.063"	-40%	30.000	3.060	0.00331
		-20%	30.000	4.080	0.00341
		0%	30.000	5.100	0.00351
		20%	30.000	6.120	0.00361
		40%	30.000	7.140	0.00371

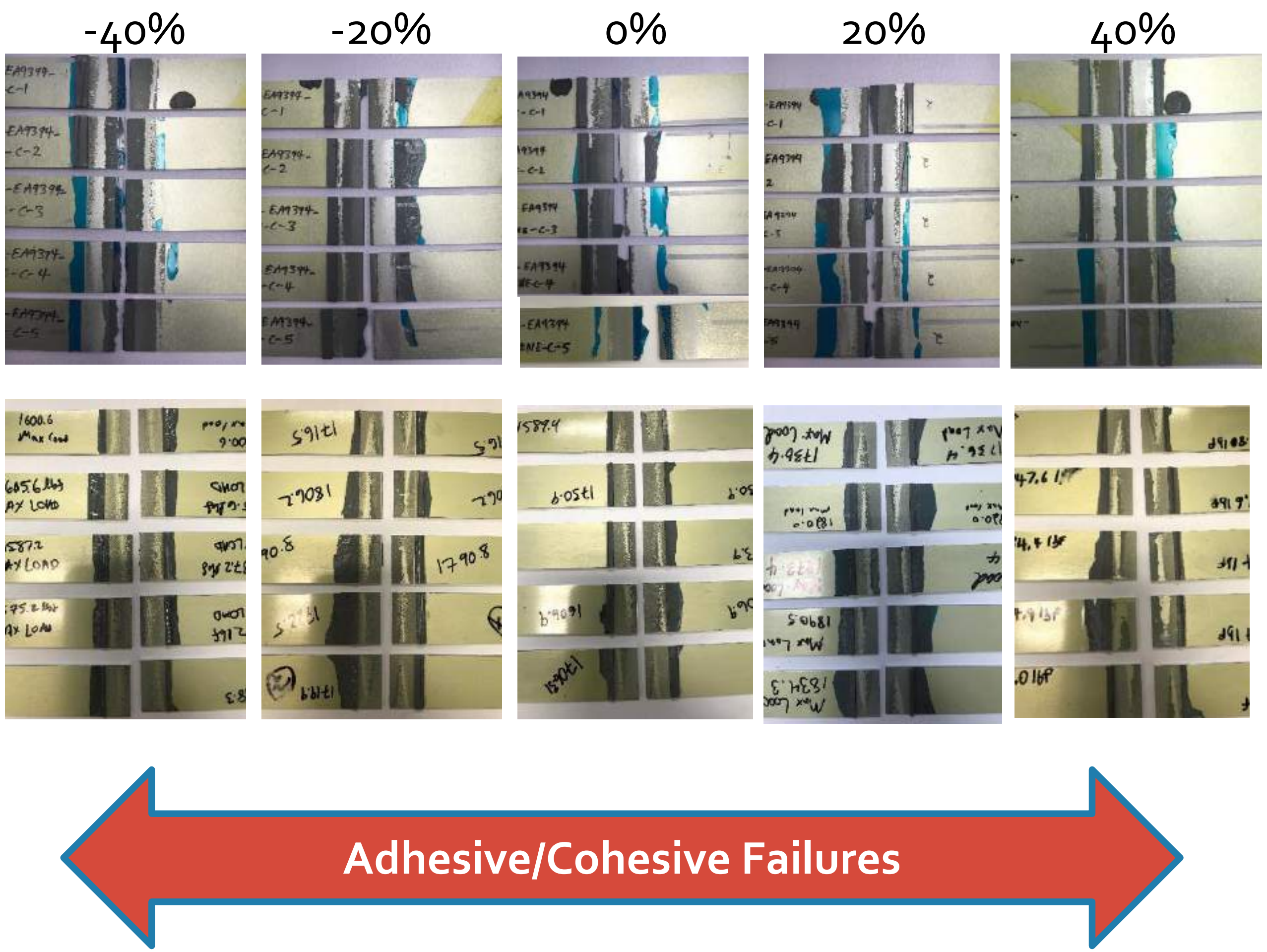





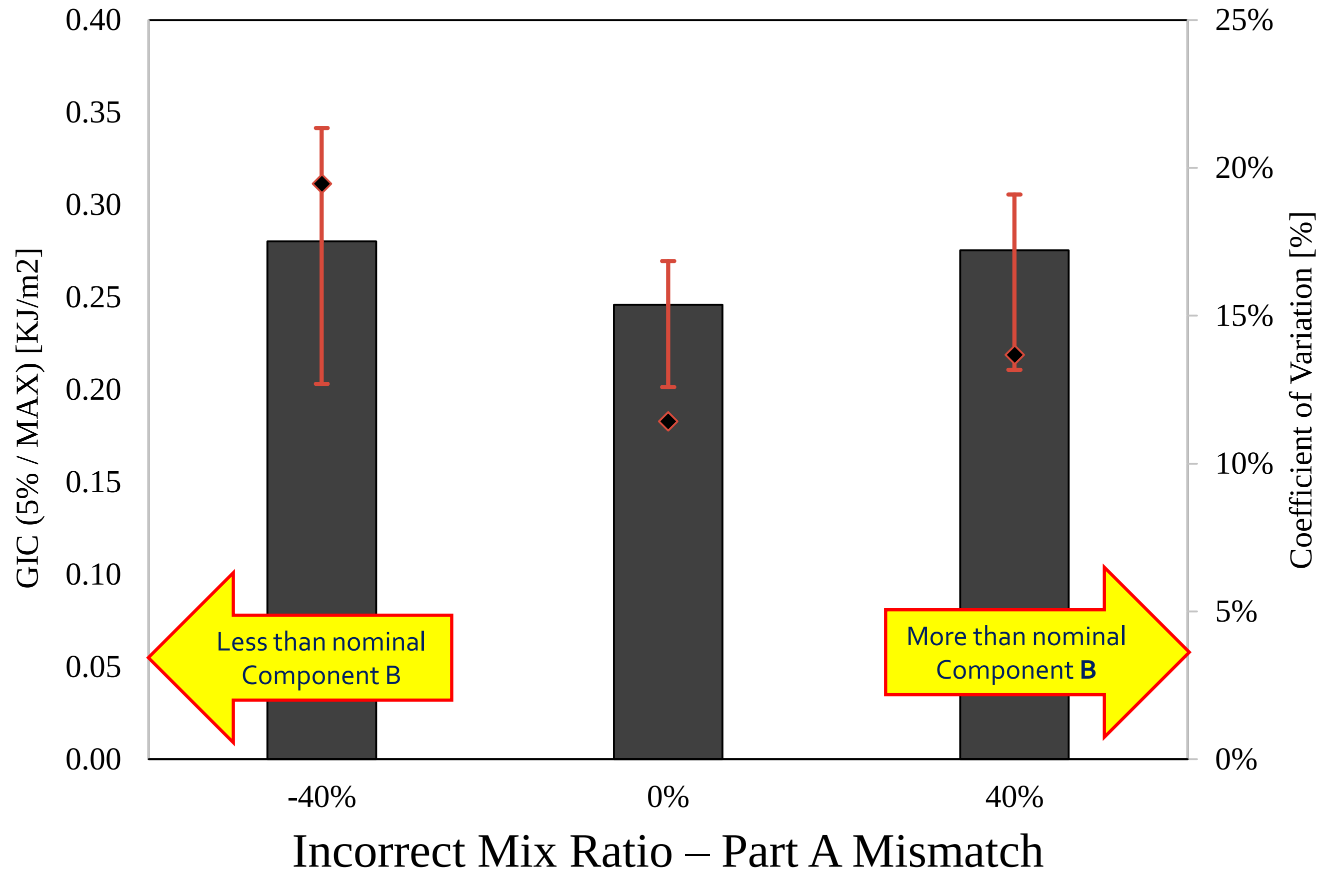
Effects of incorrect mix-ratio in two part paste adhesives

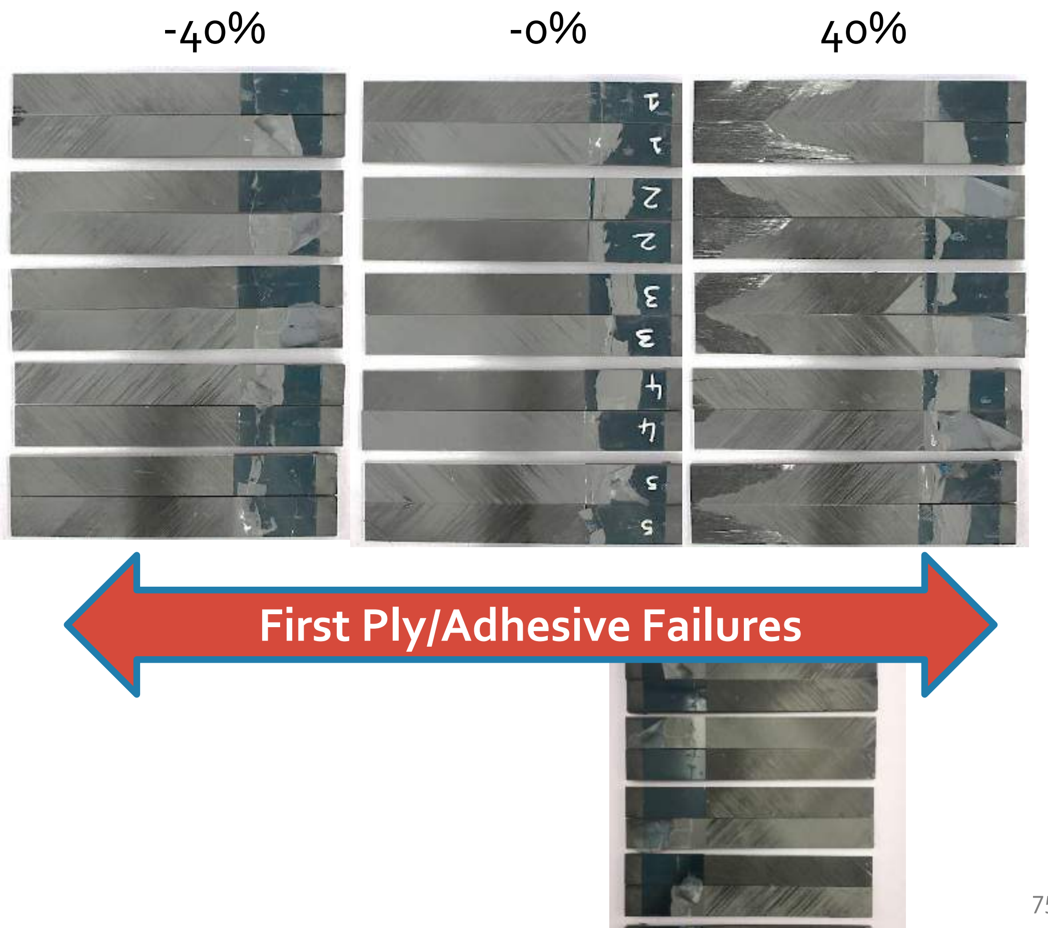
Test Results – Single Lap Shear – ASTM D1002



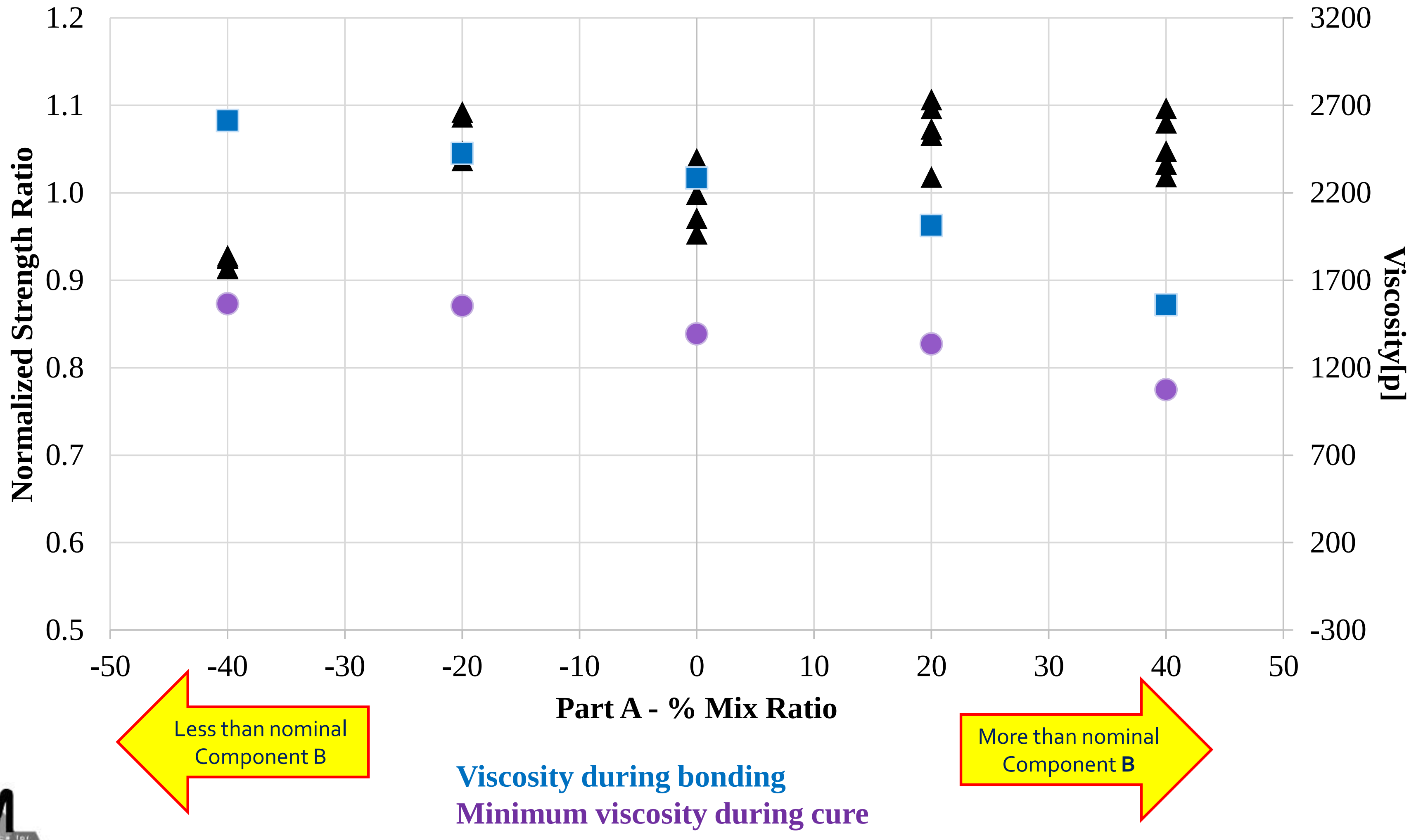


Effects of incorrect mix-ratio in two part paste adhesives
Test Results – Mode I – ASTM D5528



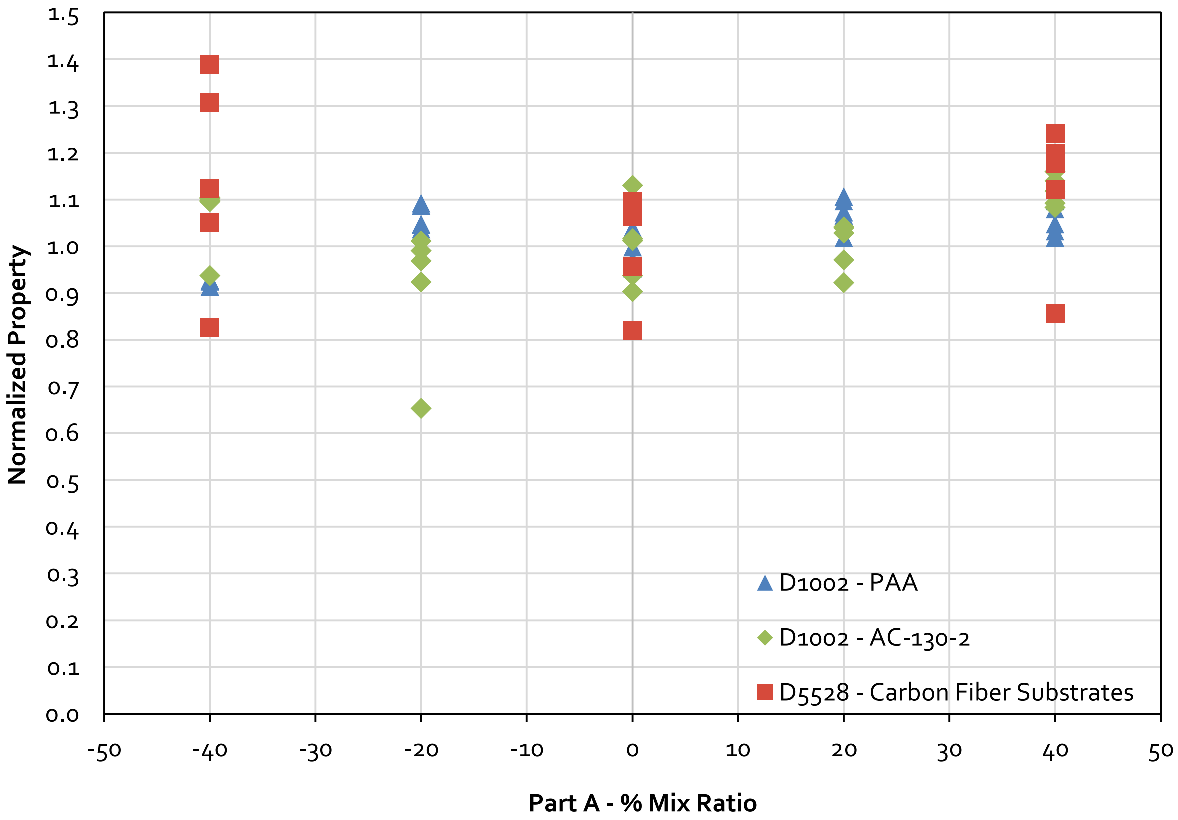


Effects of incorrect mix-ratio in two part paste adhesives
Test Results – Viscosity Response



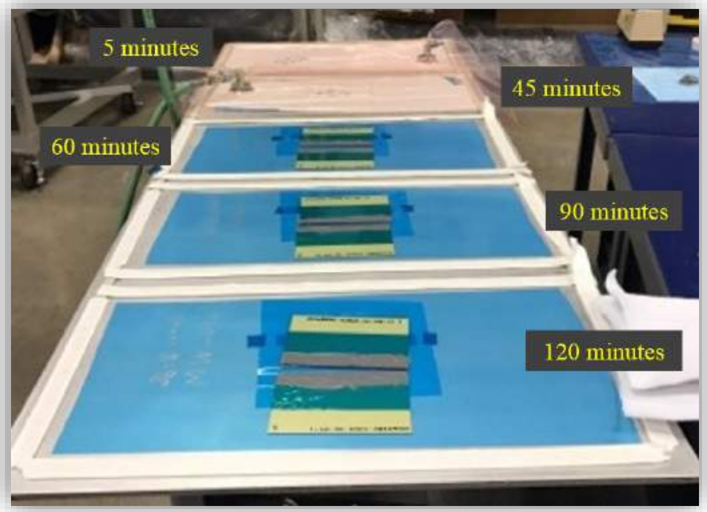
Effects of incorrect mix-ratio in two part paste adhesives
Test results – Summary

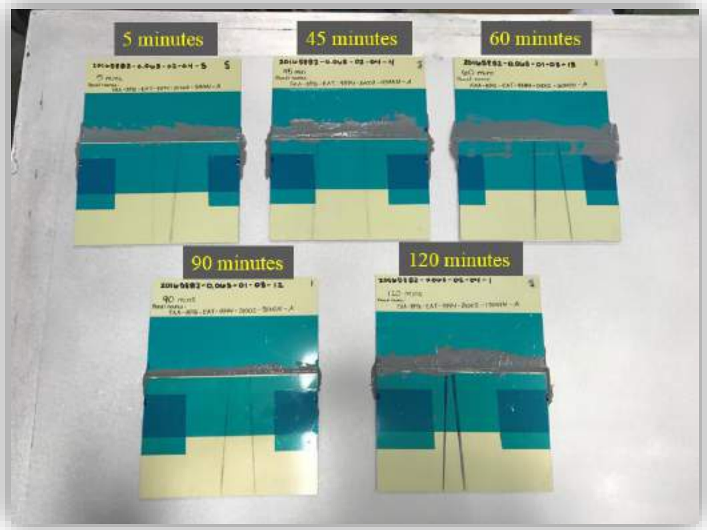
- EA9394 adhesive system showed a ~±15% change in the mechanical properties of single lap shear and fracture toughness at the extreme mix ratios between -40% & +40%
- Repeatability of the experiment was validated with a second data set. Data correlates will with the original testing.
- No change in the 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.



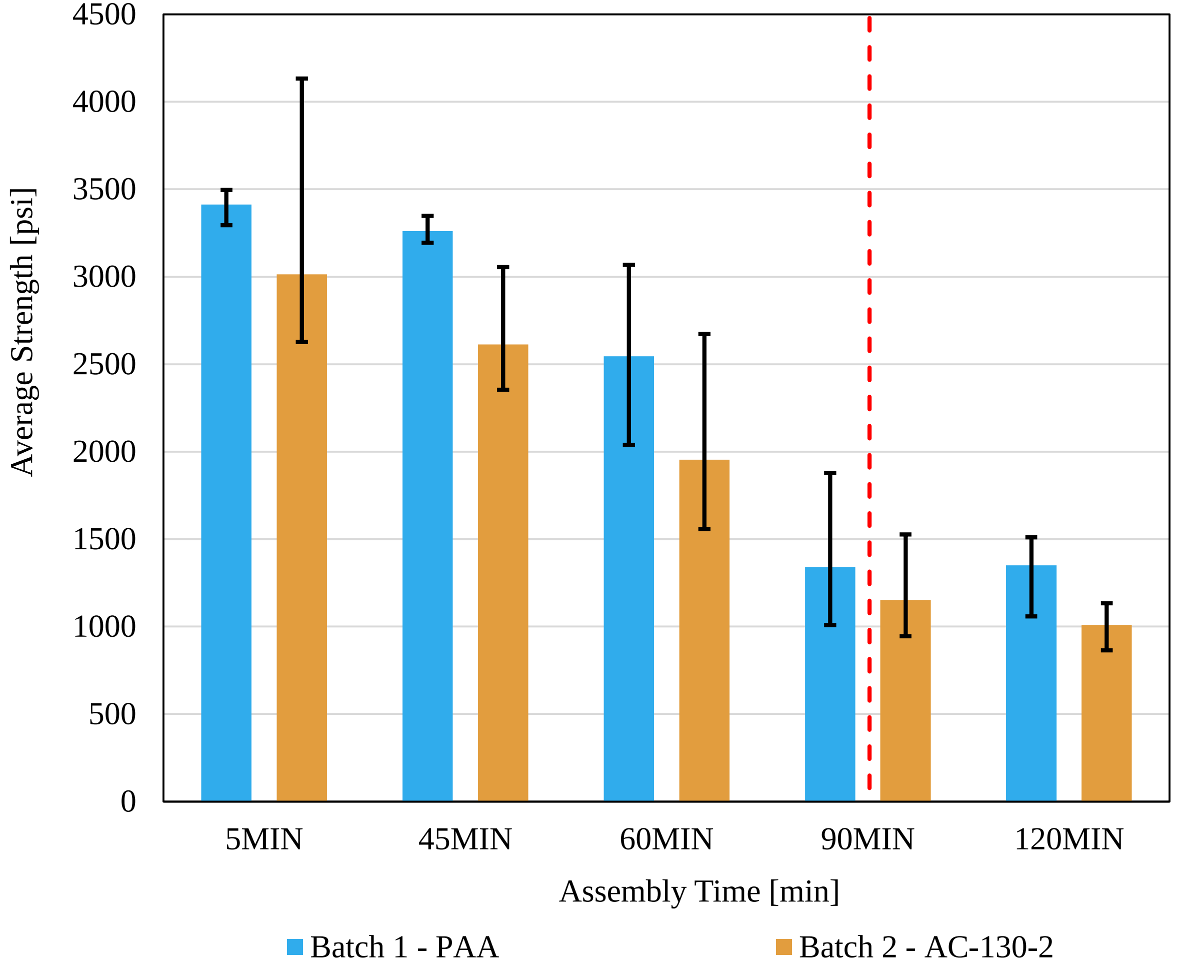
Evaluation of assembly time in paste adhesives

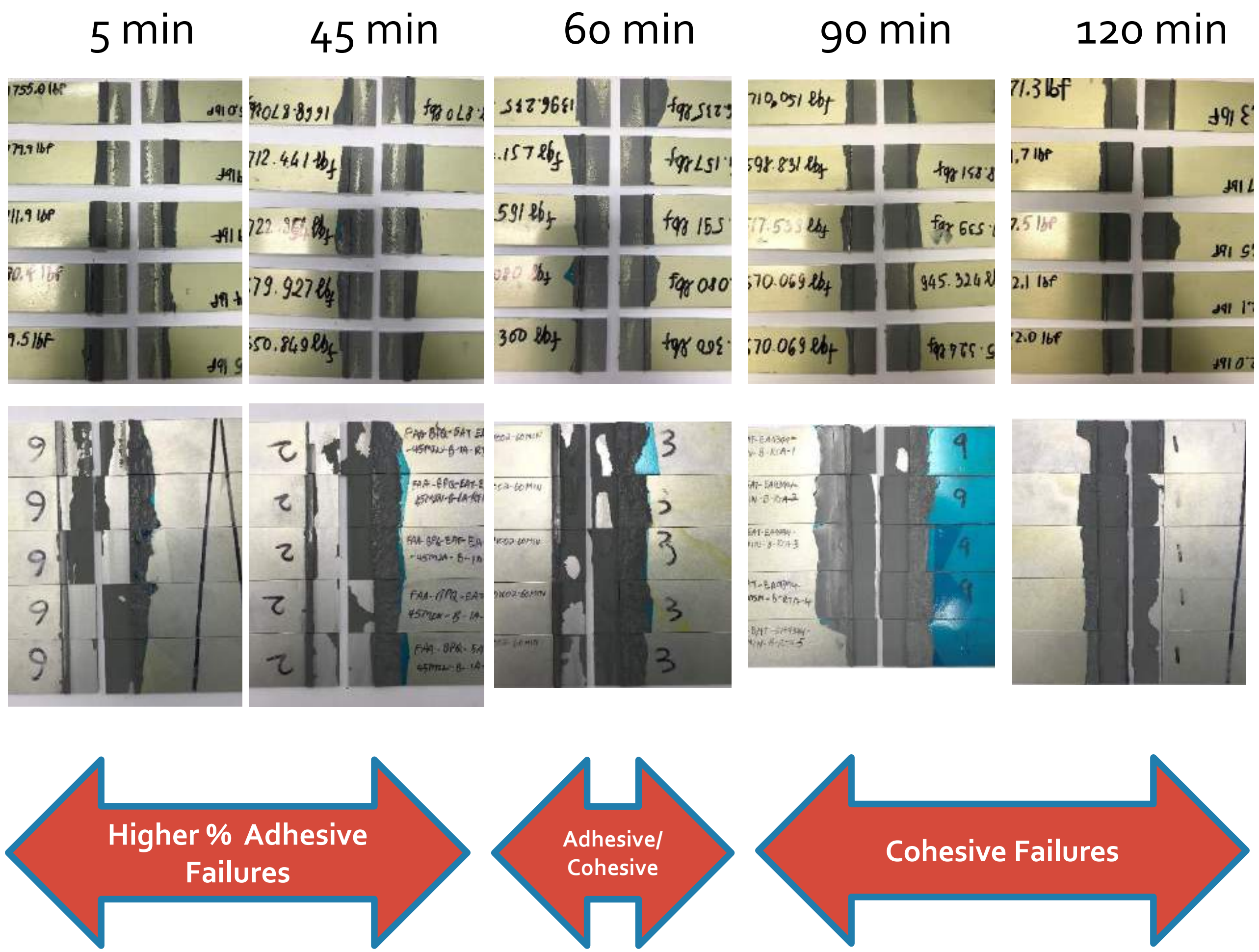
- Manufacturer provided pot life is to be used as a material specification. In bonding applications, assembly time is defined as the time it takes to mix, apply adhesive and mate the two parts together. Depending on the bond area and the complexity (contour) of the structure, this could be a critical 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



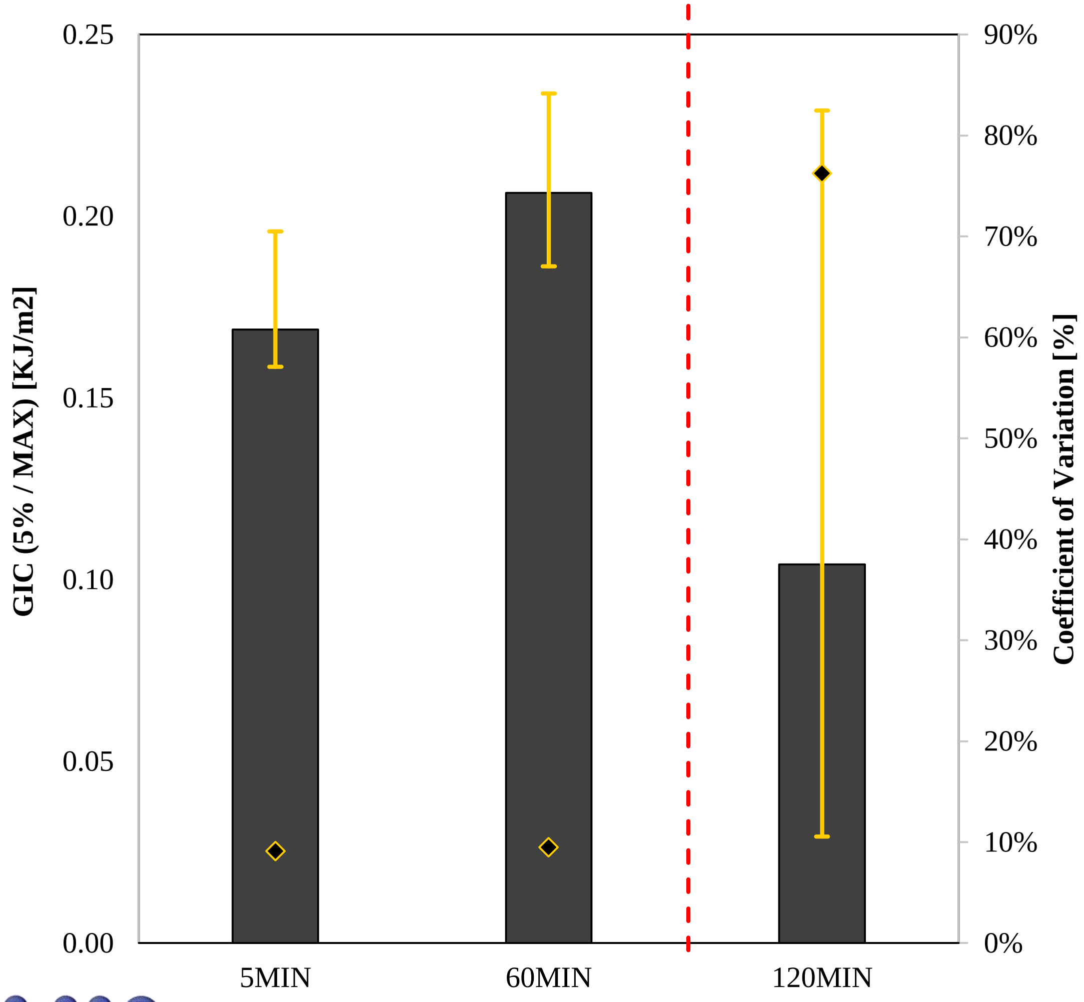


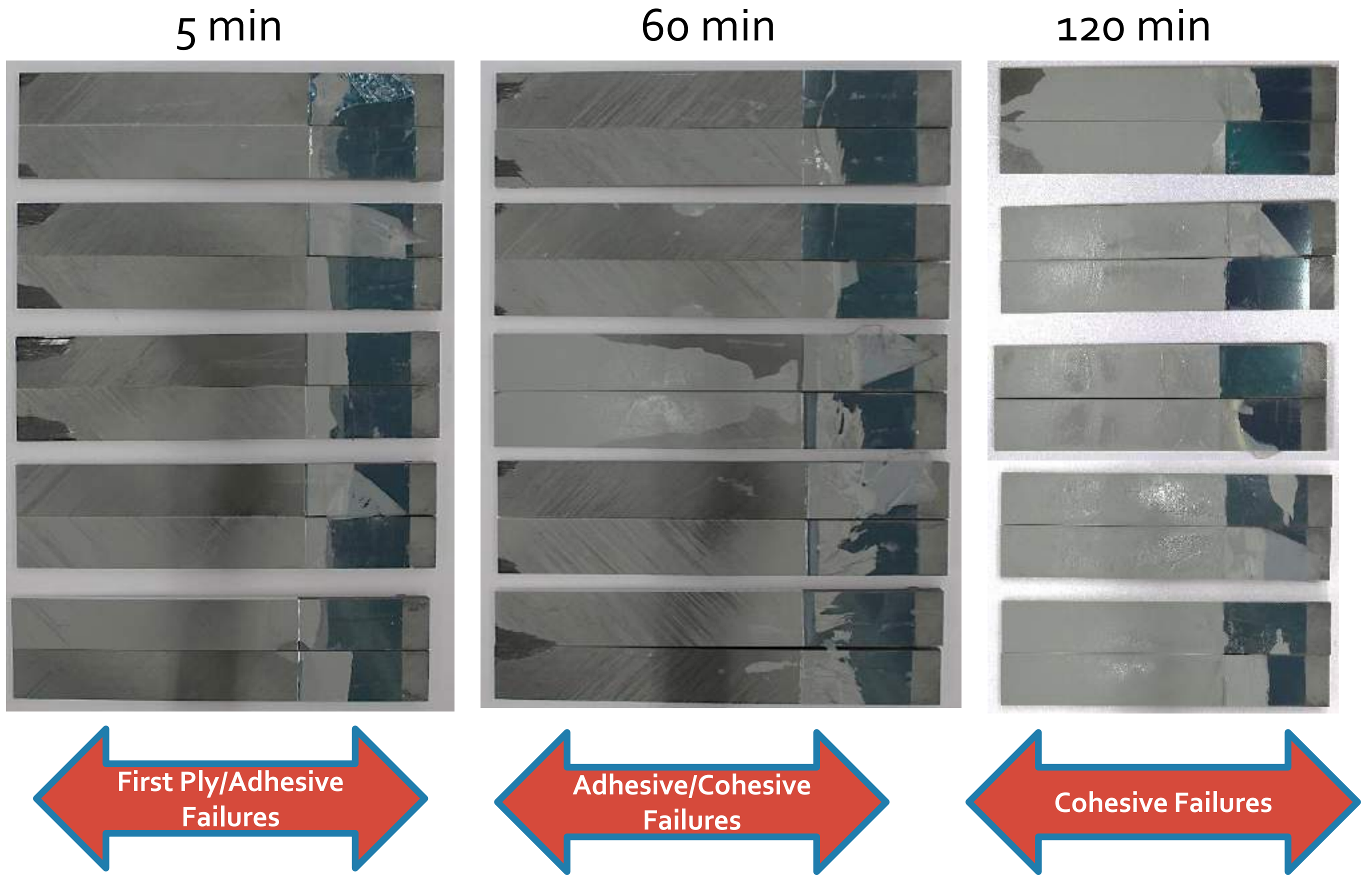
Evaluation of assembly time in paste adhesives
Test Results – Single Lap Shear – ASTM D1002



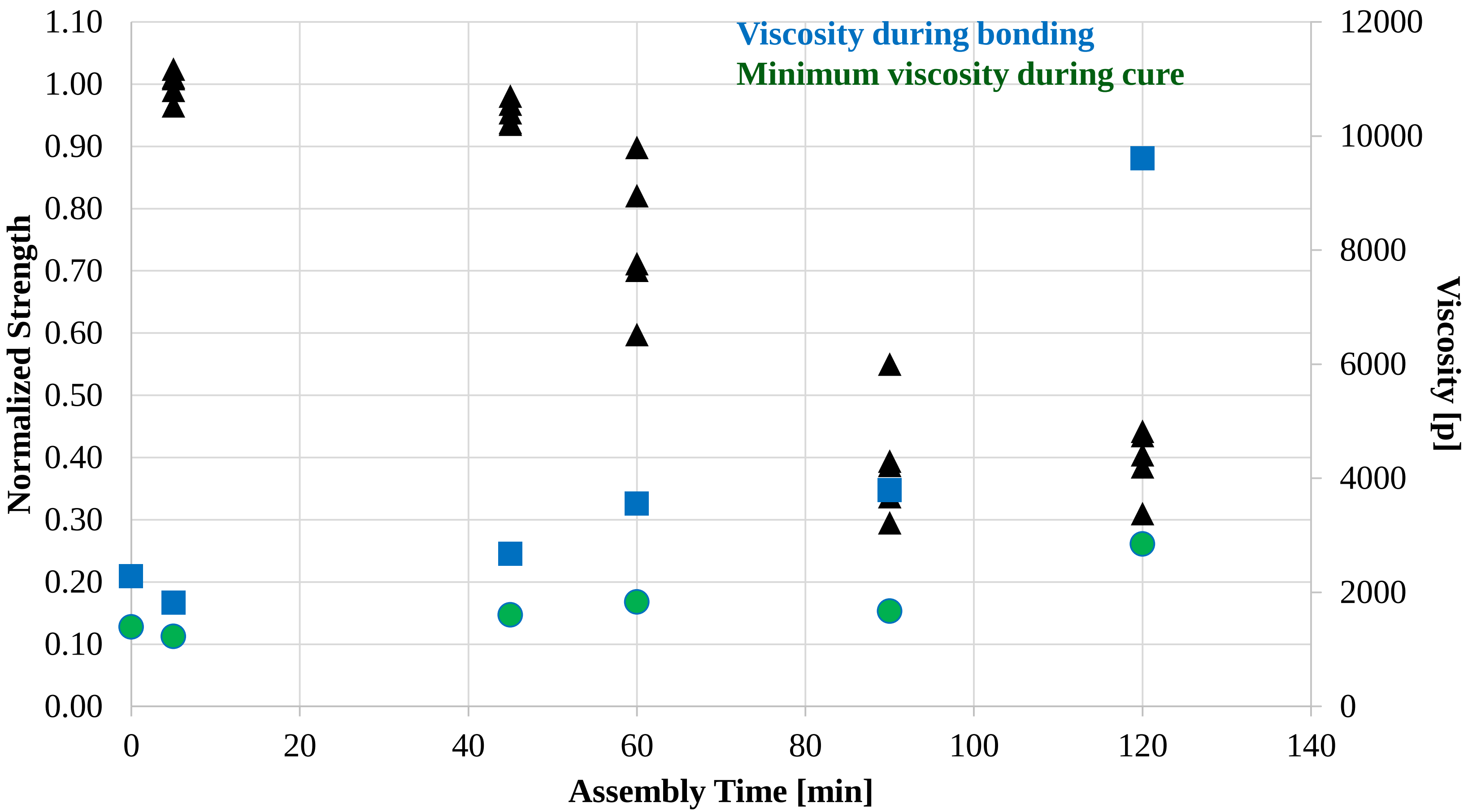


Evaluation of assembly time in paste adhesives
Test Results – Mode I – ASTM D5528



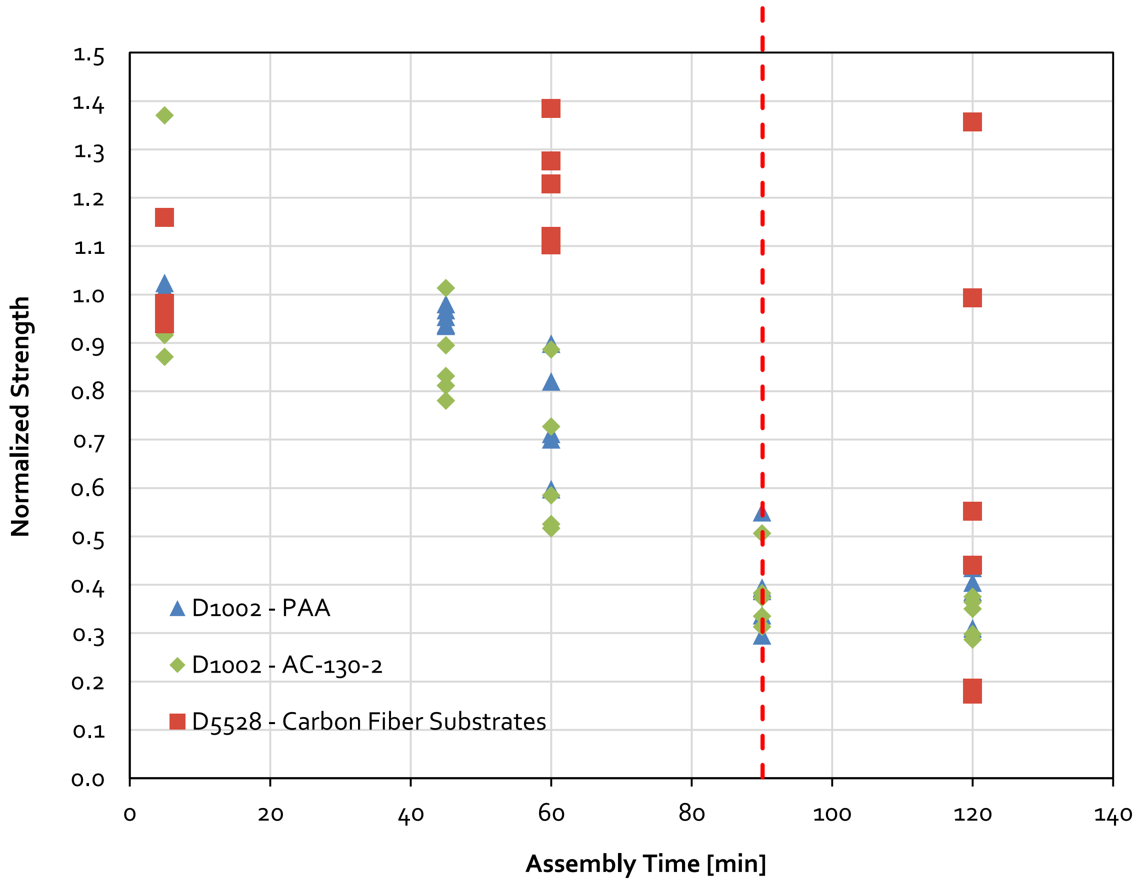


Evaluation of assembly time in paste adhesives
Test Results – Viscosity Response



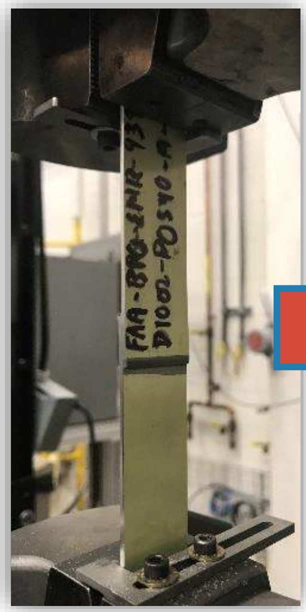
Evaluation of assembly time in paste adhesives
Test results – Summary

- 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.

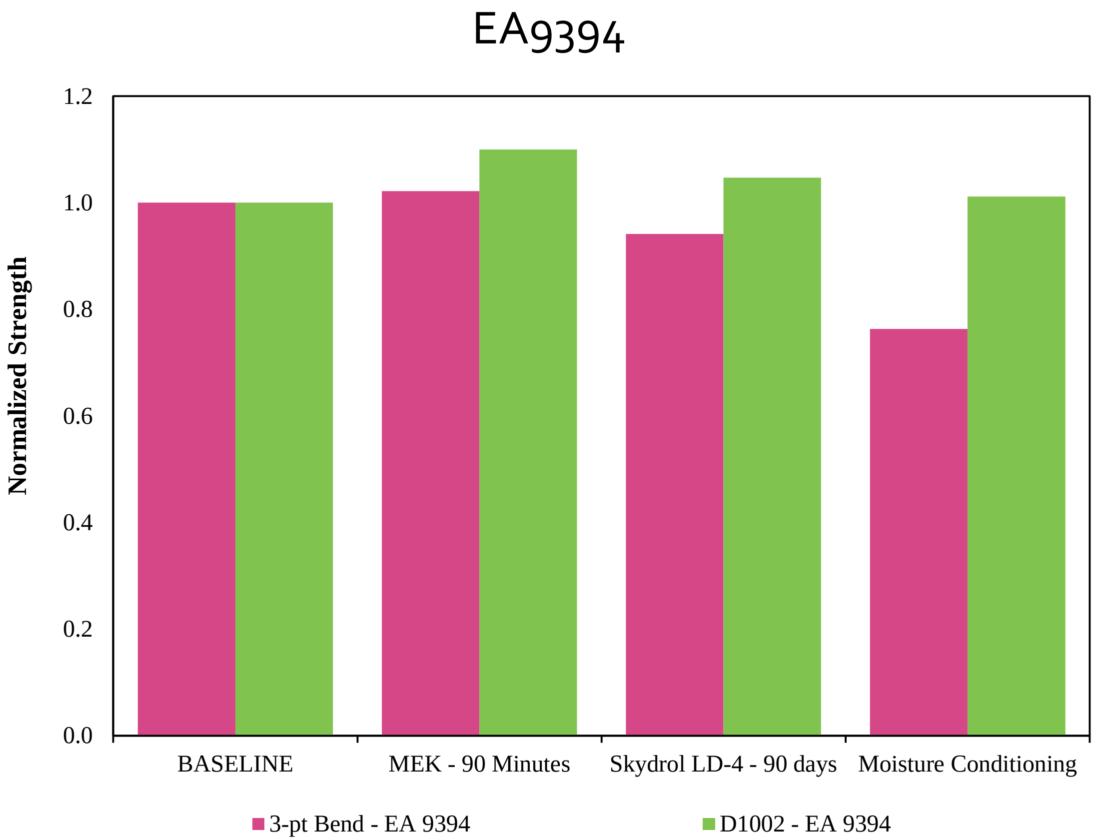


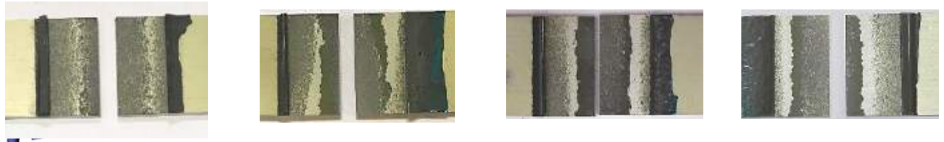
Fluid sensitivity of adhesive

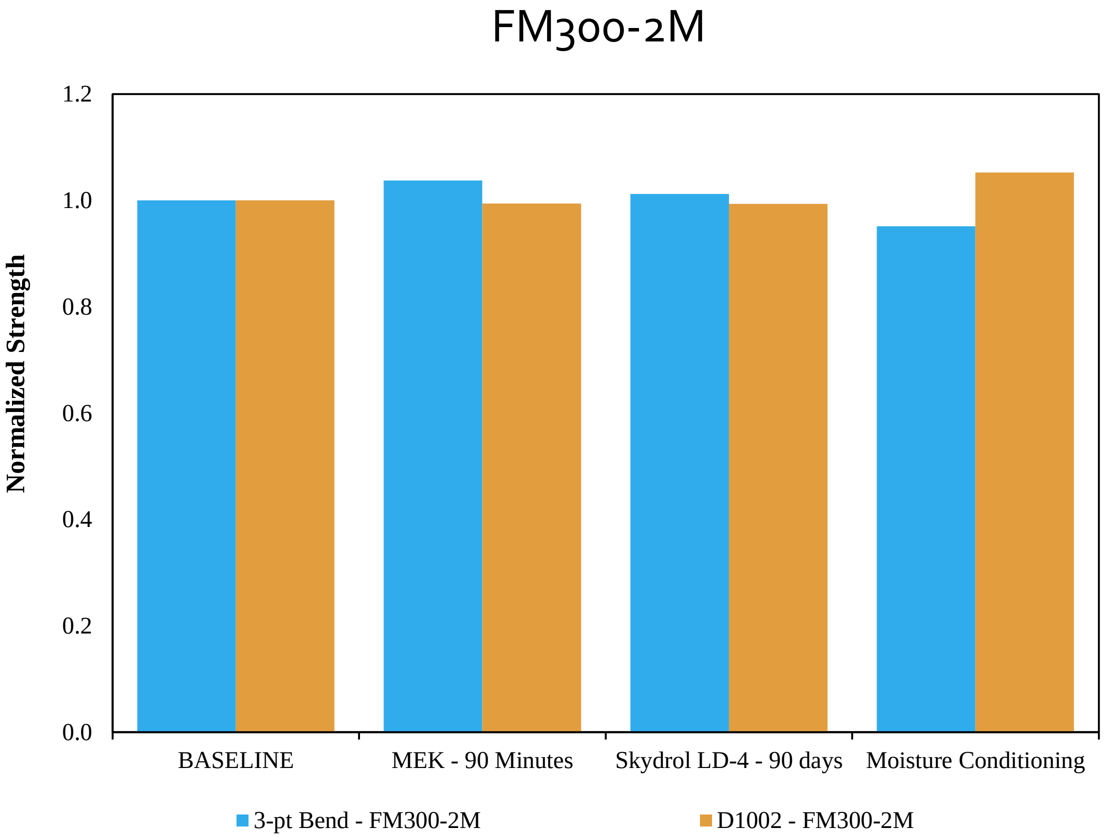
- Current method used to evaluate the fluid sensitivity of adhesives is the D1002 single lap shear specimen configurations.
- D1002 Lap Shear specimen configurations requires treated substrate materials which involves numerous steps form preparation to storage.
- The adhesive area exposed to fluids is 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

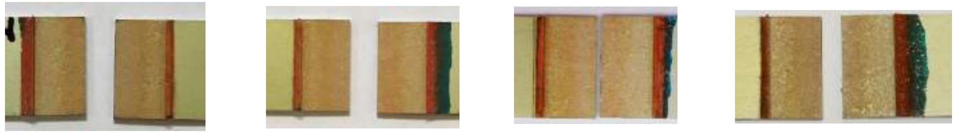


Fluid sensitivity of adhesive Test Results





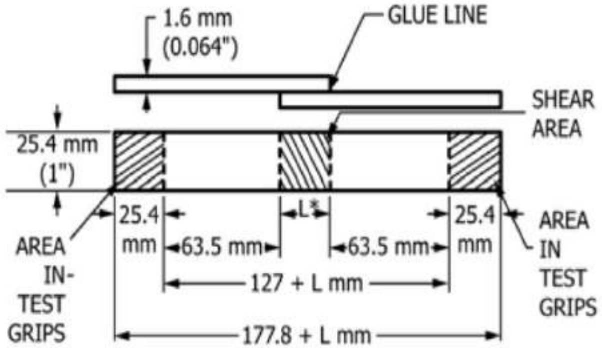




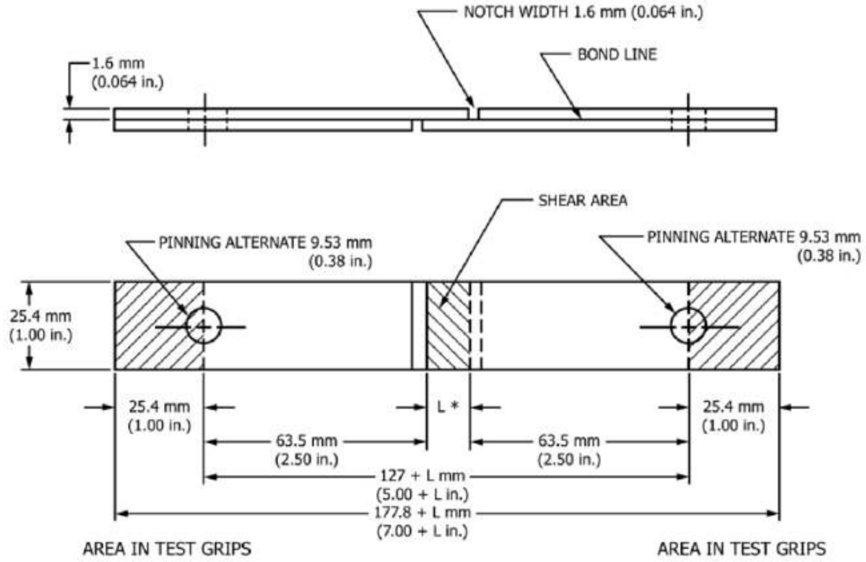
Adhesive Screening Test Methods

- 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 – 0.062-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

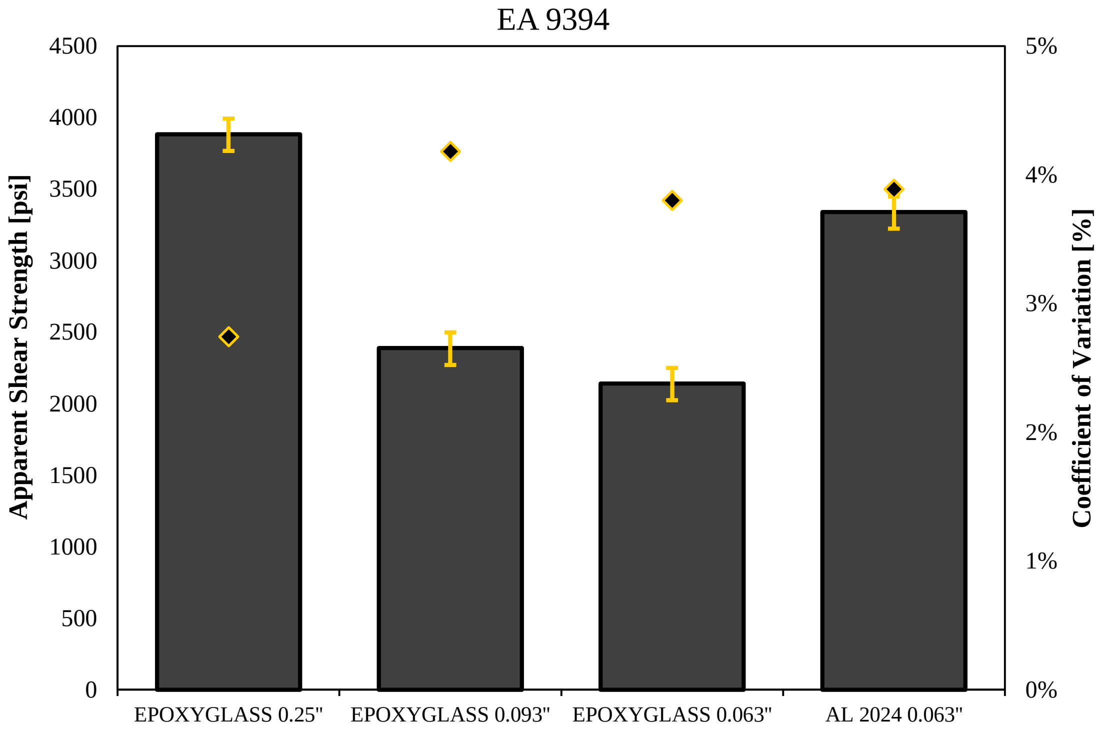
ASTM D1002

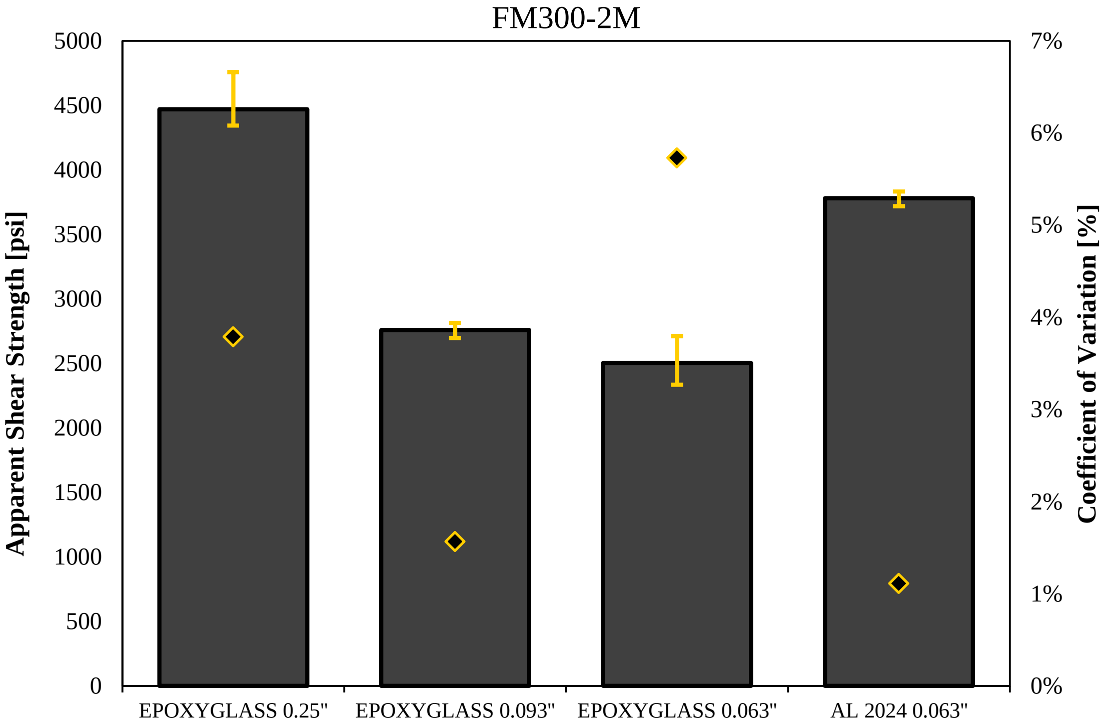


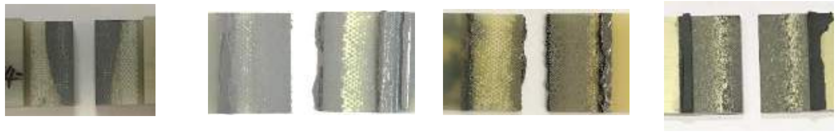
ASTM D3165



Adhesive Screening Test Methods
Test Results







Adhesive Failures | Adhesive/Cohesive Failures

Adhesive Failures | Adhesive/Cohesive Failures

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

Acknowledgement

- Advisory Committee and Program Guidance (FAA)
  - Cindy Ashforth
  - Curt Davies
  - Larry Ilcewicz
  - Ahmet Oztekin
- Program Support
  - Shannon Jones (Textron Aviation)
  - Tony Hinman (Solvay)
  - John Marsicano (Henkel)
- Document Review
  - Rachael Andrulonis (NIAR)
  - Bryce Floryancic
  - Shannon Jones (Textron Aviation)
  - Wei Kwan
  - Royal Lovingfoss (NIAR)
  - Molly Stone
  - Mike Stuart (Solvay)