Joint Centers of Excellence for Advanced Materials

Bond Process Qualification Protocols for Aircraft Design and Certification

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JAMS Technical Review
April 2023
Seattle, WA

Wichita State University

Bond Process Qualification Protocols for Aircraft Design and Certification

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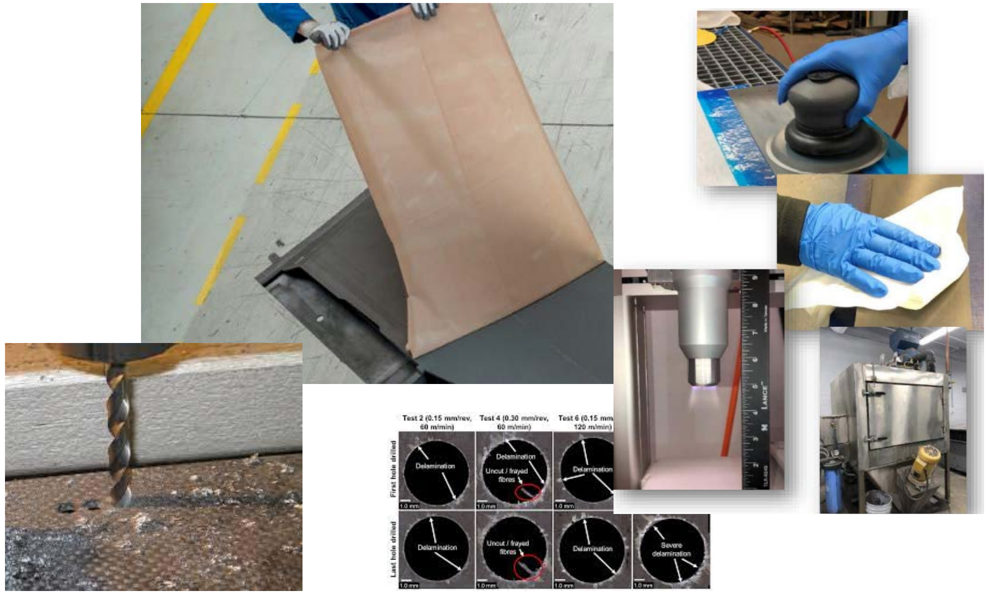
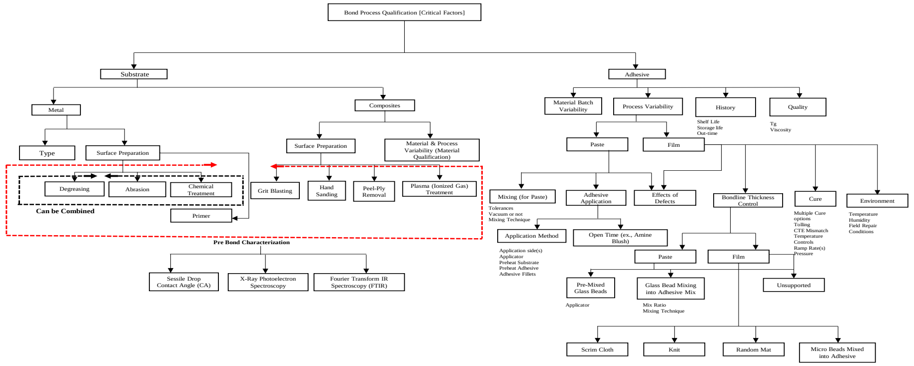
Background & Motivation

- Use of **Bonded Joints** in primary and secondary aircraft structures are preferred over mechanical joins by aircraft manufacturing community due to :
  - Weight reduction compared to fastened structures.
  - Less labor intensive process compared to hole drilling, fastener installation and inspection procedures.
  - Minimum local stress concentrations.
- Despite the many advantages of using Bonded Joints, there are few **challenges** in using bonded joints in aircraft structures
  - Qualification challenges of the **bond process**.
  - Bonding process **sensitivity**.
  - High complexity of **multiple parameters** in the bond system.
  - High variability seen in **technician** involved processes.
  - Lack of effective means to assess the **quality of the bond process** (surface preparation)



The primary goal of this research program is to develop guidance material for bond process qualification protocols and support development of certification road map for bonded structures.



Road Map & Bonded Joint Certification Approach

- 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

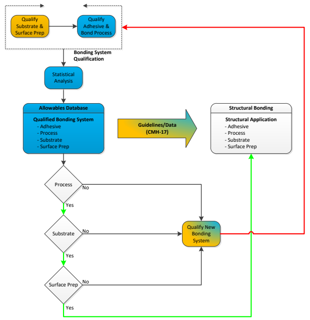
Design and Preliminary Bond System Assessment

Design and Preliminary Bond System Assessment

Bond Process Qualification and Protocol Generation

Structural Certification of Bonded Structure & Maintenance

Objective
Manufacture a Bonded 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

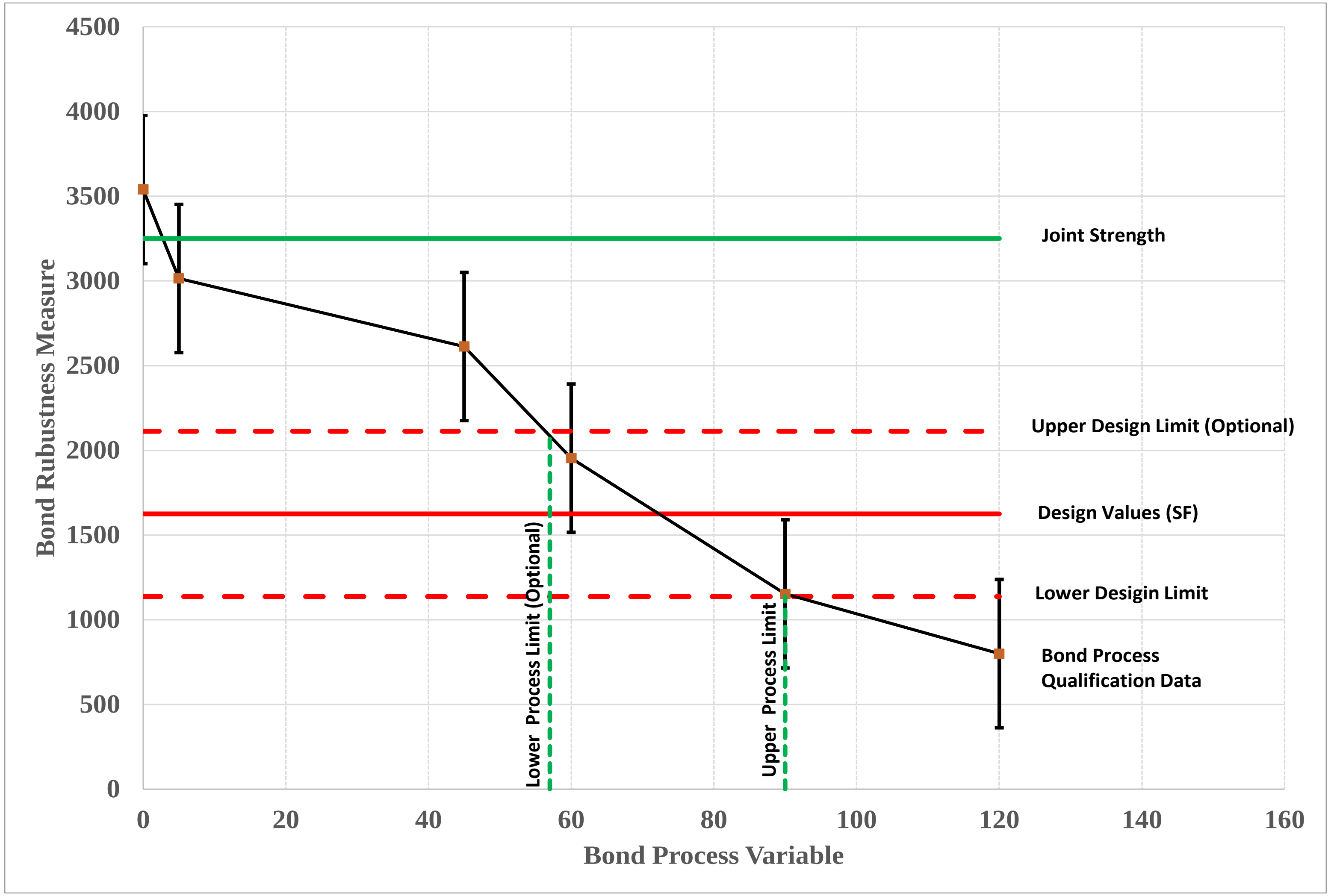
Bond Process Qualification Plan and Protocol Development
1. Finalized bond system
2. Based on the parameters, compose a test matrix to ensure quality assurance of surface preparation and processing parameters
3. Generation of quality assurance methodologies

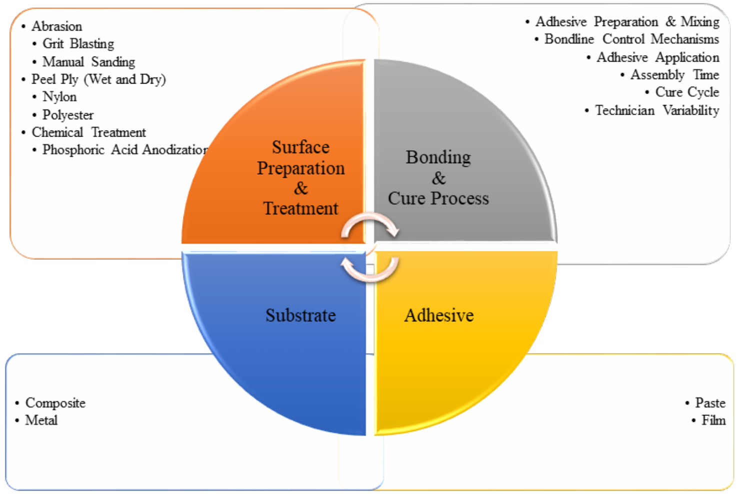
Preliminary Selection and Screening of Substrate and Adhesive Materials
1. Material allowable – (Material databases)
2. Adhesive & Substrate compatibility assessment & wettability assessment Selection of surface preparation methodology
3. Adhesive processing parameters of a representative design
4. Conduct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

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

Bonding Process Limit Determination





Selection of Parameters for Bond System

Step 1
Selection of Adhesive & Substrate

- Substrate
- Adhesive

Step 2
Surface Preparation Effectiveness Assessment

- Abrasion
- Peel Ply
- Solvent Cleaning
- Chemical
- Plasma Treatment

Step 3
Surface Preparation to Bonding Time Frame Evaluation

- Exposed Environment
- Time from Surface Preparation to Bonding

Step 4
Adhesive Preparation and Application (Paste)

- Mix Ratio (2 part)
- Assembly Time
- Mixing Method (2 part)
- Application Method
- Bondline Control Mechanism

Step 5
Pressure Application & Cure Cycle

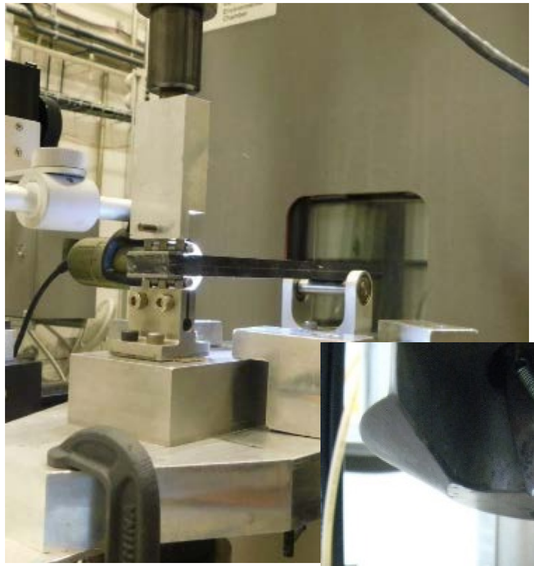
- Applied Pressure During Cure Cycle
- Cure Cycle

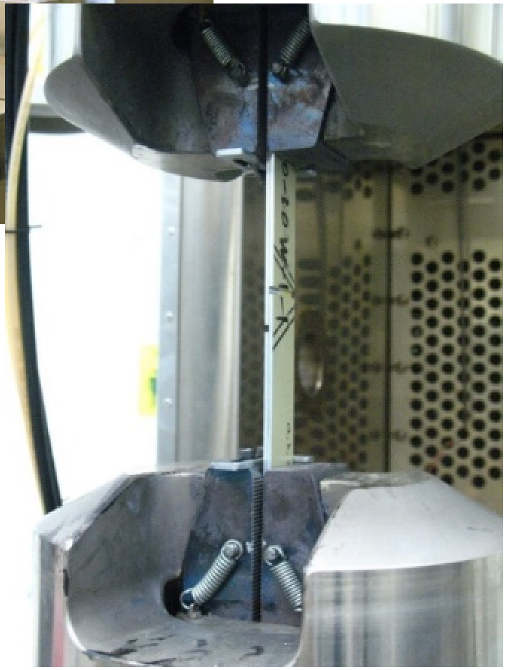
Finalized Bond System Parameters

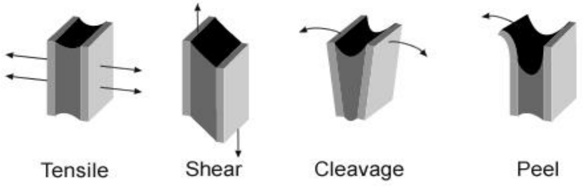
- Substrate
- Adhesive
- Surface Preparation Technique
- Exposure Environment & Time Frame
- Adhesive Preparation and Application
- Cure Cycle and Pressure Application

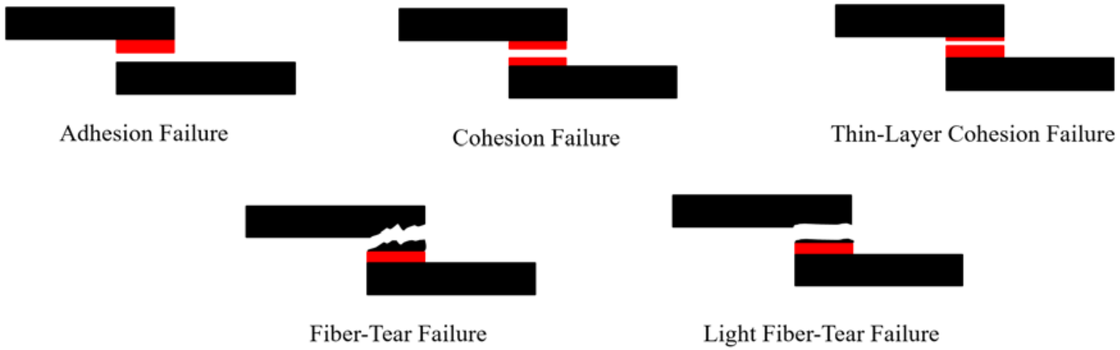
Test Methods for Bond Process Evaluations

Joint Property Under Investigation	Bond System Component Under Investigation			
	Surface Preparation		Adhesive Preparation and Cure Process	
	Surface Preparation Method	Test Method	Surface Preparation Method	Test Method
Peel/Fracture Toughness/Mode I	Variable	ASTM D5528	Fixed Based on Step 2	ASTM D5528
Shear		ASTM D3165		ASTM D3165







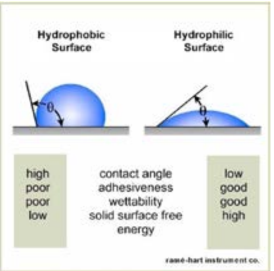


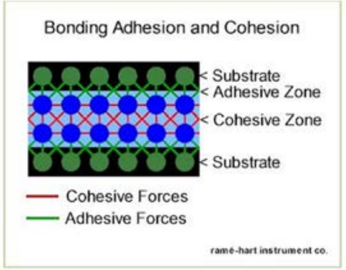
Surface Preparation Evaluation – Step 2

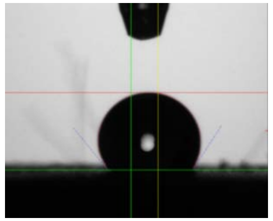
Surface Preparation Effectiveness Measurement

Surface Preparation Quality Assessment

- Surface Free Energy Measurement
- Water Contract Angle Measurement

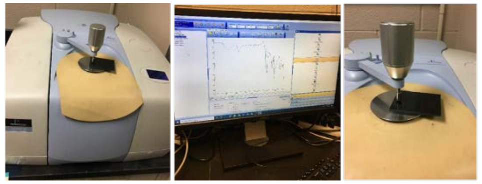


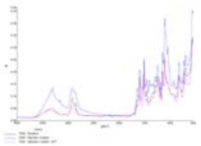


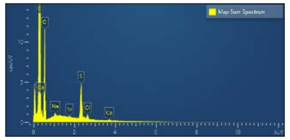


Surface Contamination Assessment

- Fourier Transform Infrared Spectroscopy (FTIR)
- Energy Dispersive X-ray spectroscopy (EDS)

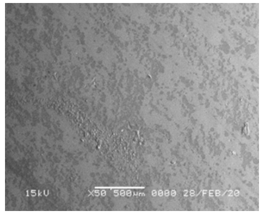


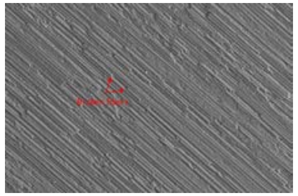




Substrate Integrity Assessment

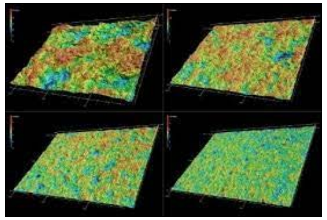
- Scanning Electron Microscopy (SEM)
- Optical Microscopy

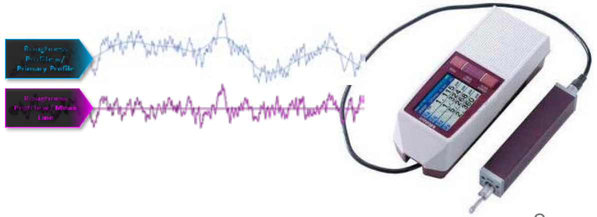




Surface Morphology /Roughness Assessment

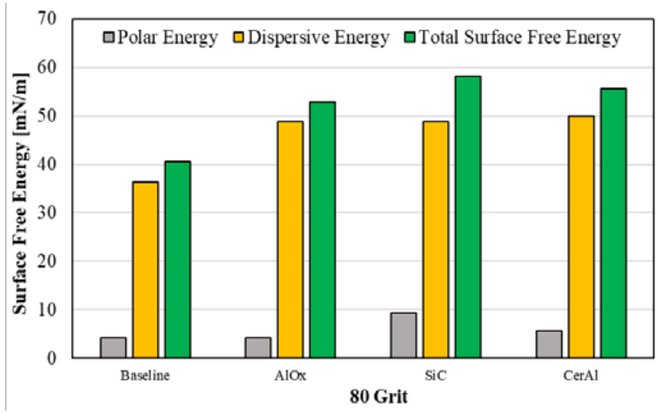
- Optical Profilometry
- Surface Roughness Measurements



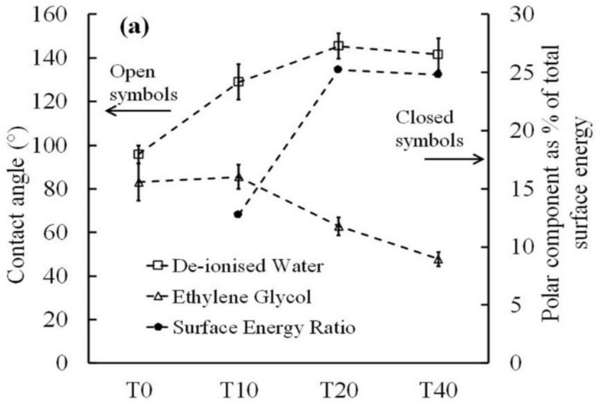


Surface Preparation Evaluation Test Matrix
Manual and Machine Assisted Abrasion

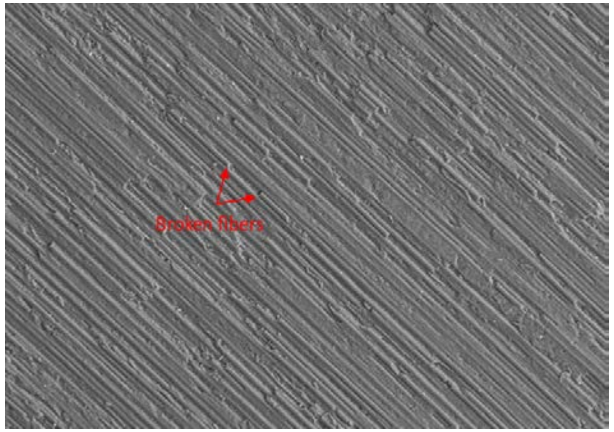
- Potential Parameters
  - Grit Size, Type
  - Duration of Sanding
  - Applied Pressure (Technician Variability)
  - Sanding Direction
  - Sanding Repetitions
  - Sander Type (Disk, Orbital)



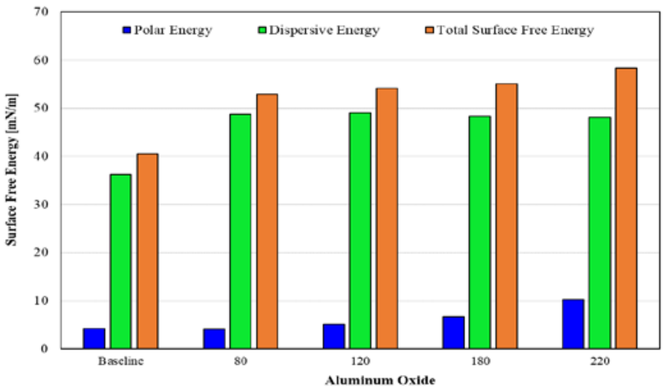
Effects of grit type on SFE (machine assisted)
Baseline is untreated



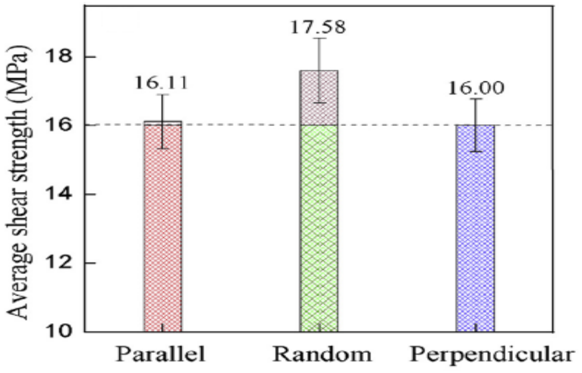
Effects of Abrasion Duration [2]



***SEM** image of showing **broken fibers** after machine assisted abrasion (x200)*



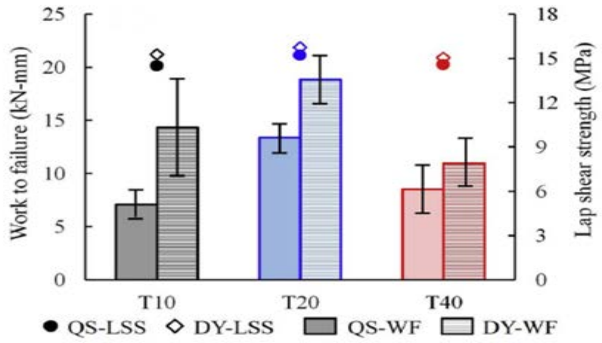
Effects of grit size on SFE (machine assisted)
Baseline is untreated



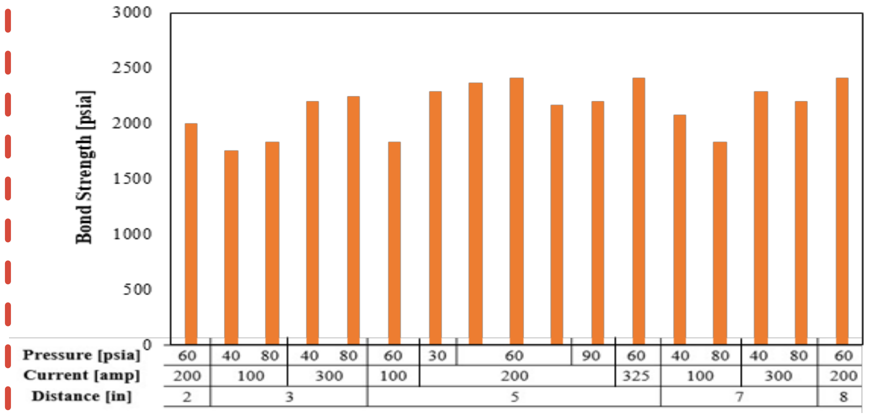
Effects of Sanding Direction [1]
Surface Preparation: 220 Grit Al Ox sandpaper

Surface Preparation Evaluation Test Matrix
Grit Blasting

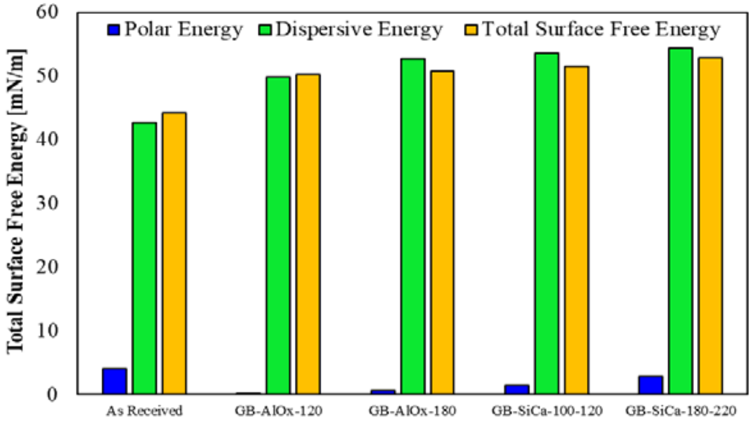
- Potential Parameters
  - Grit Size, Type
  - Application Angle & Nozzle Geometry
  - Pressure
  - Standard off Distance
  - Sanding Repetitions
  - Blasting Configuration (Dry/Wet)



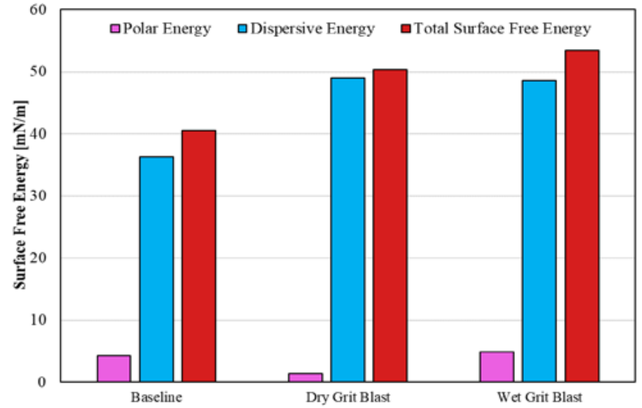
Effects of Grit Blasting Duration [2]
Surface Preparation: Grit Blast, 60 Grit Al Ox for 10, 20, and 40 seconds



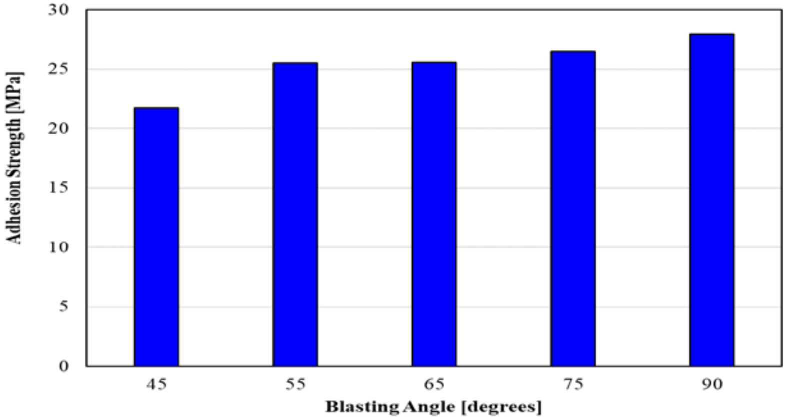
Effects of Grit Blasting Pressure and Distance [4]



Effects of grit type and grit size on SFE (grit blast)
Baseline is untreated
AlOx -Aluminum Oxide
SiCa - Silicon Carbide

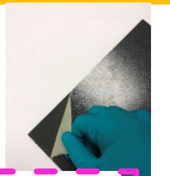


Effects of grit blasting configuration on SFE (grit blast)
Baseline is untreated
Surface preparation: Dry and wet grit blasting with aluminum oxide grit

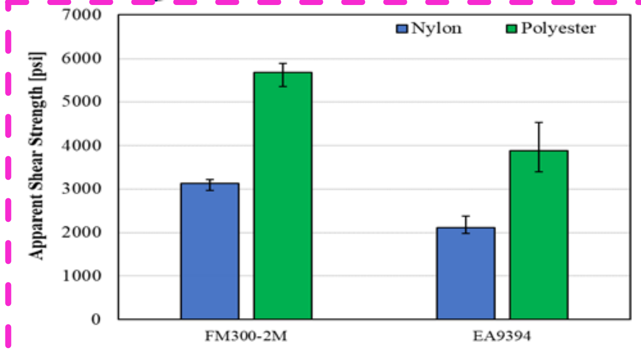


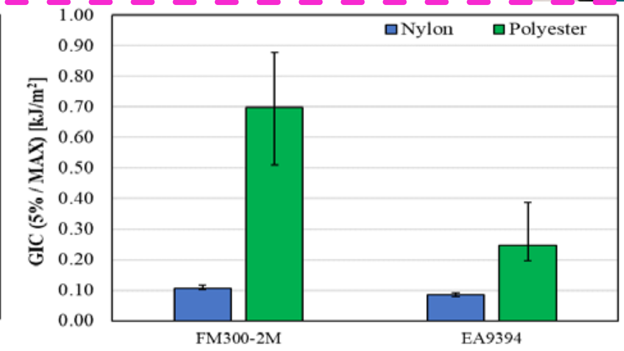
Effects of Grit Blasting Angle [1]
Substrate Material: Titanium
Surface Preparation: Grit Blast with 60 mesh size aluminum oxide grit
Test Method: Thermal Spray Adhesion Testing (ASTM C633)

Surface Preparation Evaluation Test Matrix
Peel Ply



- Potential Parameters
  - Material Type, Thickness, & Resin Content
  - Placement Location
  - Peel Ply Removal Direction & Angle
  - Removal Time Frame
  - Repetitive Cure Processes
- Recommended Additional Evaluations
  - Fiber Volume Fraction & Resin Content
  - Degree of Cure

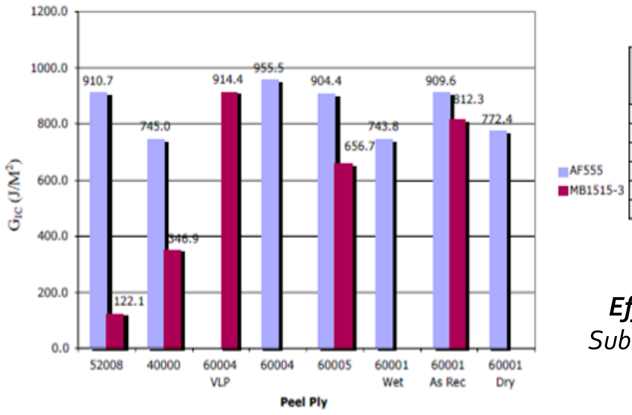




Effects of Peel Ply Material on single lap shear strength (ASTM D3165) and Mode I fracture toughness (ASTM D5528)

Substrate Material: Toray T800H/3900-2

Material	Code	Style	Thickness [in]	Color	Warp [ends/in]	Fill [picks/in]
Nylon	51789	52006	0.0045 – 0.0055	White	160	103
Polyester	60004	56111	0.0045 – 0.0055	White	120	59

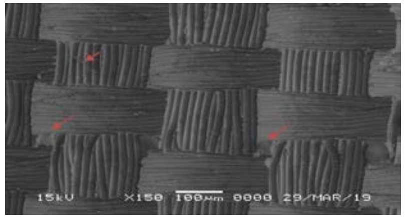


Filament Material	Precision Fabrics Code	Warp (ends/mm) (ends/in.)	Fill (picks/mm) (ends/in.)	Peel Ply Thickness (mm)
Polyester	60001	2.75 (70)	1.97 (50)	0.13-0.15
Polyester	60004 VLP	4.72 (120)	2.32 (59)	0.13-0.15
Polyester	60004	4.72 (120)	2.32 (59)	0.11- 0.14
Polyester	60005	3.54 (90)	2.28 (58)	0.15-0.18
Nylon 6,6	52008	3.98 (101)	3.23 (82)	0.10-0.13
Nylon 6,6	40000	2.99 (76)	2.01 (51)	0.19-0.22

Effects of Peel Ply Pattern/Texture/Thickness [6]

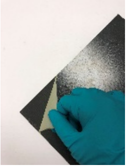
Substrate Material: Carbon fiber/epoxy unidirectional material

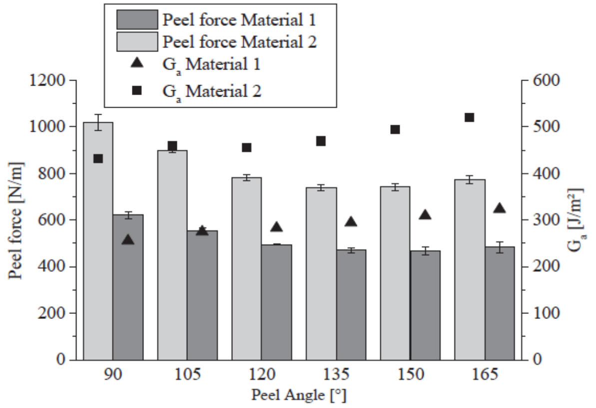
Test Method: ASTM D5528



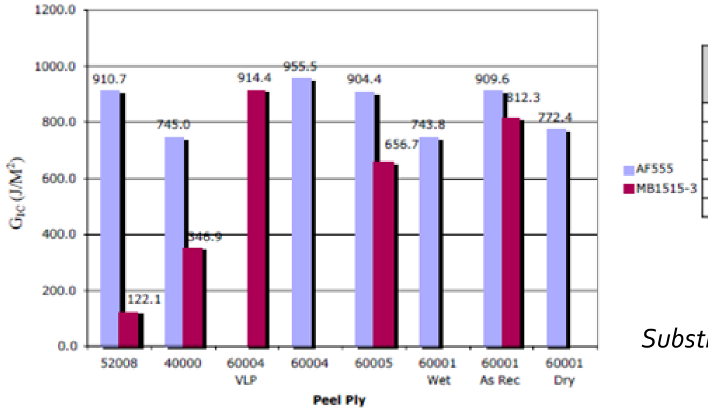
SEM image showing peel ply remnants after removal (x150)

Surface Preparation Evaluation Test Matrix
Peel Ply (Cont.)





Effects of Peel Force needed to remove peel ply [5]

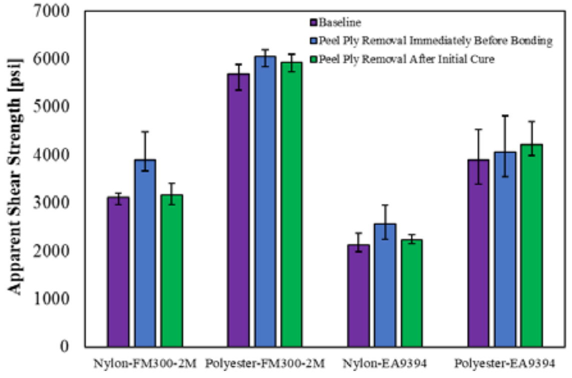


Filament Material	Precision Fabrics Code	Warp (ends/mm) (ends/in.)	Fill (picks/mm) (ends/in.)	Peel Ply Thickness (mm)
Polyester	60001	2.75 (70)	1.97 (50)	0.13-0.15
Polyester	60004 VLP	4.72 (120)	2.32 (59)	0.13-0.15
Polyester	60004	4.72 (120)	2.32 (59)	0.11- 0.14
Polyester	60005	3.54 (90)	2.28 (58)	0.15-0.18
Nylon 6,6	52008	3.98 (101)	3.23 (82)	0.10-0.13
Nylon 6,6	40000	2.99 (76)	2.01 (51)	0.19-0.22

Effects of Peel Ply Pattern/Texture/Thickness [6]

Substrate Material: Carbon fiber/epoxy unidirectional material

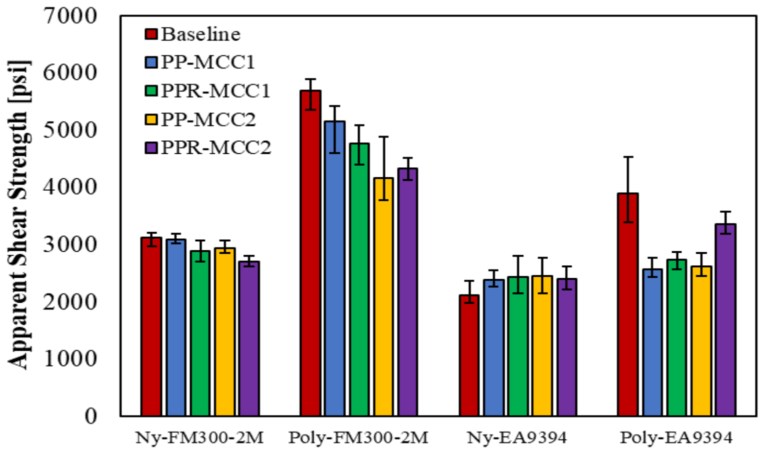
Test Method: ASTM D5528



Effects of Peel Ply Removal Time Frame

Test Method: ASTM D3165

Substrate Material: Toray T800H/3900-2



Effects of Repetitive Cure Processes

Test Method: ASTM D3165

Substrate Material: Toray T800H/3900-2

PP: Peel ply intact during post-cure

PPR: Peel ply removed during post-cure

MCCx: No. of post-cure cycles (1 or 2)

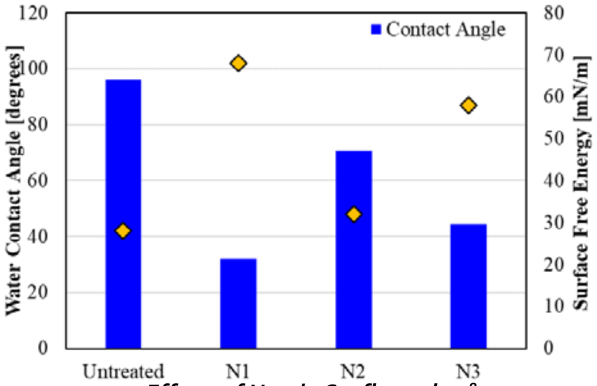
Material	Code	Style	Thickness [in]	Color	Warp [ends/in]	Fill [picks/in]
Nylon	51789	52006	0.0045 – 0.0055	White	160	103
Polyester	60004	56111	0.0045 – 0.0055	White	120	59

Surface Preparation Evaluation Test Matrix

Plasma Treatment

Note:- Surface Morphology or Roughness changes are not seen in plasma treatment

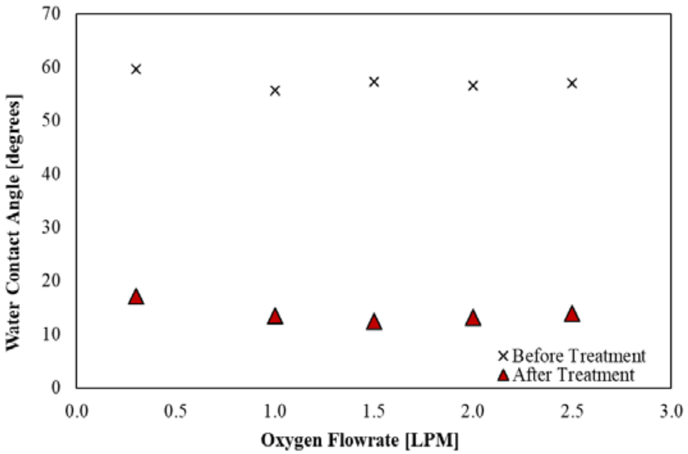
- Potential Parameters (For each plasma recipe)
  - Nozzle Configuration, Diameter/Area
  - Nozzle Height and Speed
  - Treatment Repetition and Overlap
- Additional Parameters for Gas Plasma Treatment
  - Primary Gas
  - Process Gas
  - Gas flow mix ratio



Effects of Nozzle Configuration [8]

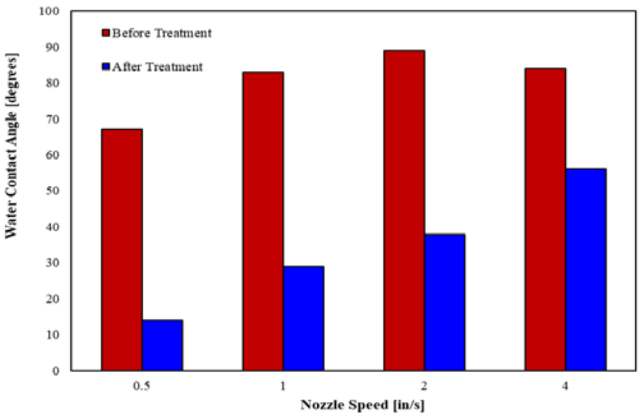
Substrate Material: Polypropylene

Nozzle Code	N1	N2	N3
Nozzle Type	22826	22892	10145
Hole Diameter [mm]	4	4	4
Angle [degrees]	14	32	-
Exposure Width [mm]	22	50	8



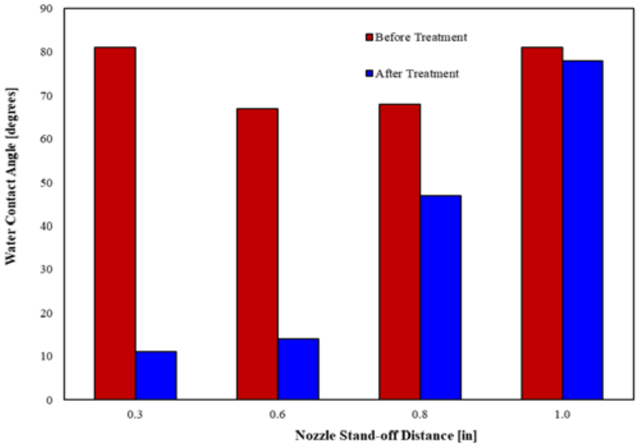
Effects of Gas Flow Rate

Substrate Material: Toray T800H/3900-2



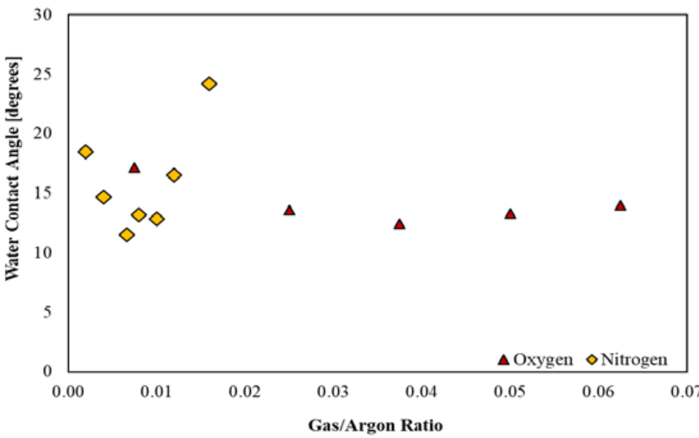
Effects of Nozzle Speed

Substrate Material: Toray T800H/3900-2



Effects of Nozzle Stand-off Distance

Substrate Material: Toray T800H/3900-2



Effects of Gas Mix Ratio

Substrate Material: Toray T800H/3900-2

Surface Preparation Quality Assurance Test Matrix

- Surface preparation technique evaluation data to be analyzed to estimate the upper and lower levels of Surface Free Energies (SFE) and/or Water Contact Angle (WCA) of prepared substrates
- Utilization of Goniometer/Surface Analysts (BTG Labs)/ Surface Analyzer (KRUSS) to measure the SFE and WCA.
- Perform mechanical testing outlined below to evaluate the bonded joint strength and failure modes.

Test Method	Standard	Substrates	Substrates Thickness [in]	Water Contact Angle/SFE				
				Low	Low-Medium	Medium	Medium-High	High
Single Lap Shear Testing	ASTM D3165	Composites Substrate Under Investigation	0.064 (minimum)	5	5	5	5	5
Mode I Fracture Toughness	ASTM D5528		0.12-0.20-inch	5	5	5	5	5


Adhesion Failure

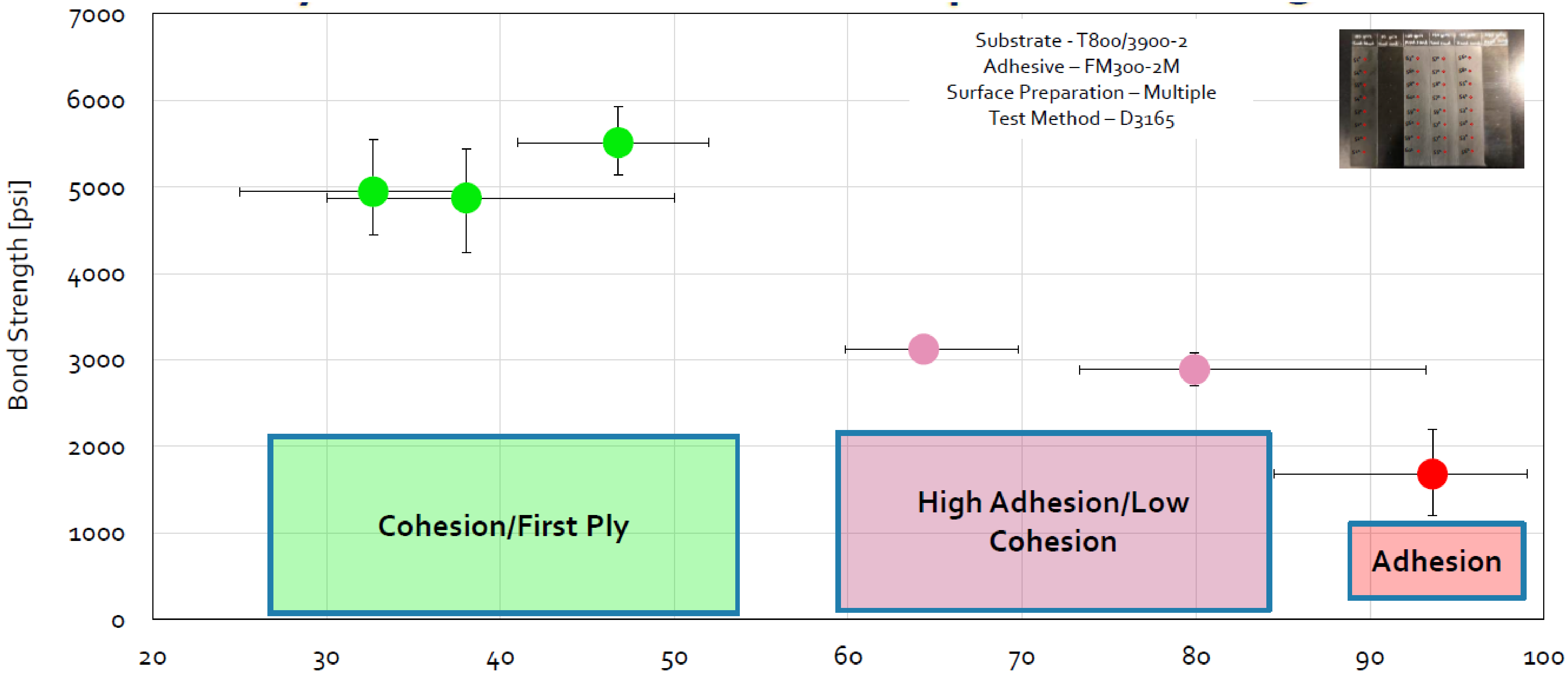

Cohesion Failure


Thin-Layer Cohesion Failure

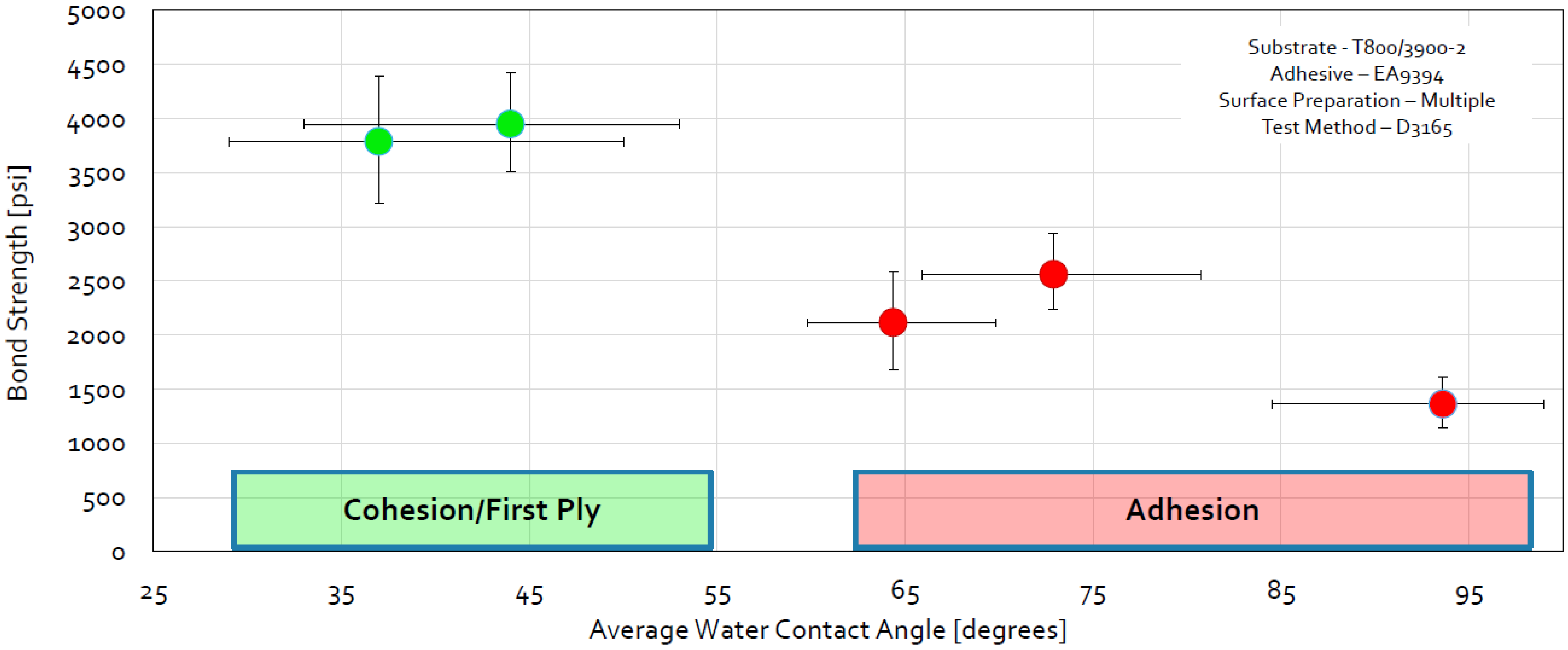

Fiber-Tear Failure

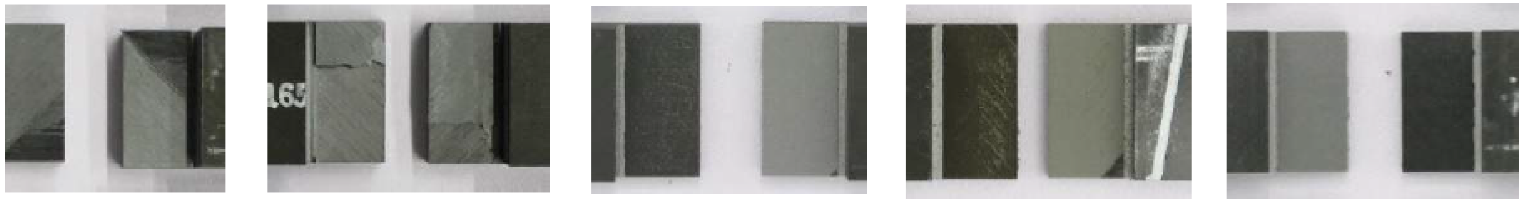

Light Fiber-Tear Failure

Surface Preparation Quality Assurance – FM300-2M



Surface Preparation Quality Assurance – EA9394

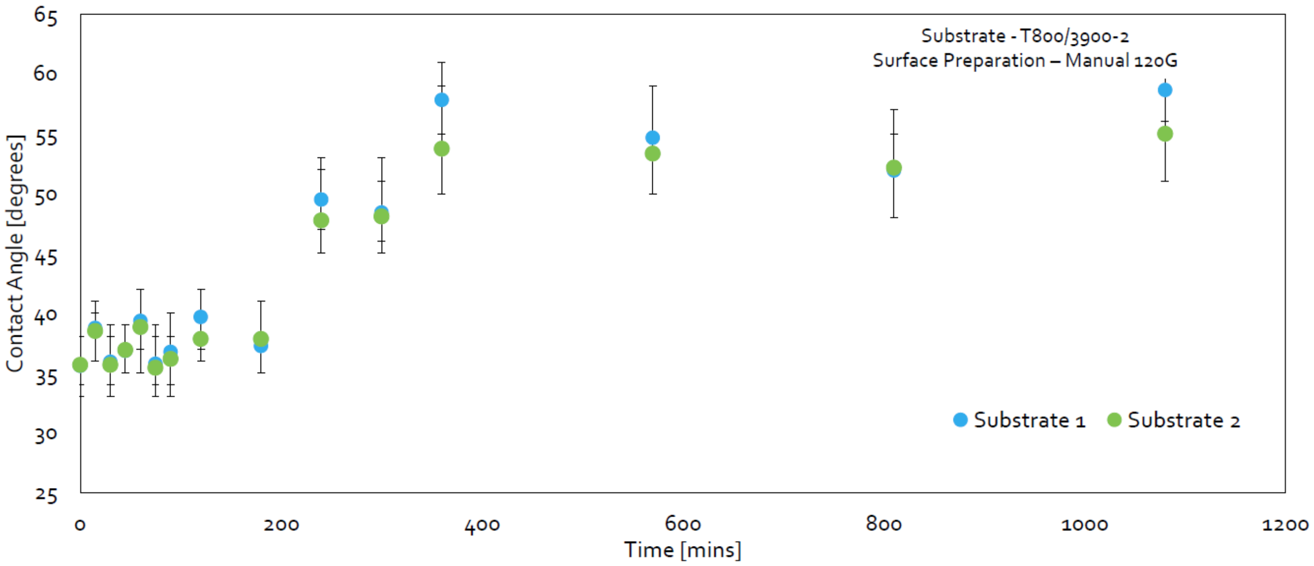


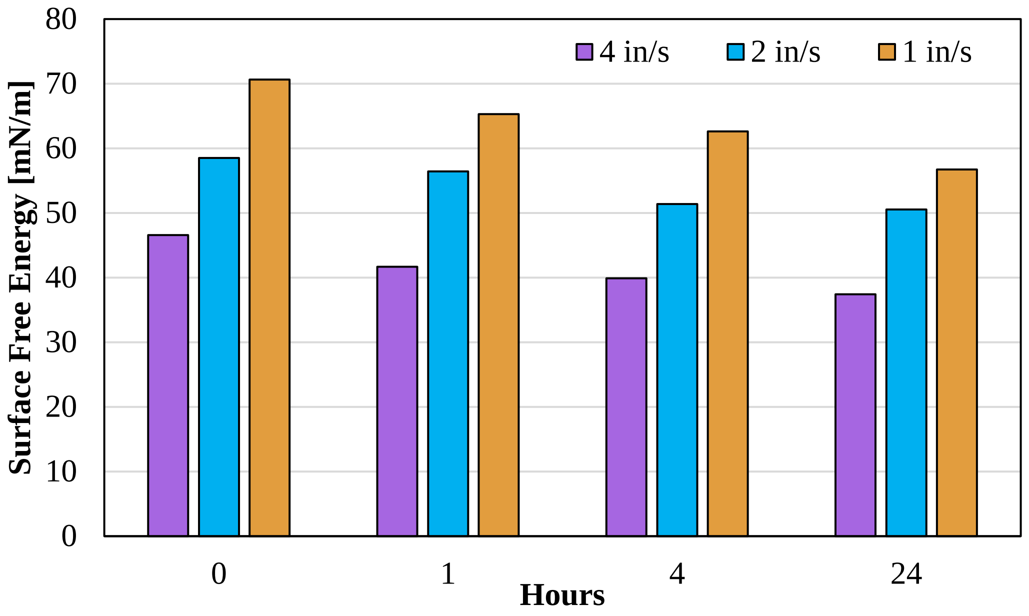


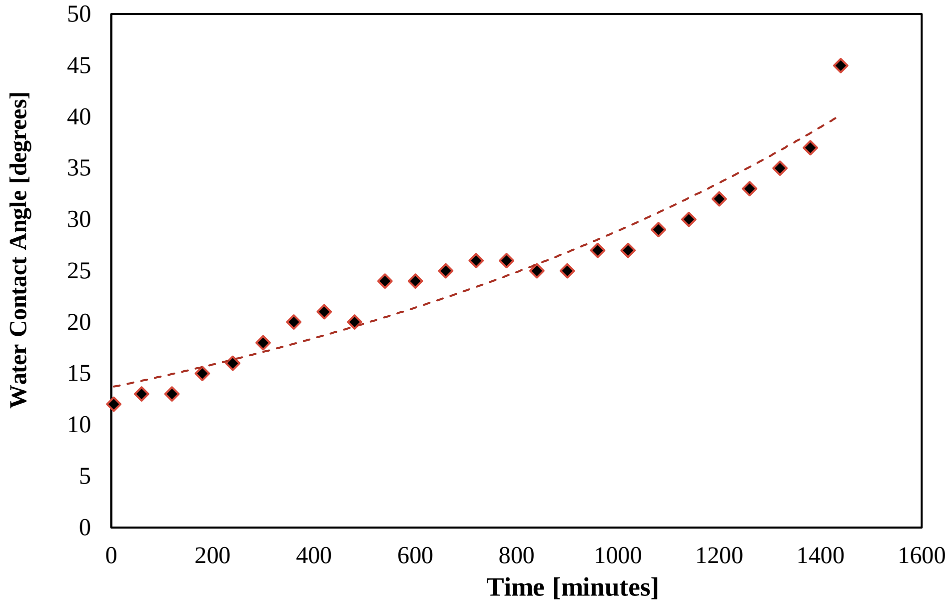
Surface Preparation to Bonding Time Frame – Step 3

Critical Parameters

1. Temperature/Humidity
2. Cleanliness/Particle count
3. Exposure Duration





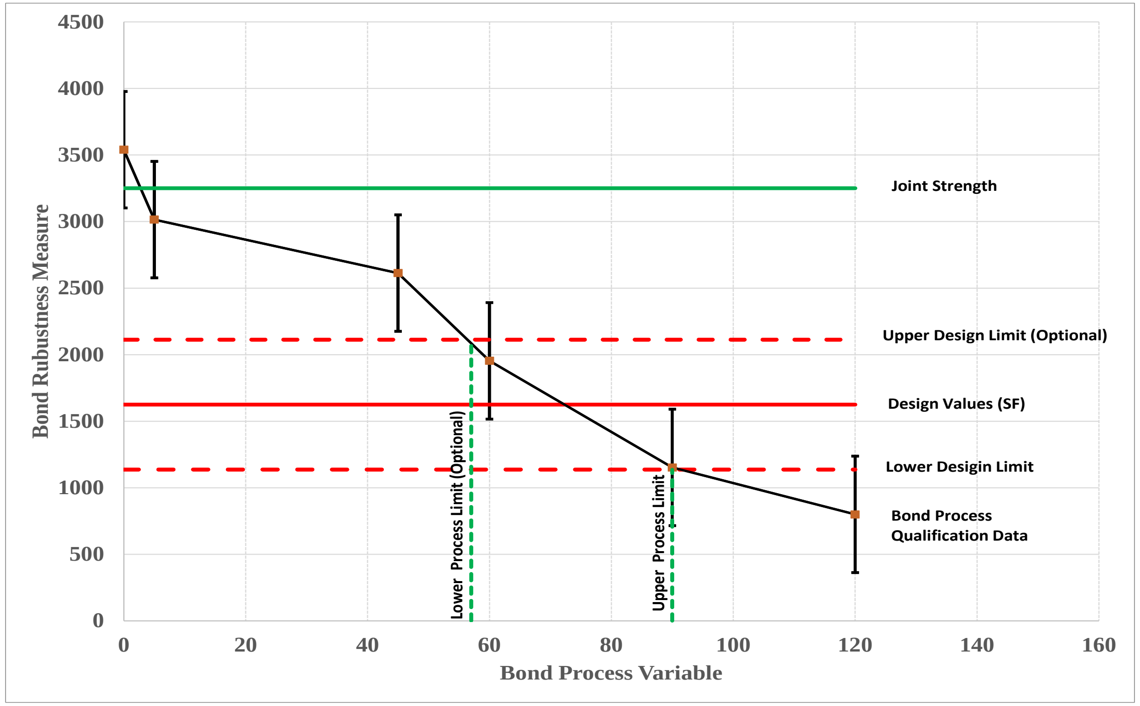


Adhesive Processing & Bonding Parameter Evaluation – Step 4 & 5

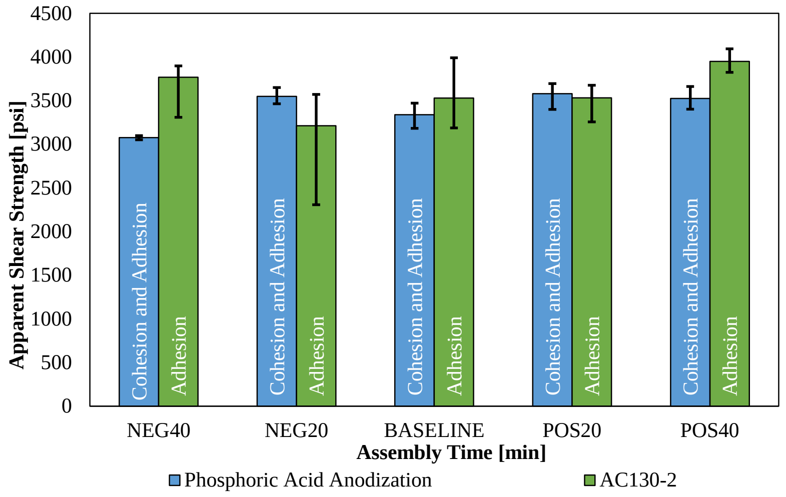
- Adhesive Processing Parameters
  - Adhesive Mix Ratio Effects (2 part adhesives)
  - Assembly Time
  - Adhesive Mixing Methodology (2 part adhesives)
  - Adhesive Application Methodology
  - Bondline Control Mechanisms
- Adhesive Curing
  - Primary Cure Cycle
  - Post Cure Cycle

- Test Methods used for Adhesive Processing and & Bonding Parameter Evaluation
  - ASTM D3165/ASTM D1002 – Single Lap Shear Strength
  - ASTM D5528 – Mode 1 Fracture Toughness
  - Adhesive Glass Transition Temperature
  - Degree of Cure of Adhesives

Joint Property Under Investigation	Bond System Component Under Investigation			
	Surface Preparation		Adhesive Preparation and Cure Process	
	Surface Preparation Method	Test Method	Surface Preparation Method	Test Method
Peel/Fracture Toughness/Mode I	Variable	ASTM D5528	Fixed Based on Step 2	ASTM D5528
Shear		ASTM D3165		ASTM D3165



Adhesive Processing & Bonding Parameter Evaluation
Effects of Mix Ratio, Assembly Time, and Adhesive Application Method



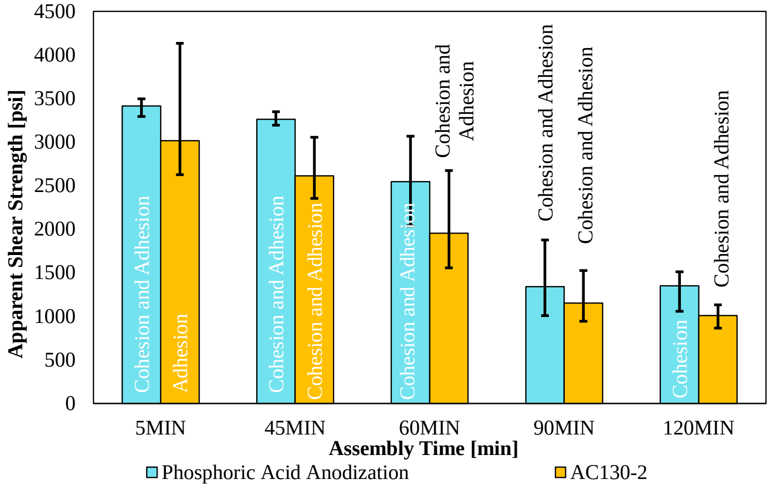
Effects of adhesive mix ratio

Material: Al 2024 T3

Surface Prep: PAA + BR127 and AC130-2 + BR 6747-1

Adhesive: EA 9394

Test Method: ASTM D1002



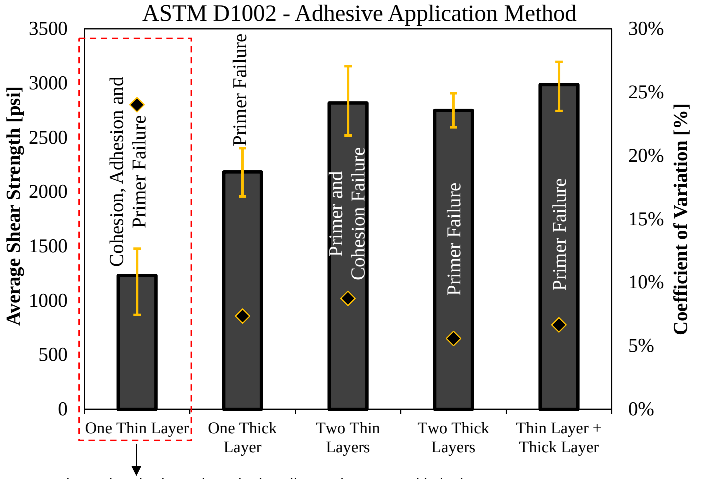
Effects of assembly time

Material: Al 2024 T3

Surface Prep: PAA + BR127 and AC130-2 + BR 6747-1

Adhesive: EA 9394

Test Method: ASTM D1002



C-scan showed multiple voids in the bondline – this is most likely due to a lack of adhesive

Effects of adhesive application method

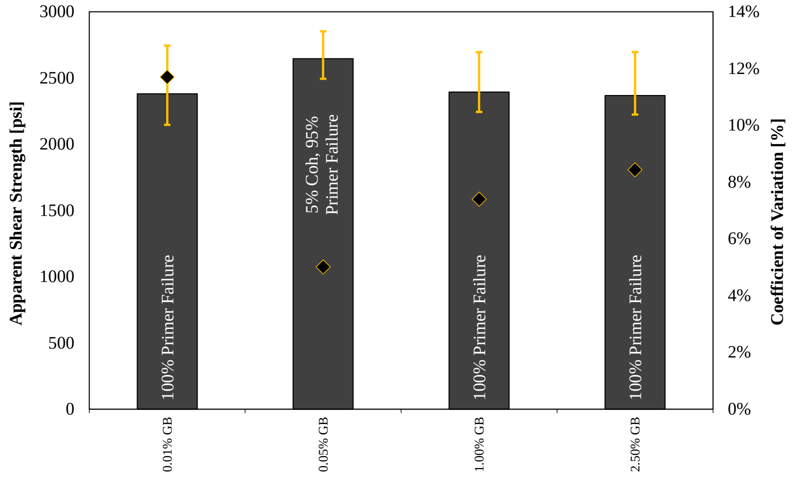
Material: Al 2024 T3

Surface Prep: AC130-2 + BR 6747-1

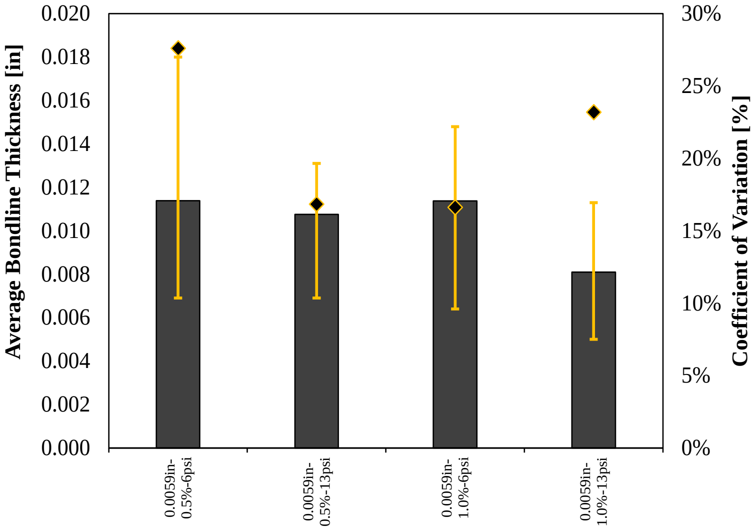
Adhesive: EA 9394

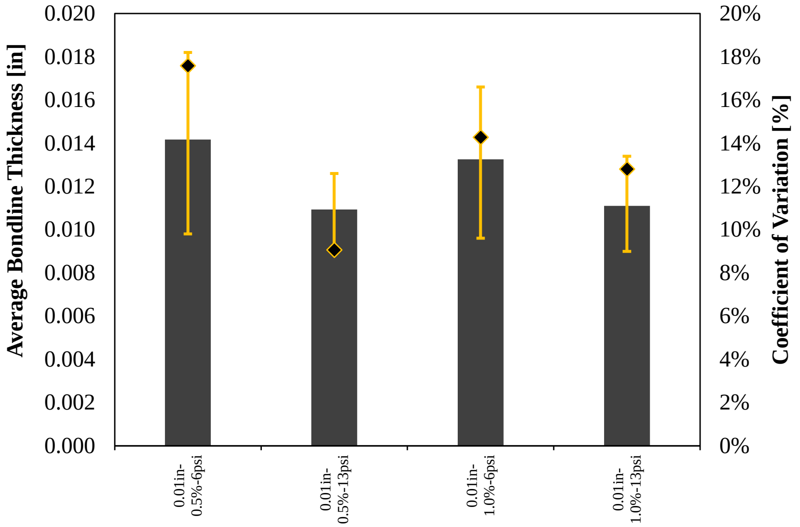
Test Method: ASTM D1002

Adhesive Processing & Bonding Parameter Evaluation
Bondline Thickness Control Mechanisms



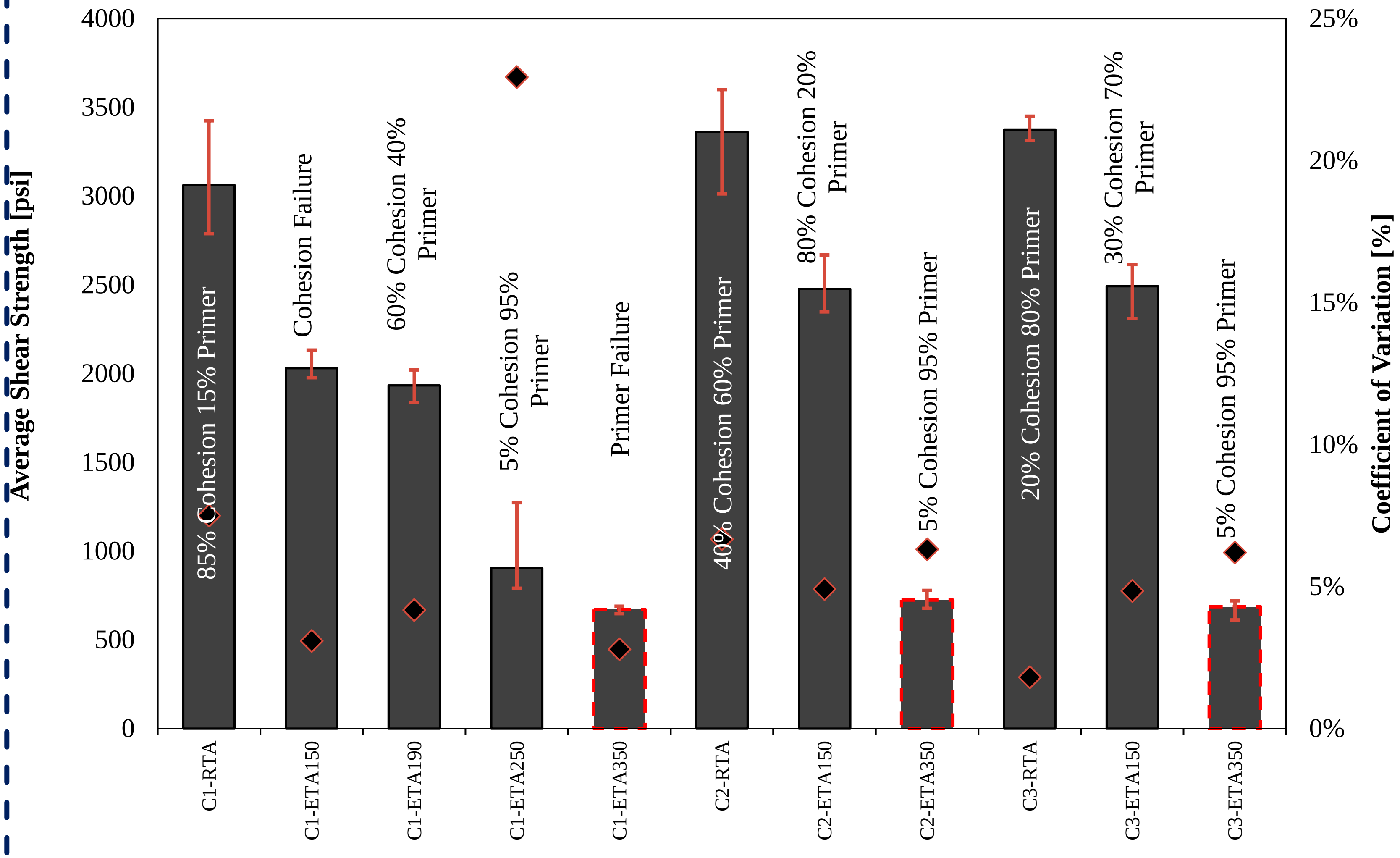
Effects of bondline thickness control mechanisms
Material: Al 2024 T3
Surface Prep: AC130-2 + BR 6747-1
Adhesive: EA 9394

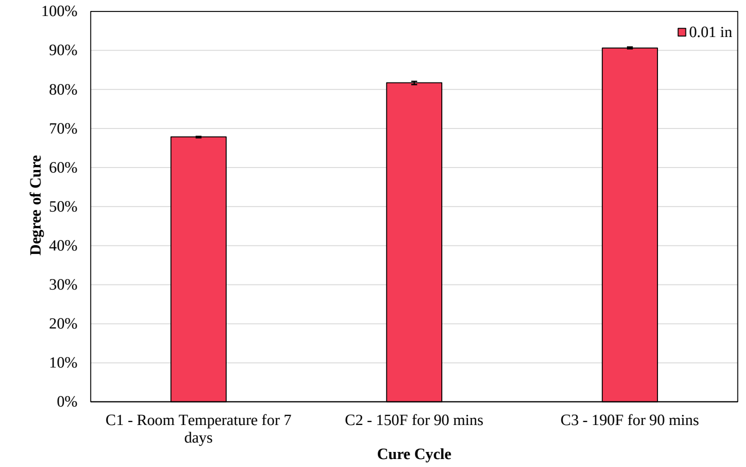


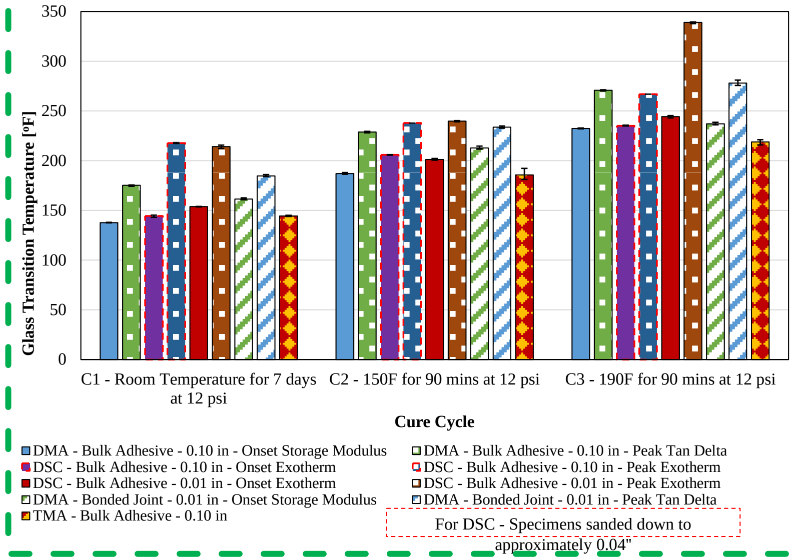


Effects of pressure application on bondline thickness
Material: T800H/3900-2
Surface Prep: None - only evaluated for bondline thickness

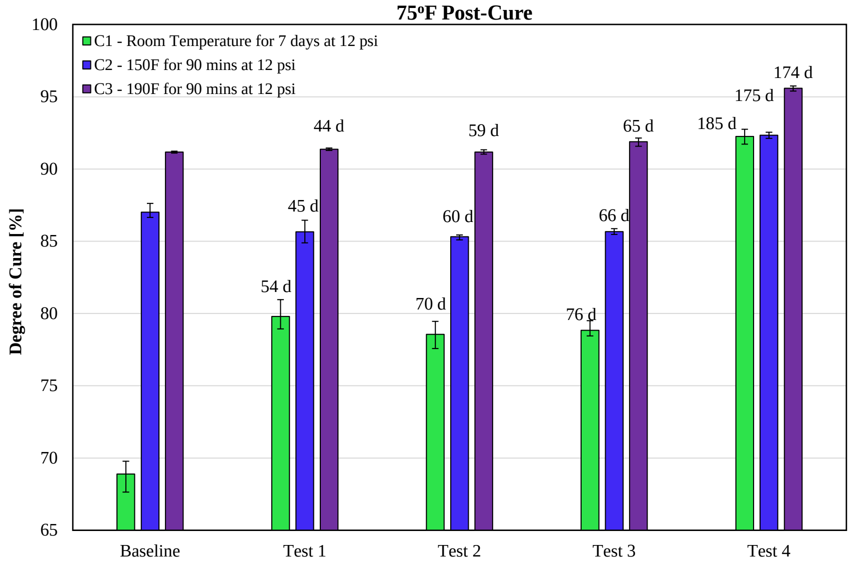
Adhesive Processing & Bonding Parameter Evaluation
Effects of Cure Cycle on Mechanical Performance, Tg, and DoC

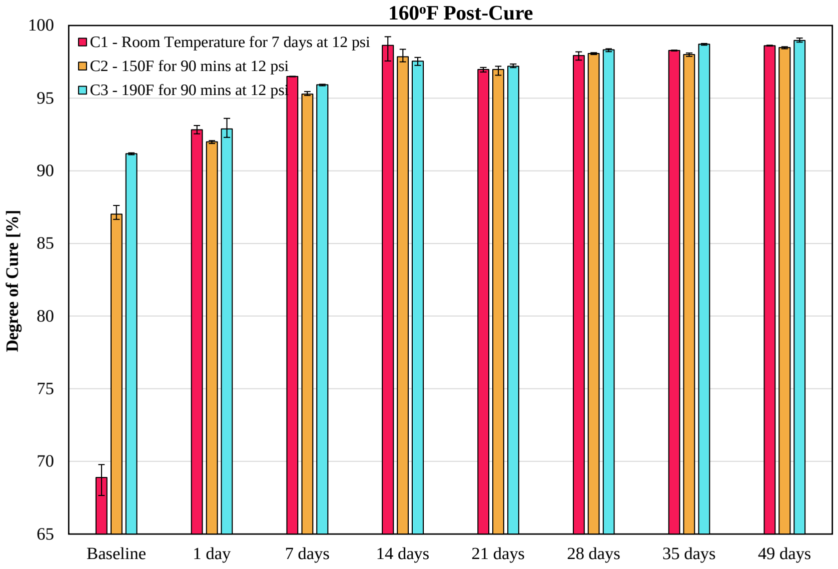


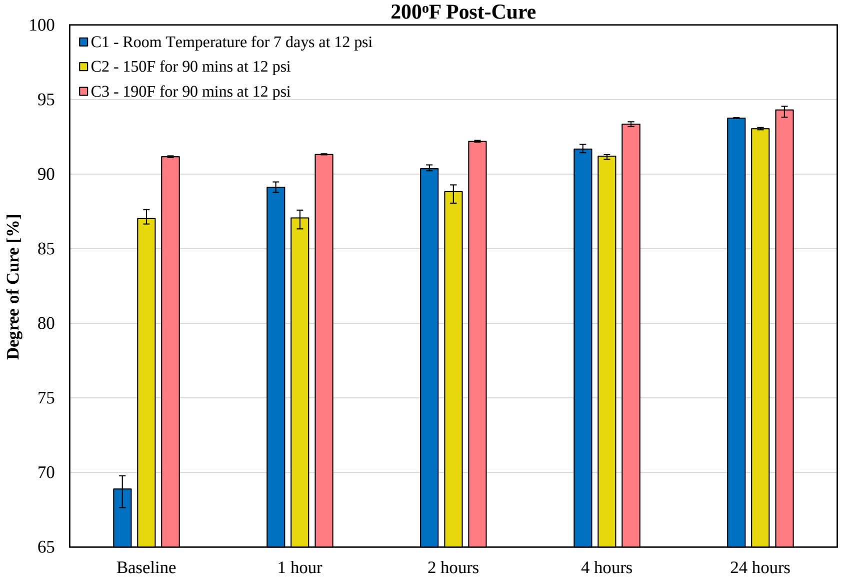


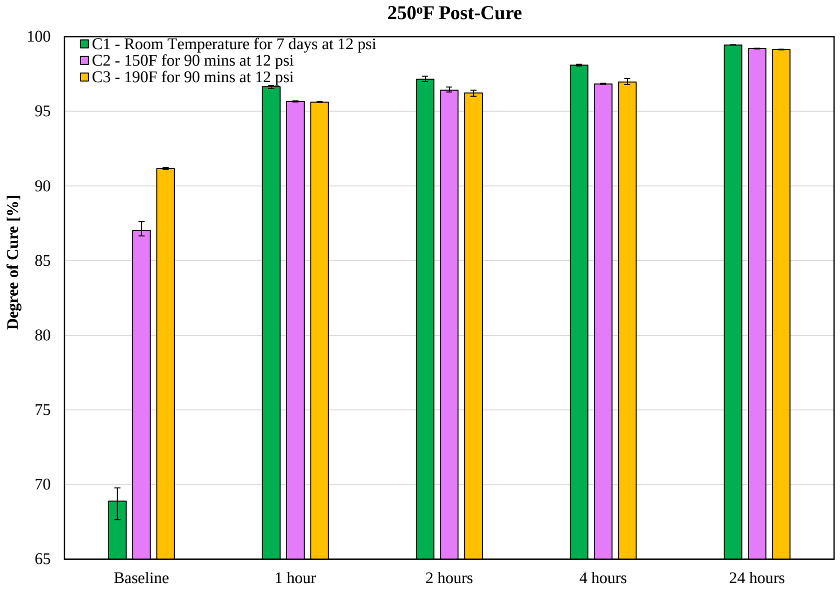


Adhesive Processing & Bonding Parameter Evaluation
Effects of Post Cure on Degree of Cure









Summary and Conclusions

- Bond system consists of four main components each containing multiple parameters that effects the integrity of the bonded joint.
- Guidelines are required to assess what parameters should be used and an quantitative measure to evaluate the effectiveness of each parameter.
- Standardized test methods and matrices are required to down select the various parameters in support of developing the protocols to quality the bond process of a joint.
- Guidelines are provided to support develop the bond process protocols and bond process qualification activities.

Thank You!

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