Adhesive Bond Qualification Guidance for Aircraft Design and Certification

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Joint Centers of Excellence for Advanced Materials

ATLAS
ADVANCED TECHNOLOGIES LAB FOR AEROSPACE SYSTEMS

Adhesive Bond Qualification Guidance for Aircraft Design and Certification

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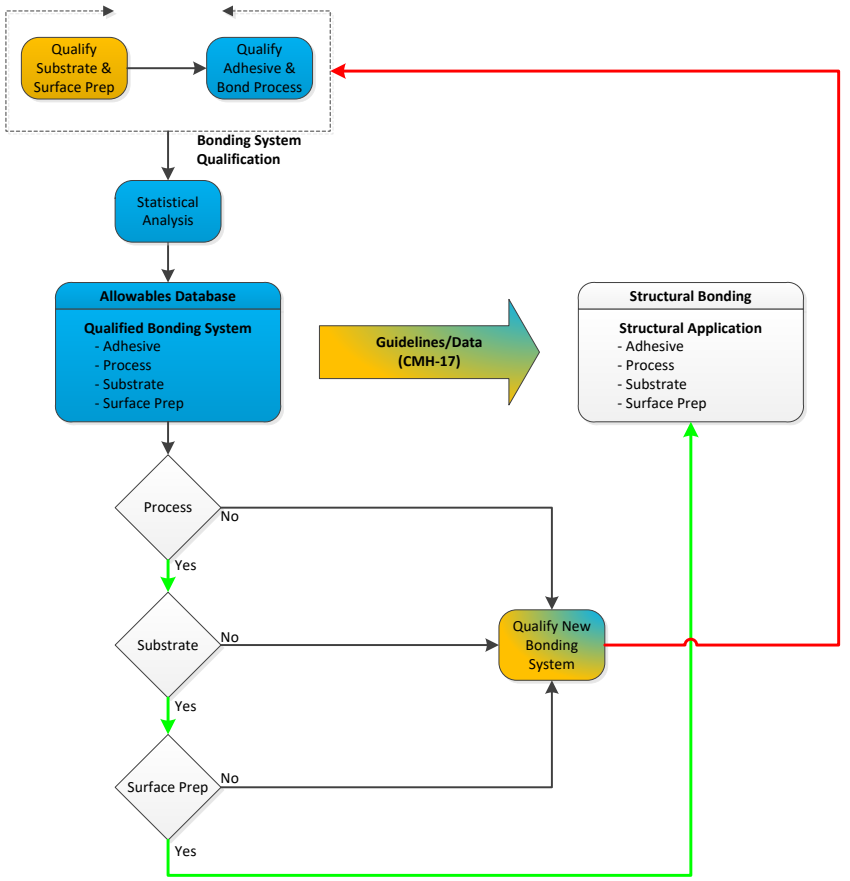
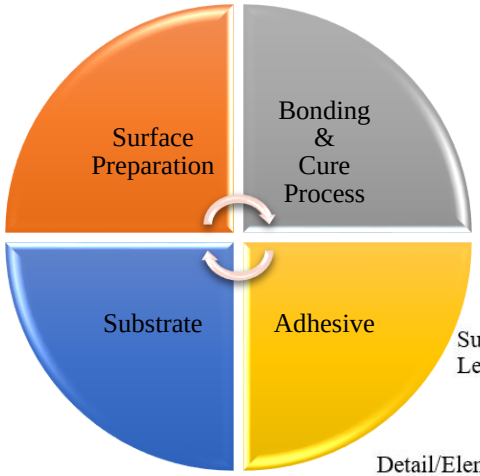
- DoD & Industry Participation
  - AFRL, Boeing, Bell Helicopter, Henkel, Honda Aircraft Co., Lockheed Martin, MMM, MTech Engineering Services, NAVAIR, Solvay Industries , Textron Aviation, Boom Aerospace

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



Bonded Joint Certification Approach



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

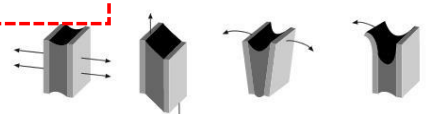
Preliminary Design Requirements/Knowledgebase of the Bonded Structure
1. General size
2. Mechanical property requirement for bonded joints
3. Environmental envelope
4. Manufacturing requirements
5. Analysis of critical bonded joint types seen in the structure
6. Bondline thickness requirements

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

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

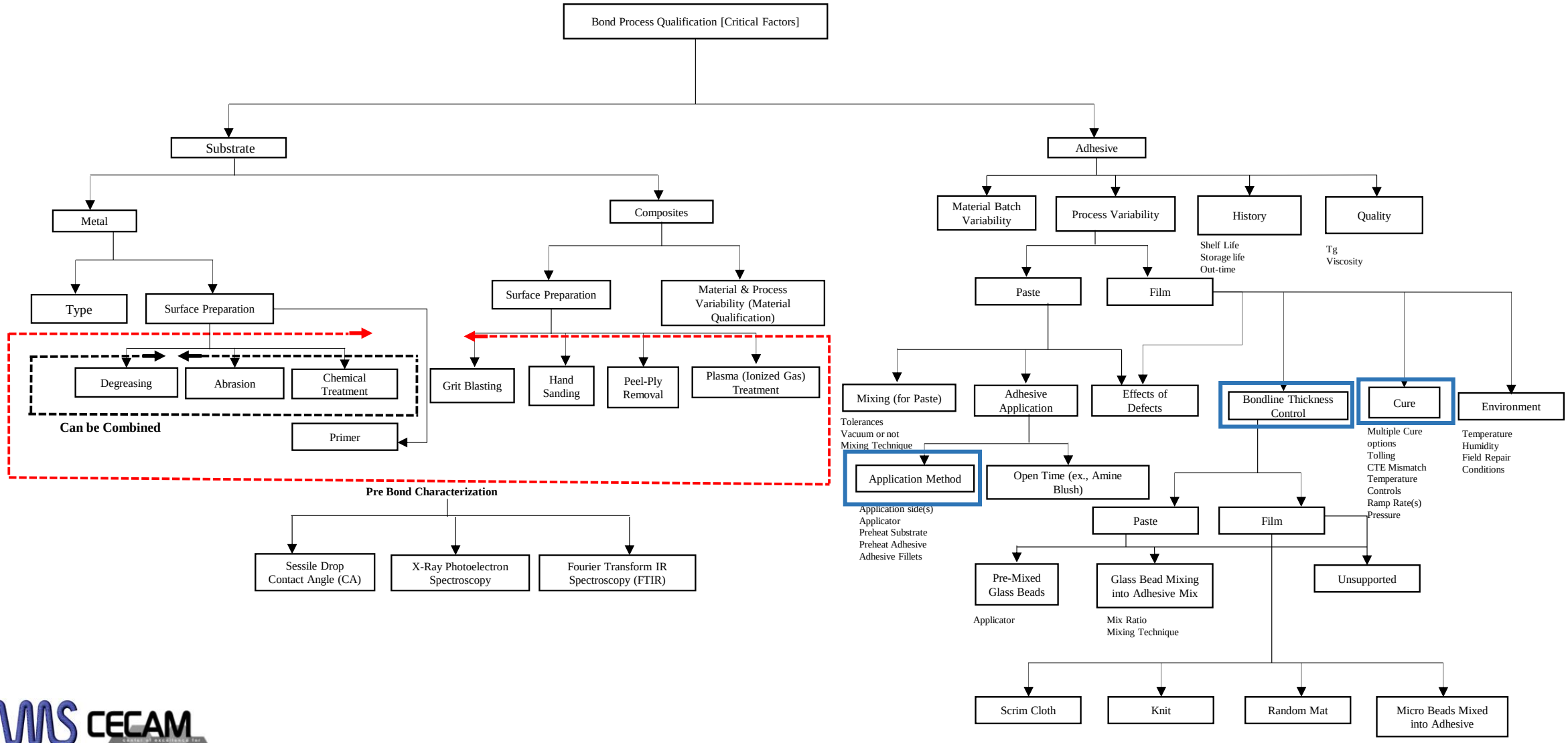
Maintenance
1. Inspection methodology development
2. Inspection methodology for bond strength degradation.
3. Identification of inspection level and frequency



Joint Property Under Investigation	Test Method: Bond System Component Under Investigation: Surface Preparation: Surface Preparation Method	Composites	Surface Preparation Method	Metallic	Adhesive and Cure Processing: Surface Preparation	Test Method (Composites & Metals)
Peel	Variable	ASTM D5528	Variable	ASTM D3167	Phosphoric Acid Anodization & Bond Primer [1]	ASTM D3167
Shear		ASTM D3165		ASTM D1002		ASTM D1002
Tensile		N/A		ASTM D897		ASTM D897
Fracture Toughness/Mode I		ASTM D5528		ASTM D3433	Atmospheric Plasma Treatment [1]	ASTM D5528(Metals) /D3433 (Composites)

Note 1 – Other compatible surface preparation methodologies can be used provided that no adhesion failures are observed during testing.

Bond Process Qualification (Critical Factors)



Critical Parameters in the Bonding Process

Surface Preparation

Manual and machine assisted abrasion:

- Grit size
- Type of grit (aluminum oxide, glass oxide etc.)
- Duration of sanding
- Frequency of sand paper change
- Direction of sanding
- Number of sanding passes
- Type of sander (for machine assisted) (Belt, Disc, Orbital, Random Orbital)
- Post treatment cleaning process
- Training level of technician

Grit Blasting

- Grit Size
- Grit Type
- Angle of application
- Nozzle configuration
- Pressure
- Number of passes
- Standoff distance
- Blasting configuration (Wet/dry)
- Post treatment cleaning process

Surface Preparation (Peel Ply)

- Peel ply specification
- Peel ply resin content (Dry/Wet)
  - Dry
    - Released
    - Unreleased
  - Wet
    - Resin type
- Material type
  - Polyester
  - Nylon
  - Glass
  - Kevlar
- Peel ply thickness
- Weave pattern
- Peel ply location on laminate (top
- Removal direction
- Removal time frame
- Post-removal cleaning
  - Solvent wipe
  - Compressed air
- Post-removal treatment
  - Abrasion
  - Chemical treatment

Surface Preparation (Plasma)

Atmospheric Plasma Treatment

- Plasma recipe
- Plasma nozzle type and configuration
- Nozzle height from the substrate
- Nozzle speed during plasma application
- Number of plasma passes
- Overlap of each plasma pass

Gas Plasma Treatment

- Plasma recipe
- Plasma nozzle type and configuration
- Nozzle height from the substrate
- Nozzle speed during plasma application
- Number of plasma passes
- Overlap of each plasma pass
- Type of gases utilized
- Gas flow rate
- Mix ratio of each gas

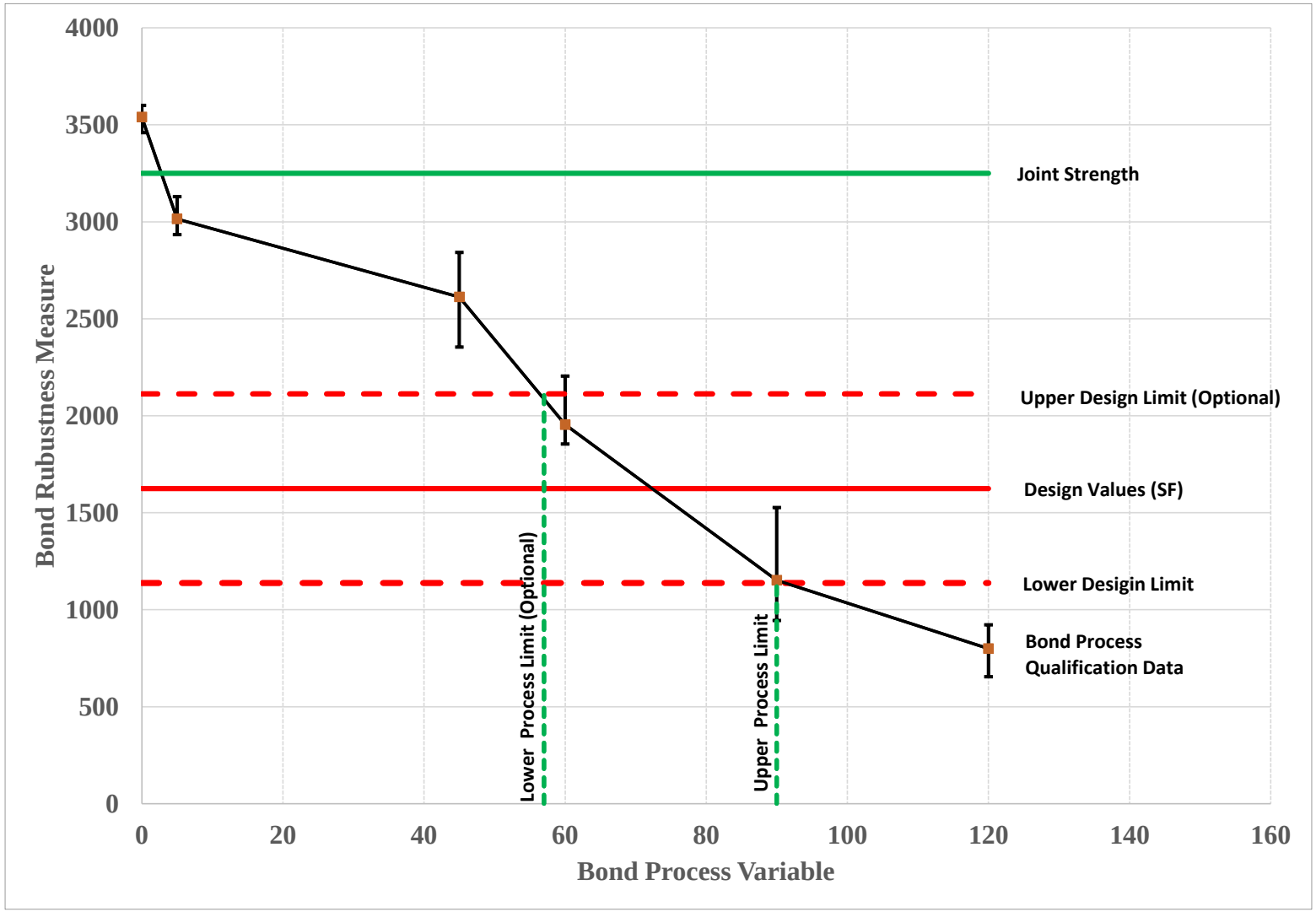
Substrate Cleaning

- Solvent type
- Solvent application
- Type of wipes/medium
- Frequency of wipe change
- Direction of the wipe
- Number of passes
- Satisfactory cleaning completion criteria

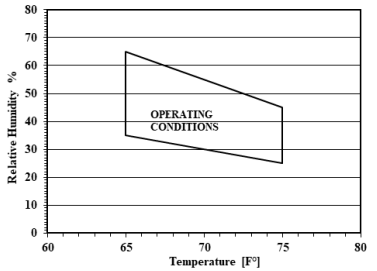
Adhesive Preparation, Mixing and Curing

- Storage environment and tolerances
- Duration of thawing after removing from cold storage
- Adhesive application temperature
- Minimum and maximum adhesive quantities to mix
- Mix ratio of multi part adhesive by weight or volume percent
- Adhesive mixing methodology
- Adhesive mixing duration and speed
- Trapped air removal mechanism
- Bondline thickness control
- Panel mating and pressure application
- Initial cure cycle & Post curing

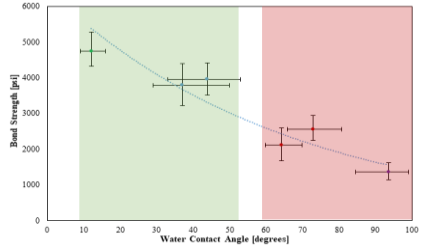
Bonding Process Limit Determination



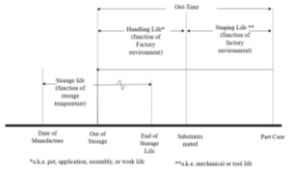
- Facility (Working Environmental Conditions)



- Surface preparation and quality assurance



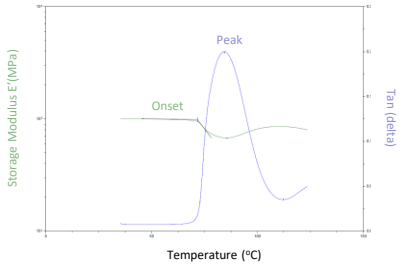
- Adhesive preparation/mixing and application

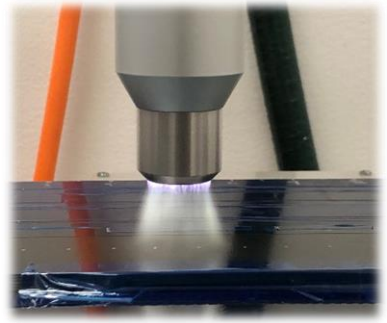


- Bonding, pressure application and curing

Effects of cure cycle and post cure (thermal exposure) on paste adhesive systems

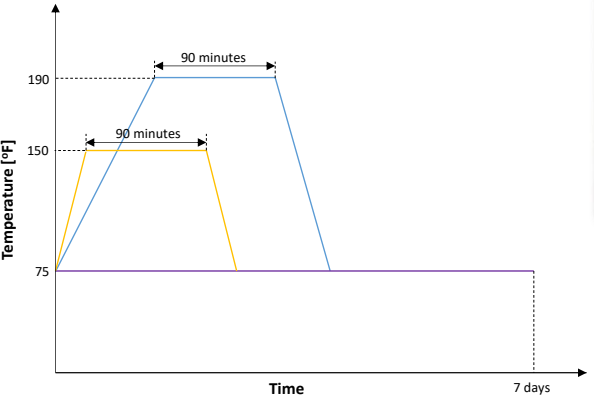
Task 1 - Evaluation of Degree of Cure of Adhesives and Effects of Degree of Cure on Static and Durability Performance of Adhesive Joints

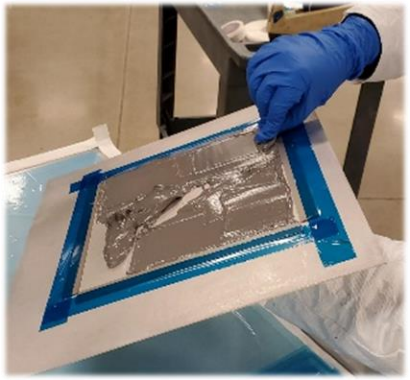


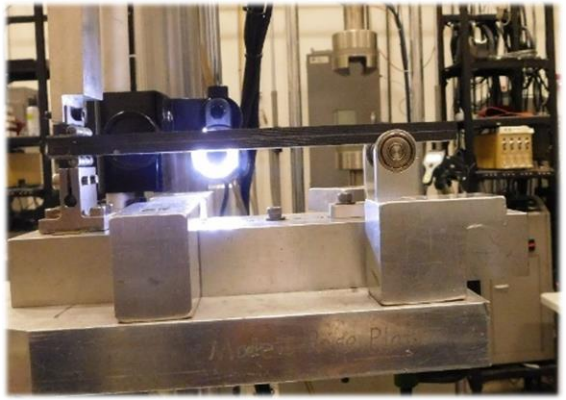


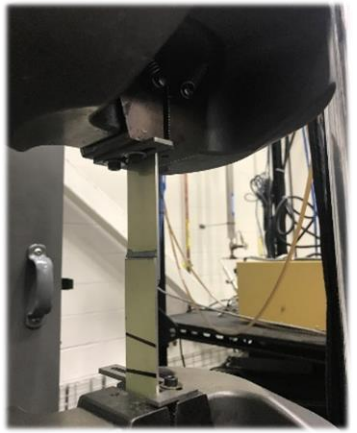
Task 1

- 1a - Evaluation of test methods to measure T_g
- 1b - Evaluation of static response of bonded joints with various degrees of cure and environmental conditions above T_g
- 1c - Evaluation of the effects of long term thermal exposure (above T_g)
- 1d - Evaluation of the durability of adhesives with varying degrees of cure
- 1e - Equivalency comparison of modified degree of cure using post curing methods









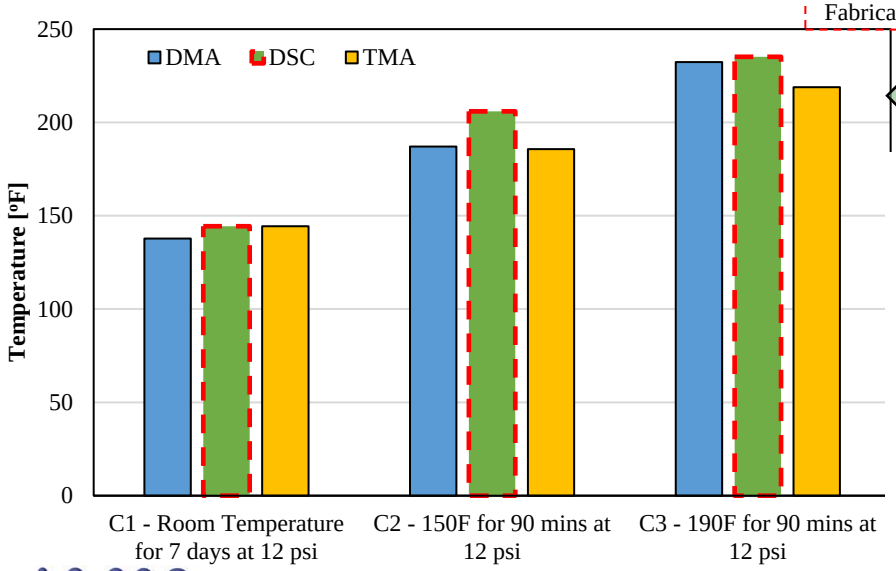
C1 - Room temperature for 7 days at 12 psi
C2 - 150°F for 90 minutes at 12 psi
C3 - 190°F for 90 minutes at 12 psi

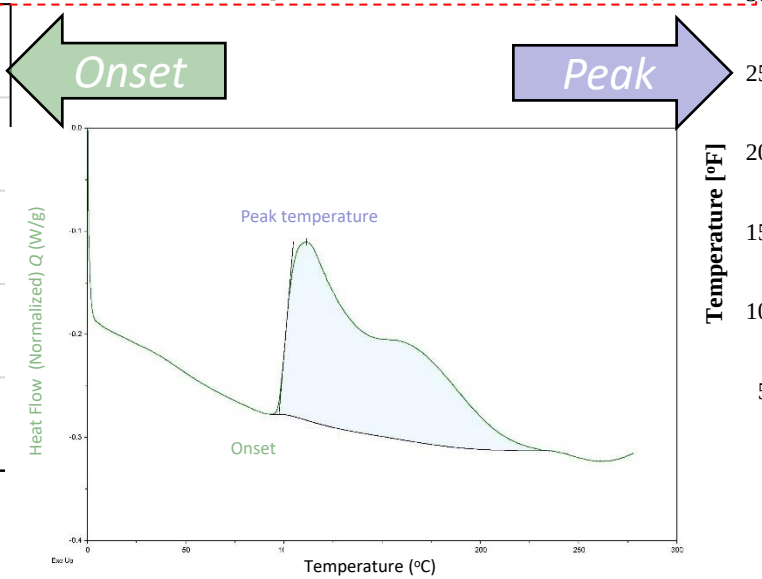
Task 1a – Glass Transition Temperature - Bulk Adhesive - 0.10”

Tg Test Method	Specimen Type	Adhesive Thickness [in]	Cure Cycle	Onset of Storage Modulus [°F]			Peak of Tan Delta [°F]			Onset of Exotherm [°F]			Peak of Exotherm [°F]			Change in CTE from Dimension Change Signal [°F]		
				Average	Max Error	Min Error	Average	Max Error	Min Error	Average	Max Error	Min Error	Average	Max Error	Min Error	Average	Max Error	Min Error
DMA	Bulk adhesive	0.10	C1	137.71	0.29	0.28	175.14	0.50	0.97									
			C2	187.01	1.05	0.75	228.84	0.68	0.87									
			C3	232.32	0.50	0.32	270.72	0.53	0.56									
DSC	Bulk adhesive	0.10*	C1							144.34	0.88	1.33	217.63	0.71	0.52			
			C2							205.90	0.23	0.41	237.90	0.10	0.17			
			C3							235.12	0.64	0.32	266.97	0.11	0.21			
TMA	Bulk adhesive	0.10	C1													144.39	0.71	0.55
			C2													185.68	6.56	4.55
			C3													218.82	2.29	2.97

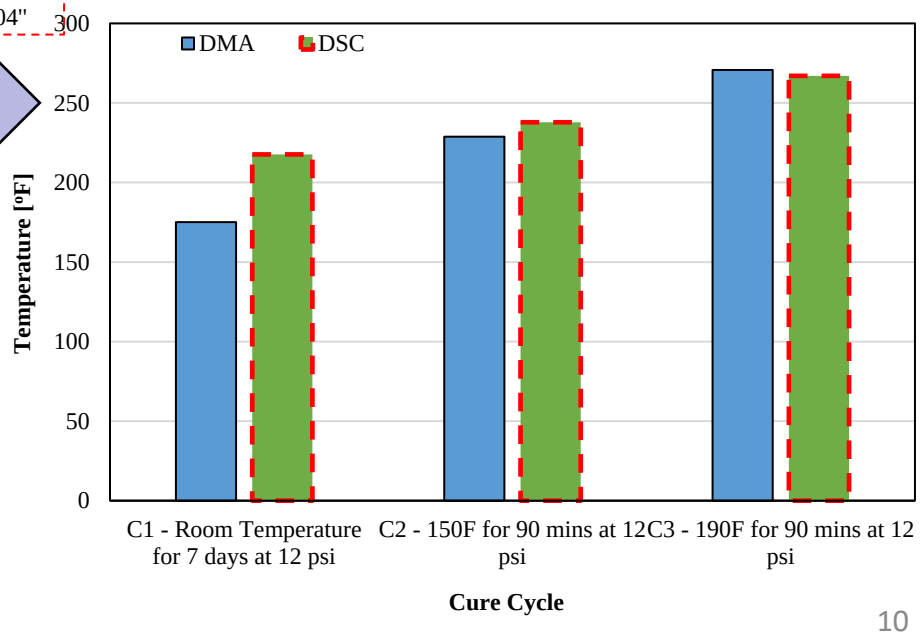
Note: TMA only records a change, there is no onset or peak. The change is closest to the onset values therefore TMA data is included only in the Onset graph.

Fabricated thickness. Final DSC specimens sanded down to approximately 0.04"



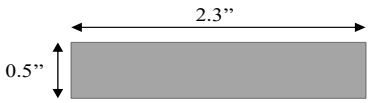


Sample DSC graph

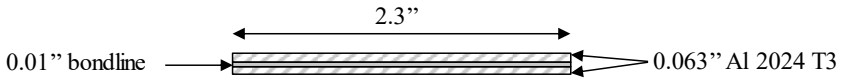


Task 1a – Glass Transition Temperature - Bulk Adhesive and Bonded Joint - DMA

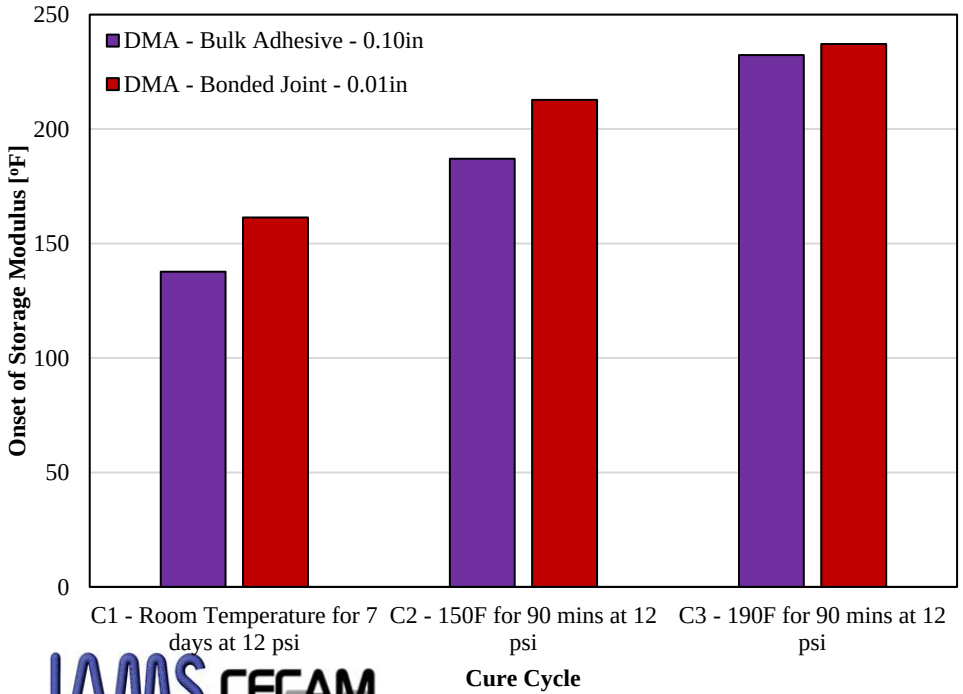
Tg Test Method	Specimen Type	Adhesive Thickness [in]	Cure Cycle	Onset of Storage Modulus [°F]			Peak of Tan Delta [°F]		
				Average	Max Error	Min Error	Average	Max Error	Min Error
DMA	Bulk adhesive	0.10	C1	137.71	0.29	0.28	175.14	0.50	0.97
			C2	187.01	1.05	0.75	228.84	0.68	0.87
			C3	232.32	0.50	0.32	270.72	0.53	0.56
	Bonded joint	0.01	C1	161.41	1.18	0.66	184.63	1.32	0.68
			C2	212.77	1.82	1.19	233.65	1.27	0.98
			C3	237.11	1.46	1.06	277.99	3.00	2.36

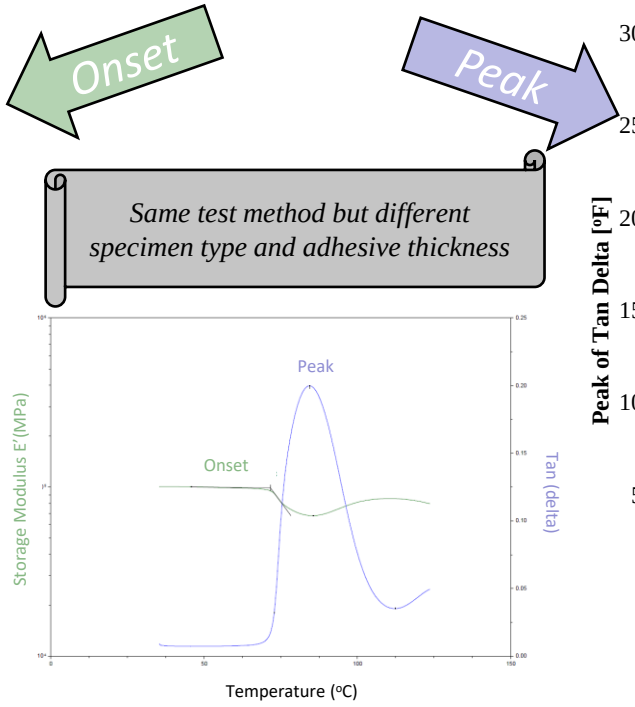


DMA specimen – 0.10”

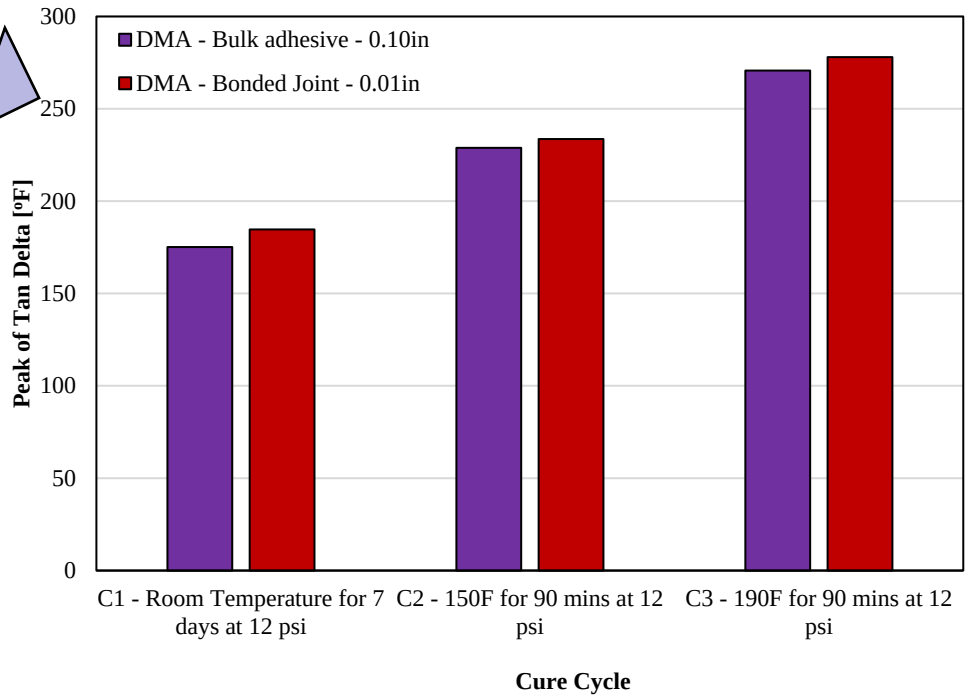


DMA specimen – 0.01”



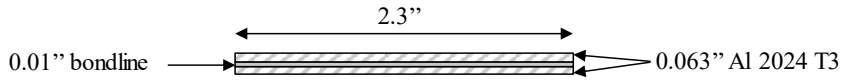


Sample DMA graph

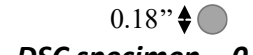


Task 1a – Glass Transition Temperature - Bulk Adhesive and Bonded Joint - 0.01”

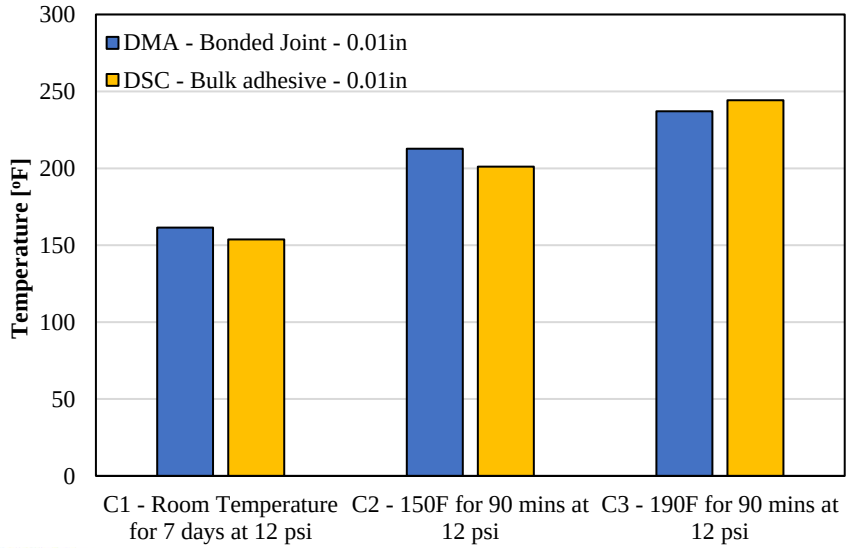
Tg Test Method	Specimen Type	Adhesive Thickness [in]	Cure Cycle	Onset of Storage Modulus [°F]			Peak of Tan Delta [°F]			Onset of Exotherm [°F]			Peak of Exotherm [°F]		
				Average	Max Error	Min Error	Average	Max Error	Min Error	Average	Max Error	Min Error	Average	Max Error	Min Error
DMA	Bonded joint	0.01	C1	161.41	1.18	0.66	184.63	1.32	0.68						
			C2	212.77	1.82	1.19	233.65	1.27	0.98						
			C3	237.11	1.46	1.06	277.99	3.00	2.36						
DSC	Bulk adhesive	0.01	C1							153.78	0.28	0.22	214.17	1.48	1.79
			C2							201.10	1.07	0.64	239.70	0.60	0.61
			C3							244.25	1.12	1.34	339.02	0.60	1.20



DMA specimen – 0.01”



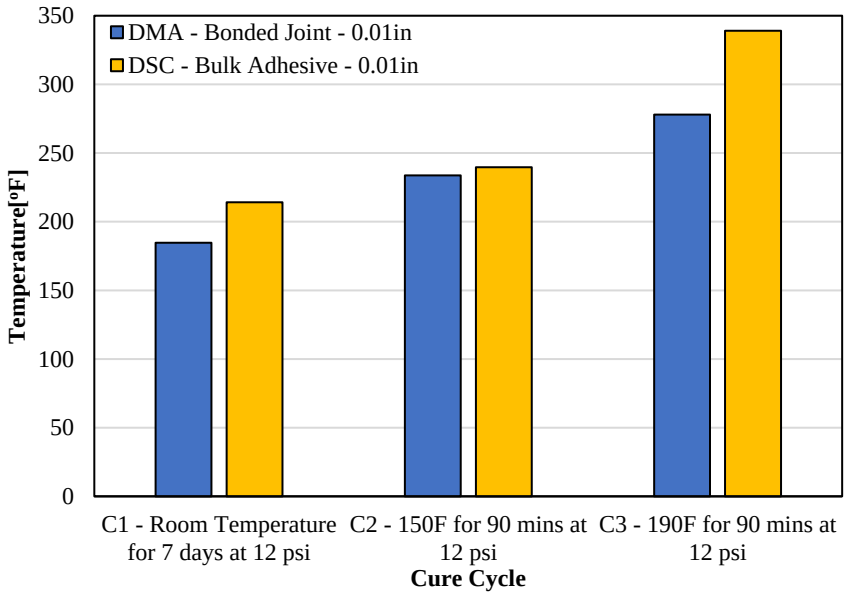
DSC specimen – 0.01”



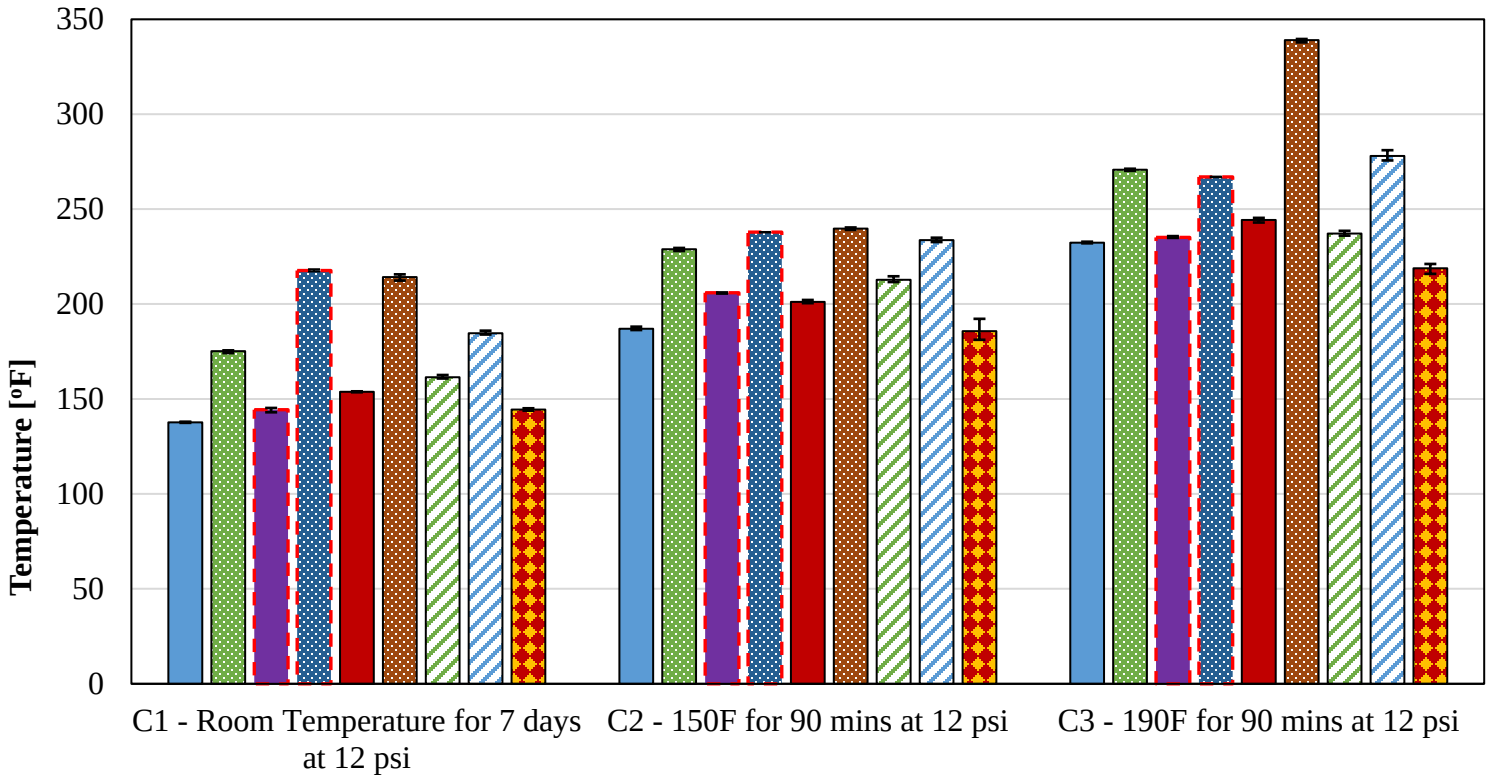
Same adhesive thickness but the specimen type and evaluation methods differed.

Onset

Peak

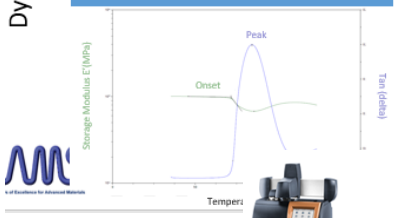


Task 1a – Glass Transition Temperature



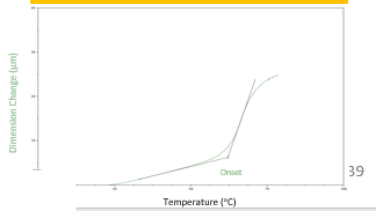
Dynamic Mechanical Analysis (DMA)

- Tg
- TA Instruments Discovery DMA 850
- Specimen dimensions - 2.3'' x 0.5'' , 0.08'' < T < 0.16''
- ASTM D7028
- Temperature at which a significant change occurs in storage modulus is recorded as Tg.



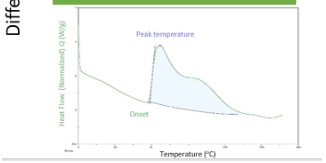
Thermo-Mechanical Analysis (TMA)

- T_g
- TA Instruments Discovery TMA 450
- Specimen dimensions - 0.3'' x 0.3'' x T > 0.08''
- ASTM E1545
- Temperature at which a significant change in CTE occurs is recorded as T_g.



Differential Scanning Calorimetry (DSC)

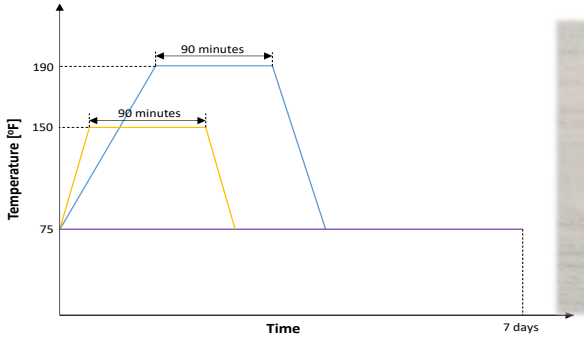
- DOC and Tg
- TA Instruments Discovery DSC 2500
- Specimen dimensions - 0.18'' diameter, T< 0.04''
- DOC - ASTM D3418
- Tg - ASTM E1356
- Temperature at which a change in the heat flow profile occurs is recorded as Tg.
- Heat of reaction of cured sample and uncured sample will be compared to obtain DOC

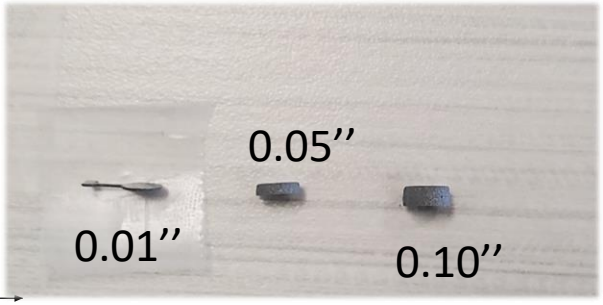


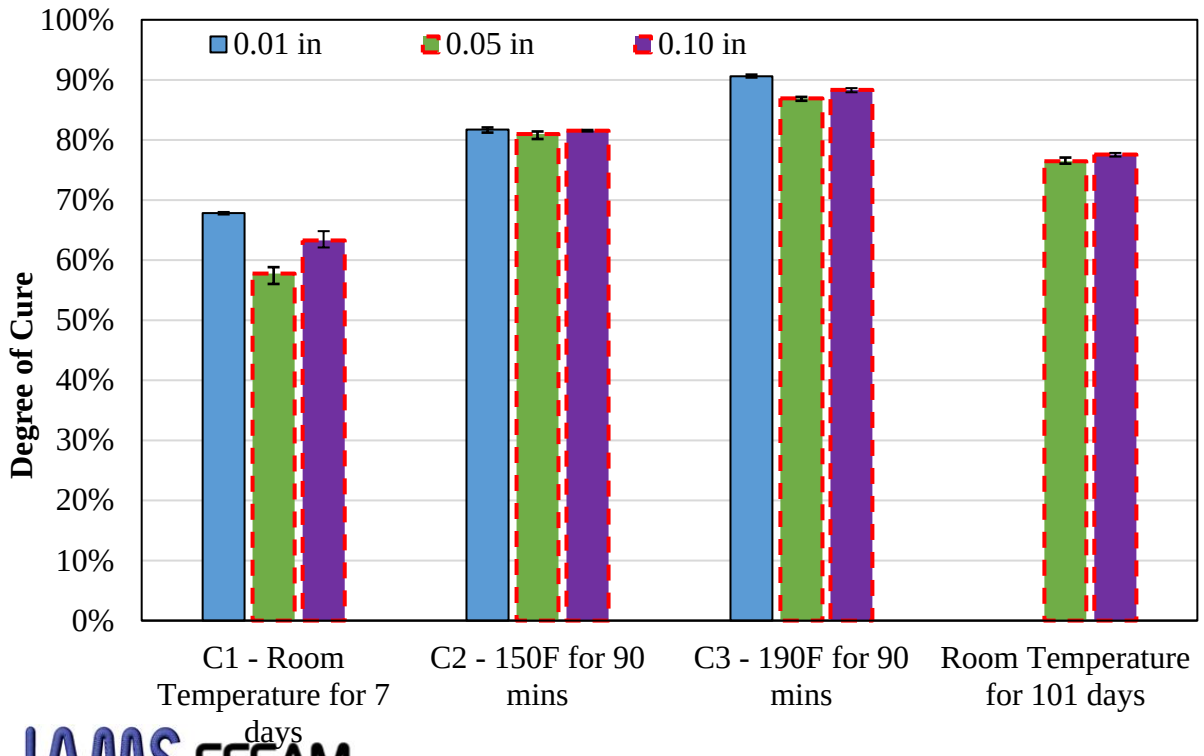
For DSC - Specimens sanded down to approximately 0.04"

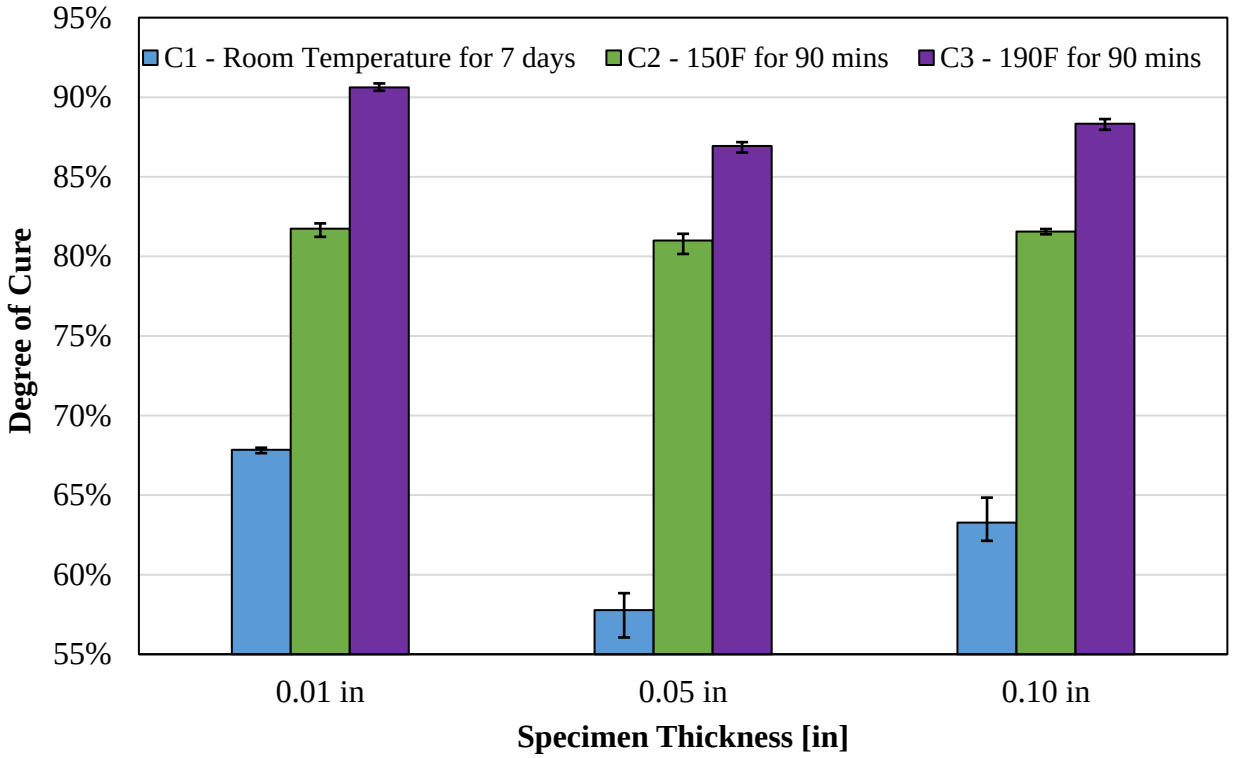
Task 1a – Degree of Cure

- Degree of Cure
  - The 0.10’’ and 0.05’’ specimens were hand sanded to approximately 0.04’’. This was due to the height limitations of the DSC machine.
    - Precautions were taken to minimize the heat generation during the machining process.
  - Additional specimens from the C1 0.10’’ and 0.05’’ panels were tested 101 days after the initial cure.

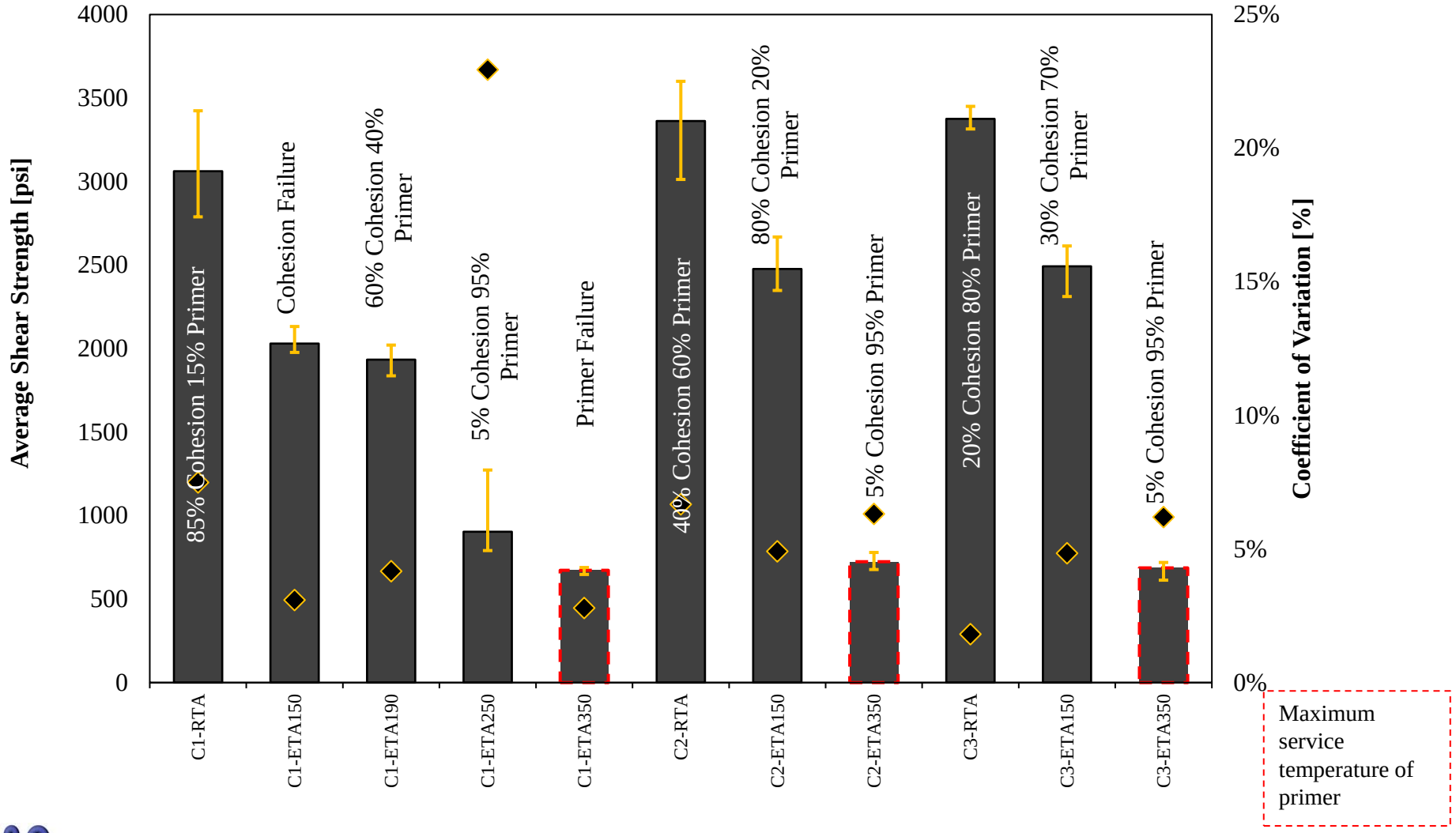




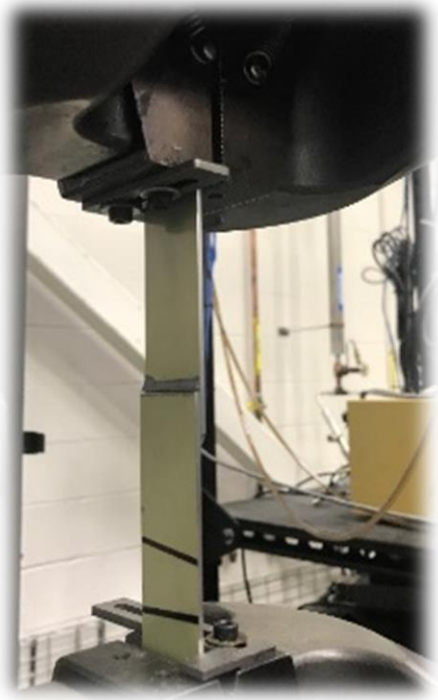




Task 1b – D1002 Mechanical Test Data



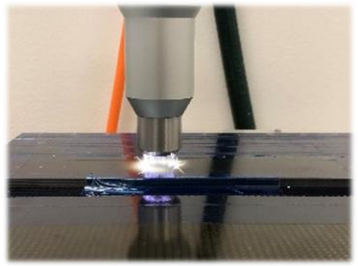
Maximum service temperature of primer

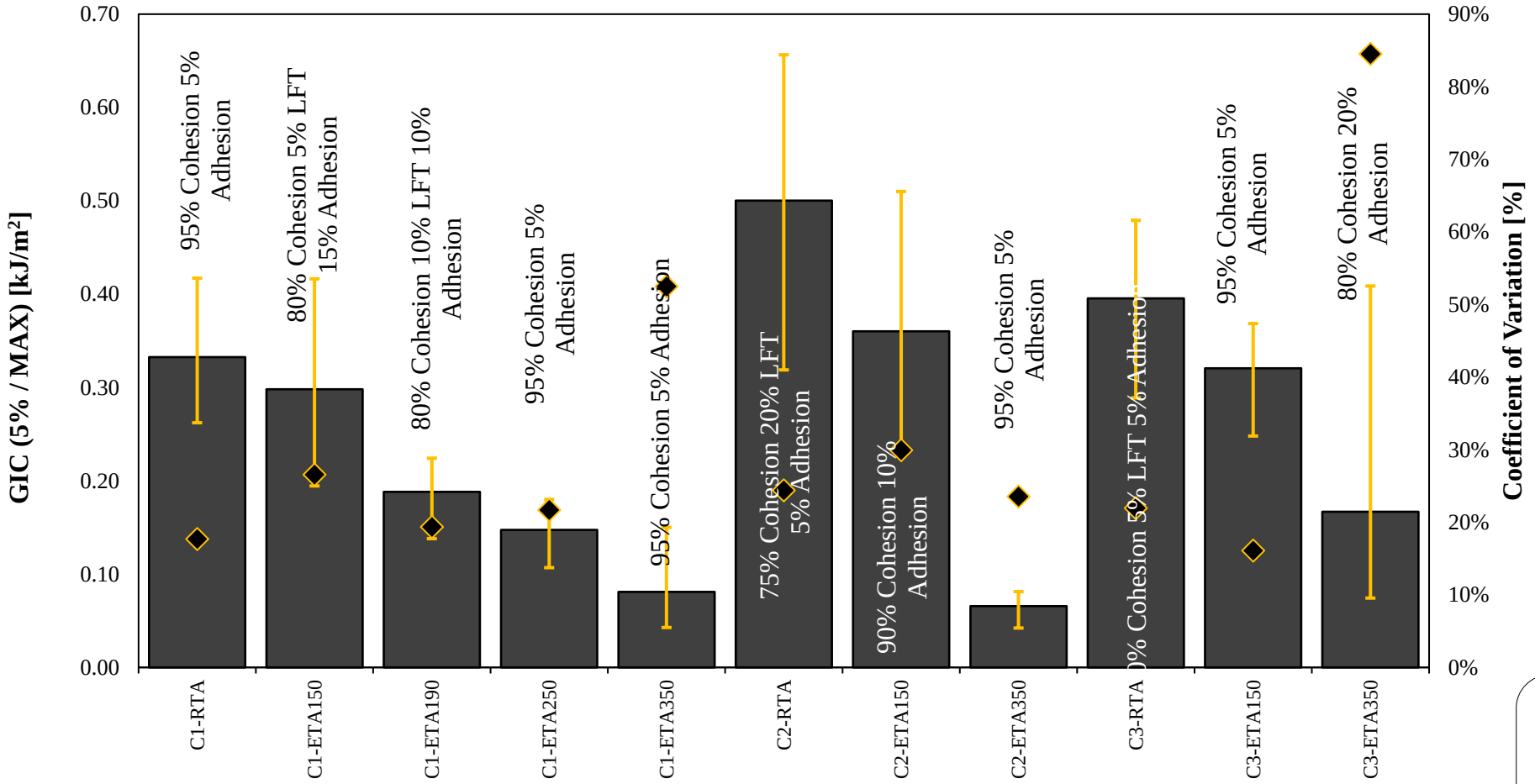


T_g
C1 - 138°F
C2 - 187°F
C3 - 232°F

Worst failure mode (highest % of adhesion failure) from each panel is given in the data labels

Task 1b - D5528 Mechanical Test Data





T_g

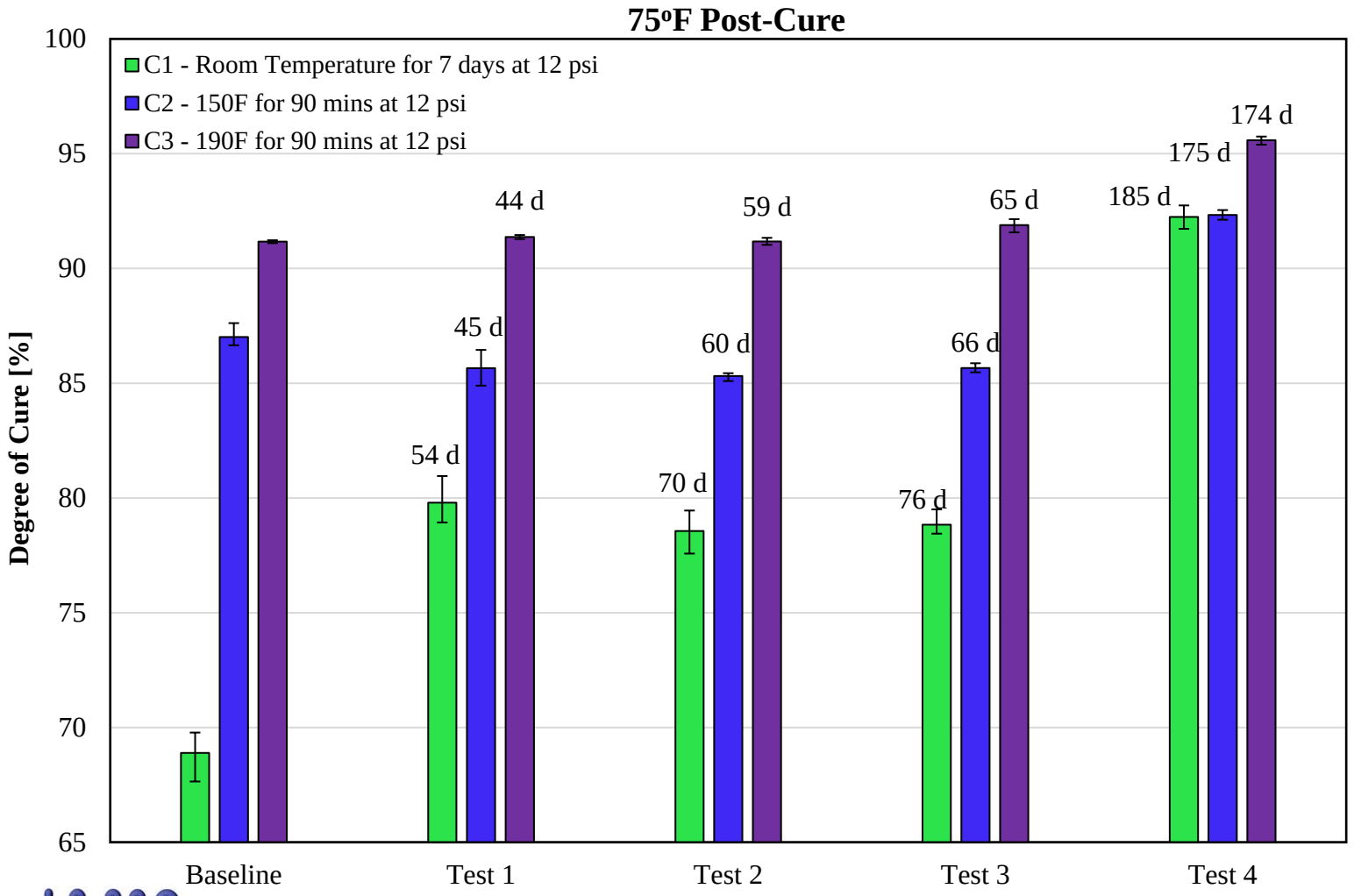
C1 - 138°F

C2 - 187°F

C3 - 232°F

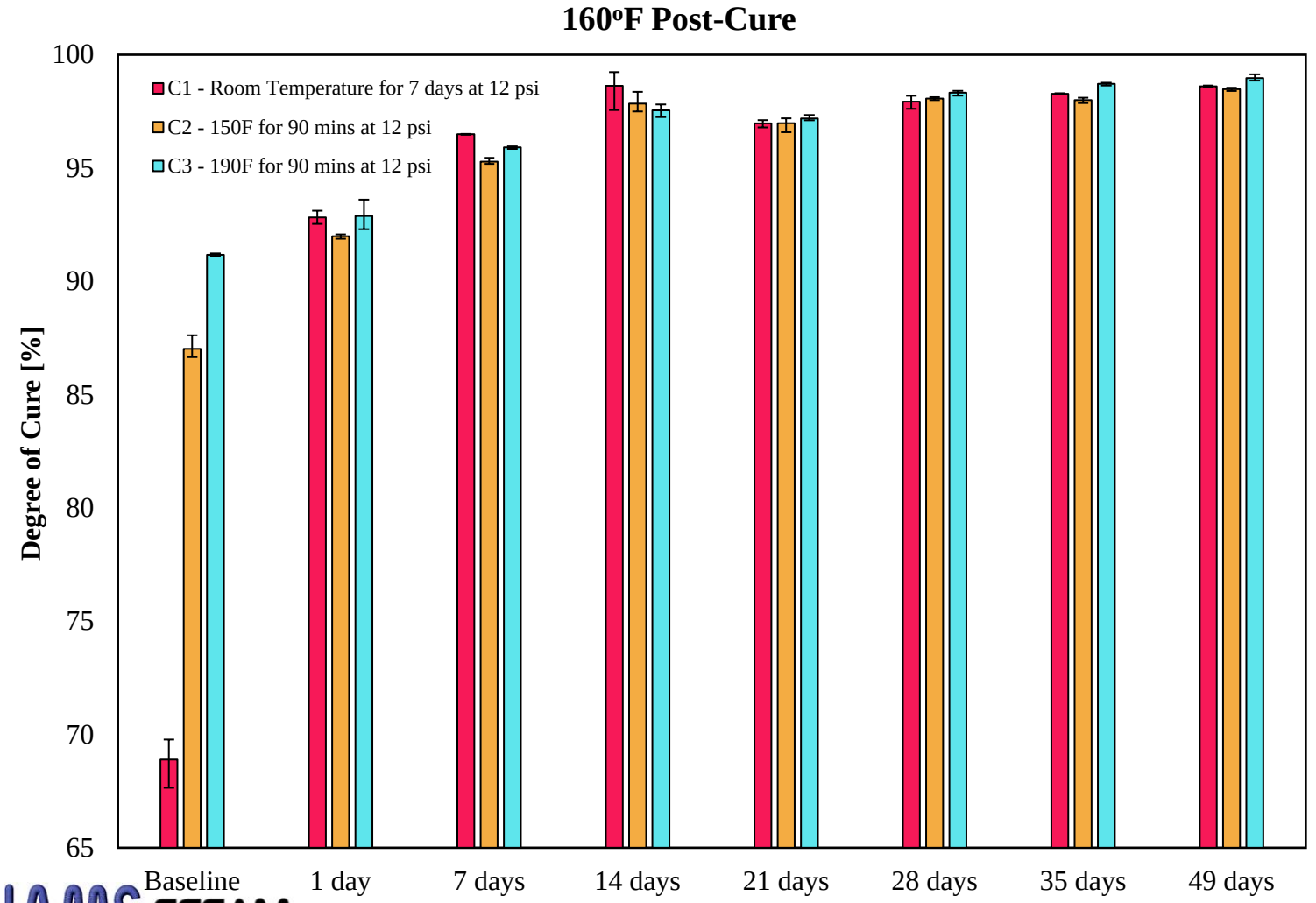
Worst failure mode (highest % of adhesion failure) from each panel is given in the data labels

Task 1c - Evaluation of the effects of long term thermal exposure above the glass transition temperature



Exposure Duration	Degree of Cure [%]		
	C1	C2	C3
Baseline	92.82	91.99	92.88
Test 1	79.79	85.66	91.37
Test 2	78.56	85.32	91.18
Test 3	78.84	85.66	91.89
Test 4	92.25	92.33	95.59

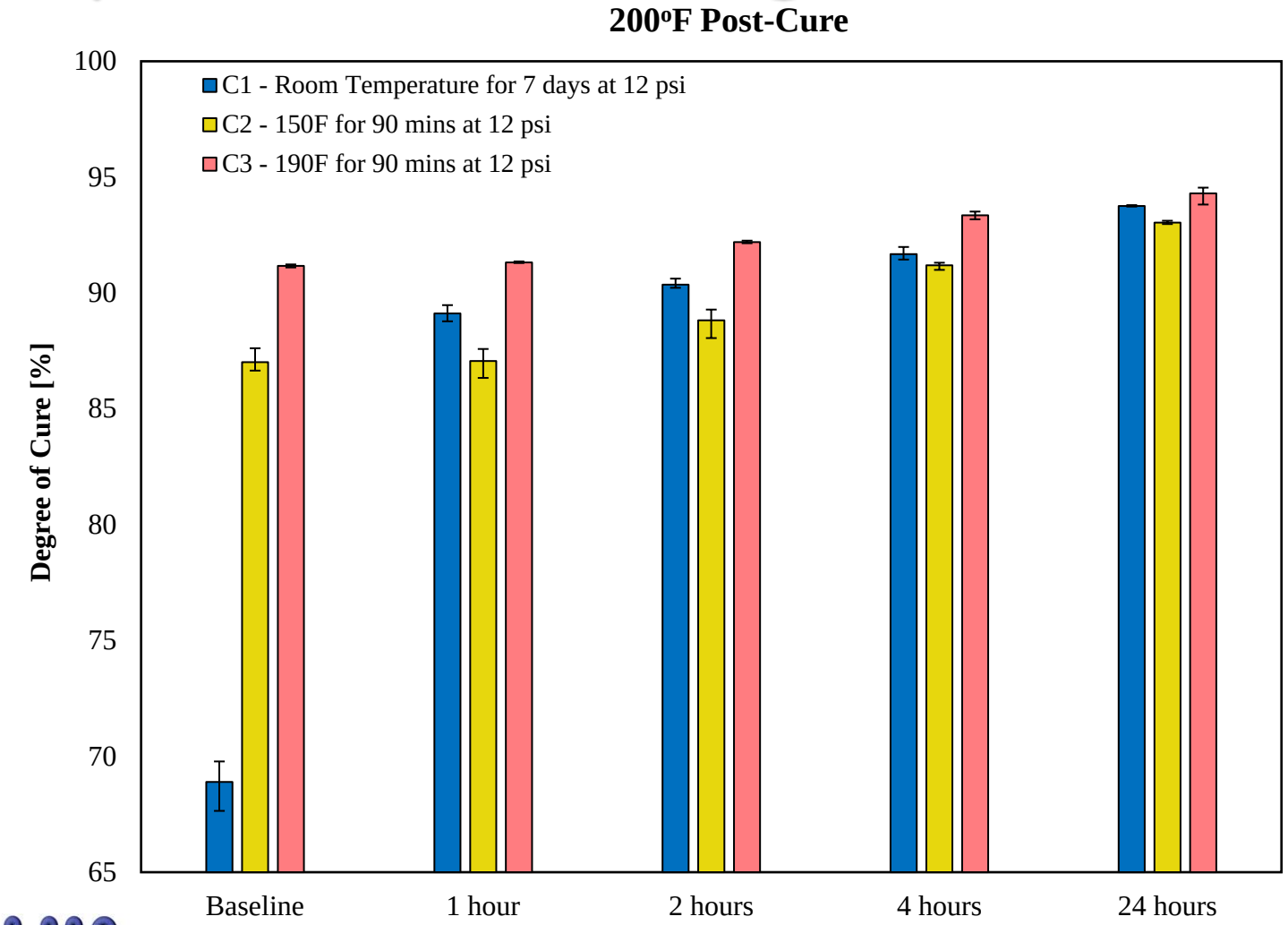
Task 1c - Evaluation of the effects of long term thermal exposure above the glass transition temperature



Exposure Duration	Degree of Cure [%]		
	C1	C2	C3
1 day	92.82	91.99	92.88
7 days	96.50	95.28	95.92
14 days	98.63	97.85	97.54
21 days	96.97	96.97	97.19
28 days	97.93	98.07	98.32
35 days	98.28	98.00	98.72
49 days	98.61	98.48	98.97

Specimens from all three cure cycles have reached at least 98% DoC after 49 days at 160°F

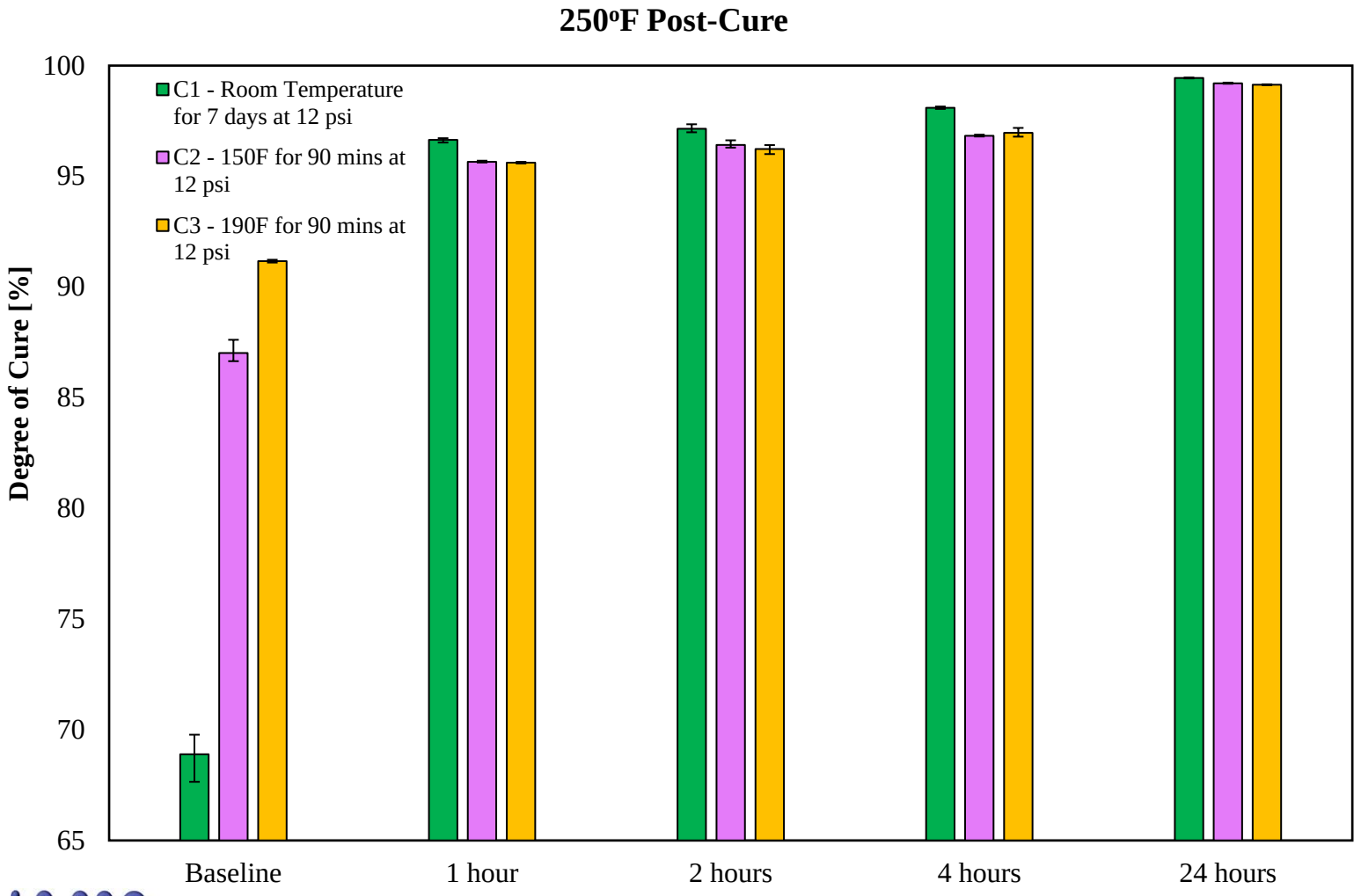
Task 1c - Evaluation of the effects of long term thermal exposure above the glass transition temperature



Exposure Duration	Degree of Cure [%]		
	C1	C2	C3
Baseline	68.89	87.02	91.17
1 hour	89.12	87.07	91.32
2 hours	90.36	88.82	92.20
4 hours	91.67	91.20	93.35
24 hours	93.76	93.04	94.30

Specimens from all three cure cycles have reached at least 93% DoC after 24 hours at 200°F

Task 1c - Evaluation of the effects of long term thermal exposure above the glass transition temperature

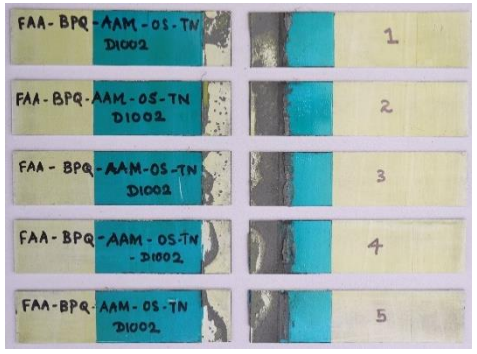


Exposure Duration	Degree of Cure [%]		
	C1	C2	C3
Baseline	68.89	87.02	91.17
1 hour	96.65	95.66	95.61
2 hours	97.15	96.42	96.23
4 hours	98.10	96.83	96.96
24 hours	99.44	99.20	99.14

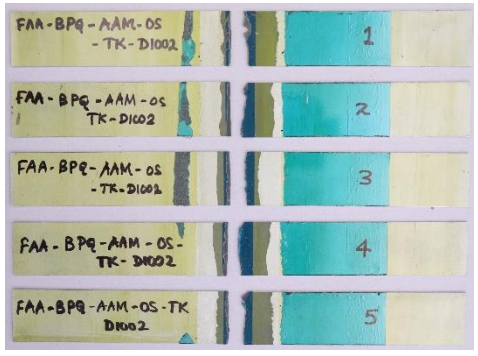
Specimens from all three cure cycles have reached at least 99% DoC after 24 hours at 250°F

Adhesive Mixing and Application

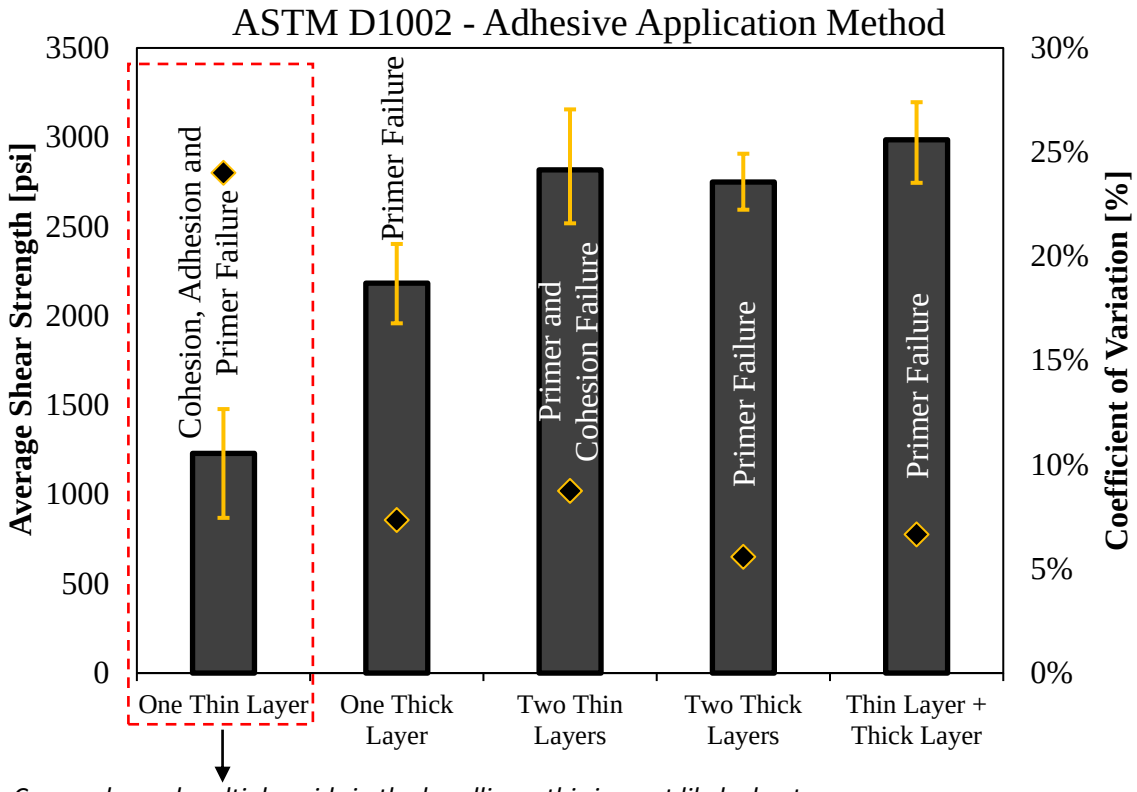
Task 2b – Adhesive Application Methodology



One Thin Layer

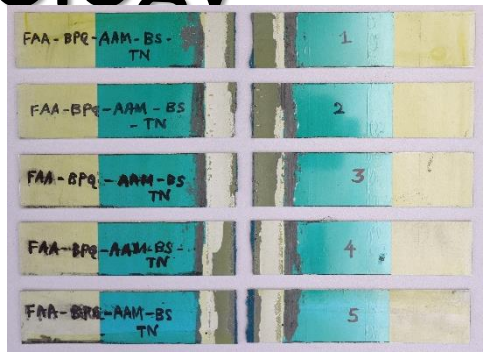


One Thick Layer

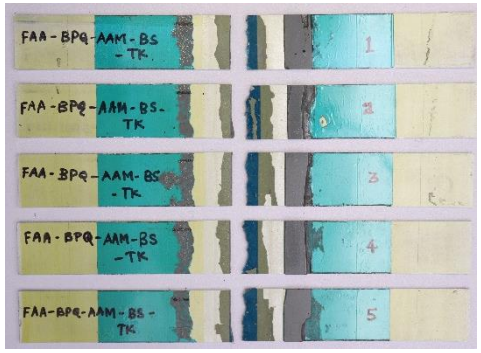


C-scan showed multiple voids in the bondline – this is most likely due to a lack of adhesive

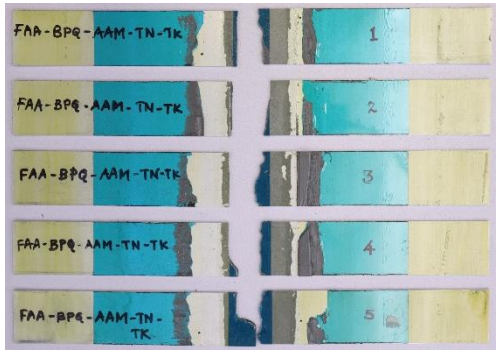
Configuration	Average Bondline Thickness [in]	CoV	Maximum Thickness [in]	Minimum Thickness [in]
One Thin Layer	0.008	7%	0.008	0.007
One Thick Layer	0.009	2%	0.010	0.009
Two Thin Layers	0.010	4%	0.011	0.010
Two Thick Layers	0.010	5%	0.011	0.009
Thin Layer + Thick Layer	0.009	3%	0.009	0.009



Two Thin Layers



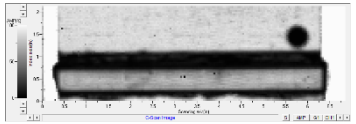
Two Thick Layers



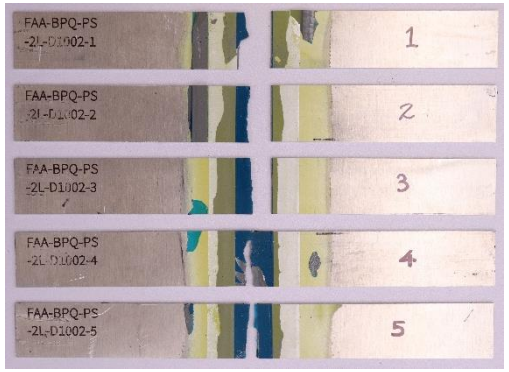
Thin Layer + Thick Layer

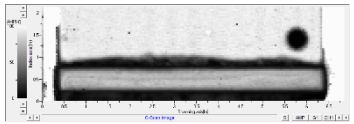
Task 2b – Primer Layer Study

- The data show that as the number of primer layers increase, the shear strength decreases.

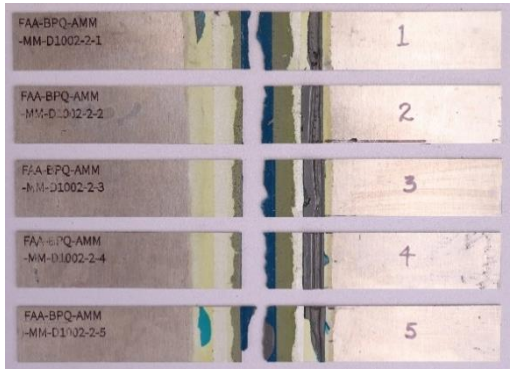


1 Layer

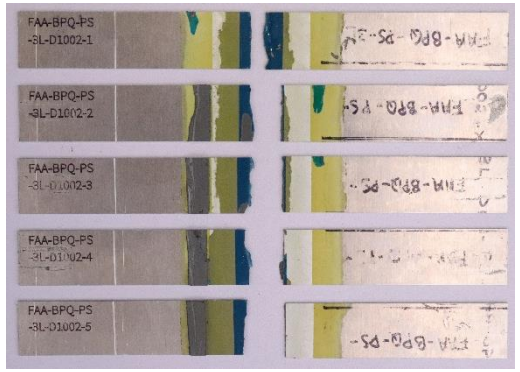


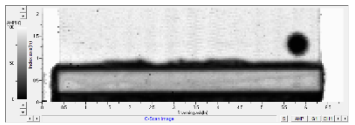


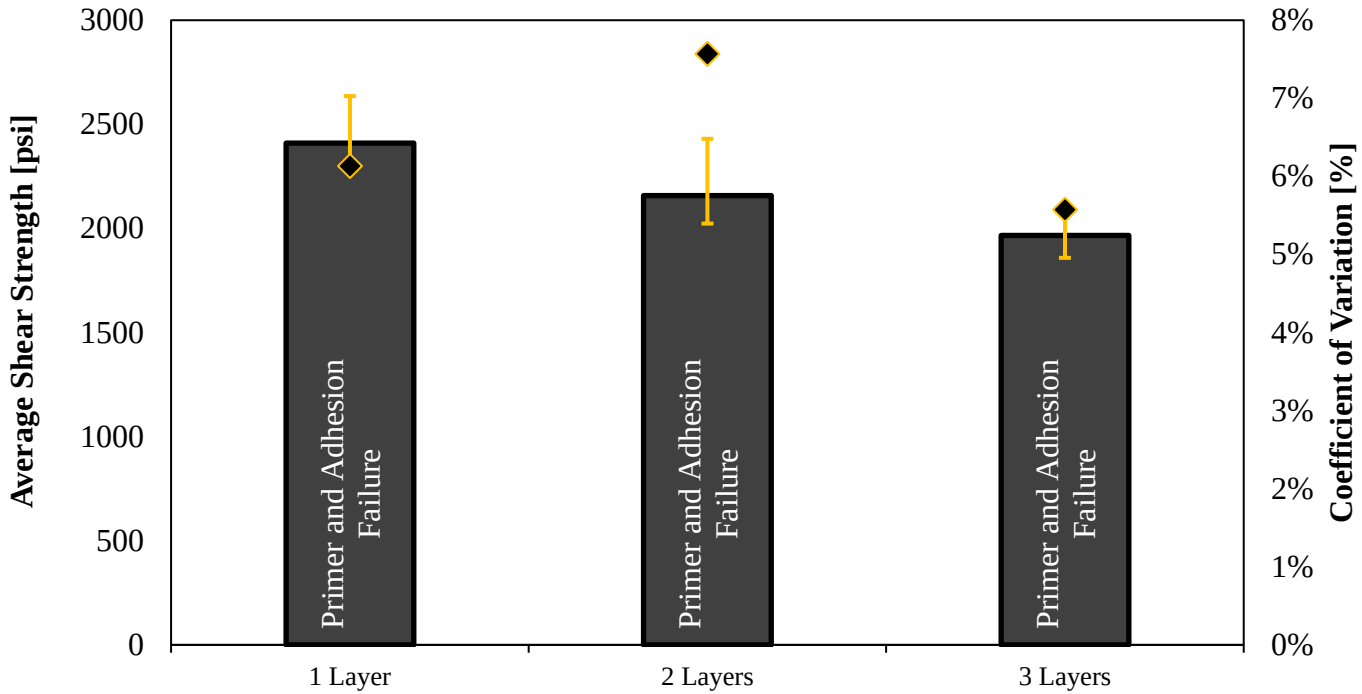
2 Layers



3 Layers



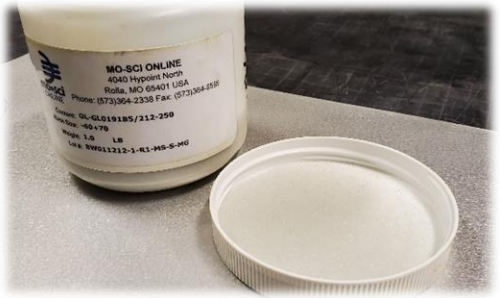


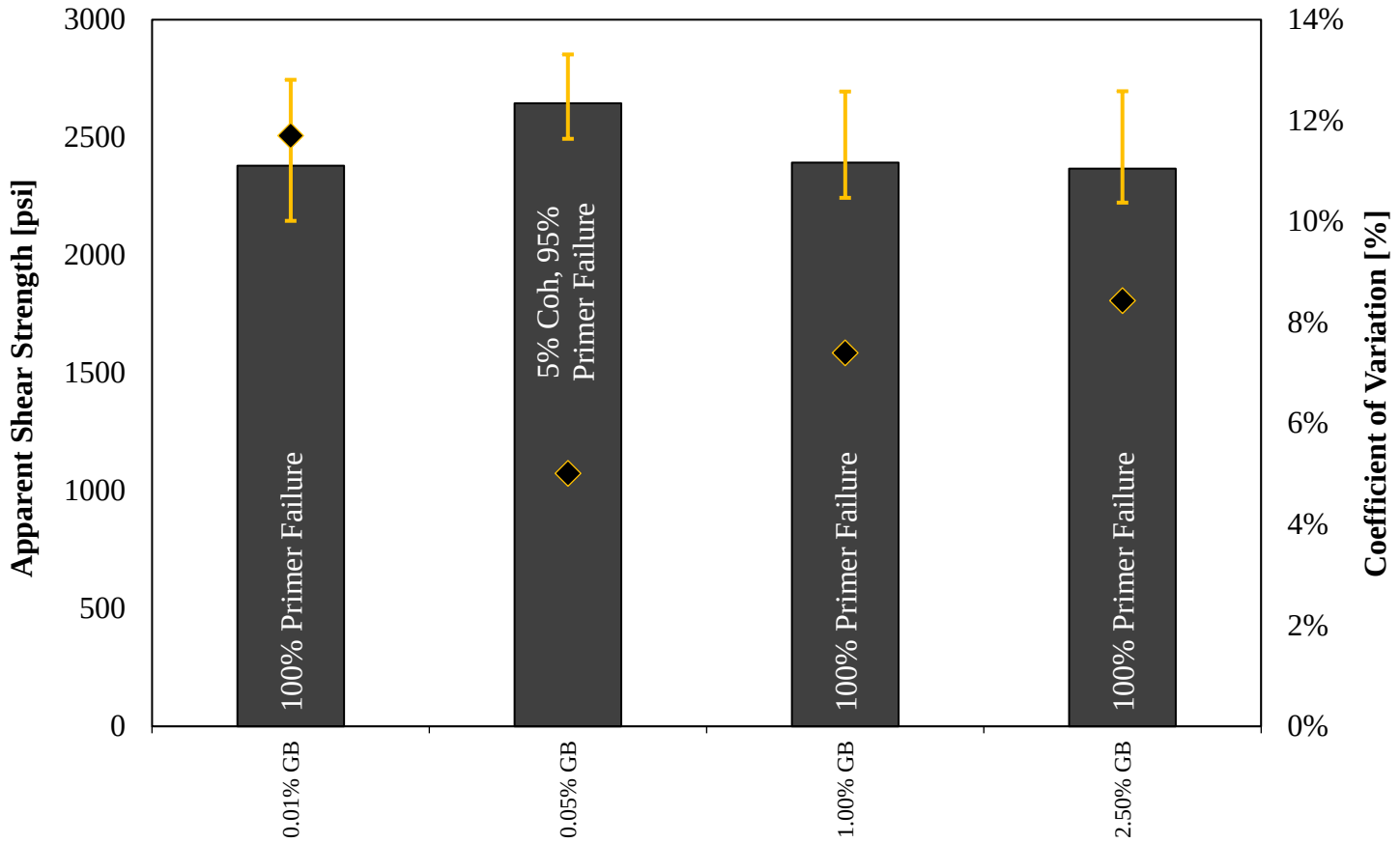


Configuration	Average Bondline Thickness [in]	CoV	Maximum Thickness [in]	Minimum Thickness [in]	Average Primer Layer Thickness [in]	CoV	Maximum Thickness [in]	Minimum Thickness [in]
1 Layer	0.009	6%	0.010	0.008	0.00033	11%	0.00030	0.00035
2 Layers	0.010	5%	0.010	0.009	0.00042	11%	0.00035	0.00055
3 Layers	0.010	5%	0.011	0.010	0.00093	24%	0.00060	0.00115

Task 2b – Bondline Control Mechanisms

- The effect of glass bead percentage on single lap shear strength was investigated.
- The amount of adhesive used for each panel was fixed at 23.4g and the amount of 0.01 in glass beads mixed in to the adhesive varied. The configurations were:
  - 0.01% - 0.00234 g
  - 0.05% - 0.01170 g
  - 1.00% - 0.23400 g
  - 2.50% - 0.58500 g





Configuration	Average Bondline Thickness [in]	CoV	Maximum Thickness [in]	Minimum Thickness [in]
0.01% GB	0.010	46%	0.015	0.005
0.05% GB	0.011	28%	0.014	0.006
1.00% GB	0.011	31%	0.016	0.007
2.50% GB	0.012	26%	0.016	0.007

Summary and Conclusions

- Adhesive thermal properties measurement techniques needs to well document as different measurement techniques and reporting values can be significantly different
- Determination of the cure cycle, maximum environmental limits is critical as adhesive and joint material properties degrade when tested above Tg.
- Long term thermal exposure to bonded joints alter the degree of cure characteristics. Effects of thermal exposure on mechanical properties needs to be determined when establishing the joint properties
- Critical factors in the bond process and methodologies to determine the bonding processing limits are critical. Processing limit establishments is required to qualify the bond process.

Thank You!

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