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Ceramic Matrix Composite Materials Guidelines for Aircraft Design

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May 23, 2024

JAMS
Joint Centers of Excellence for Advanced Materials

Federal Aviation Administration

CECAM
center of excellence for composites and advanced materials

Partners and Objectives

Principal Investigators: John Tomblin, Matthew Opliger, Rachael Andrulonis

FAA Technical Monitor: Yongzhe Tian

FAA Research Sponsor: Cindy Ashforth

Industry Partners: Axiom Materials (ox/ox prepreg and test panels), AC&A (ox/ox test panels), 3M (ox fiber/fabric), IHI Corporation (SiC/SiC test panels), 20+ steering committee members

Objectives

- Develop a framework for the qualification of CMCs, including guidelines and recommendations for their characterization, testing, design and utilization.
- Develop and execute a test plan to evaluate the durability and long-term safety of CMCs.
- Transition the CMC test data and guidelines generated in this program into shared databases, such as CMH-17.
- Coordinate with industry and government organizations, including CMH-17 CMC coordination and working groups and ASTM C28.

Federal Aviation Administration

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AXIOM MATERIALS

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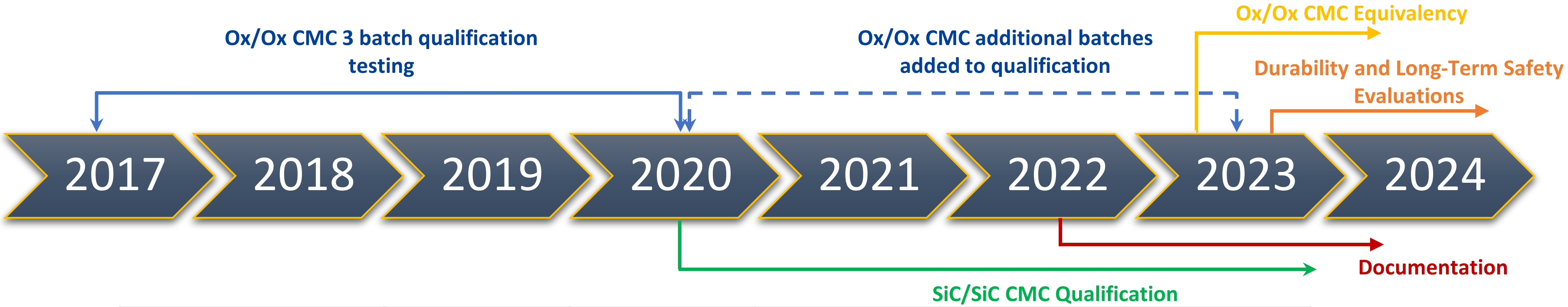
Schedule



Task	Material Specification	Process Specification	Test Plan	Panel Fabrication	Testing
Ox/Ox CMC Qualification	✓	✓	✓	✓	In Progress
SiC/SiC CMC Qualification	✓	✓	✓	In Progress	In Progress
Ox/Ox CMC Equivalency	N/A	N/A	✓	• Received Prepreg • Pending Revisions to the Process Specification • Process Parameter Evaluations In Progress	
Durability and Long-Term Safety Evaluation	N/A	N/A	✓		

Ox/Ox Qualification

Fiber Source:
3M

Weaver:
Textile Products Inc (TPI)

Prepreg Supplier:
Axiom Materials

Panel Fabricator:
AC&A, Axiom

Nextel 610 Fibers
Alumina Fiber, 3K Denier, 5-Harness Satin Weave

AX-7800 Aluminum Silicate Slurry (Water Based)

3 Batches

Autoclave Cure and Sintering

- Documents Generated for Qualification Program:
  - Material Specification
  - Process Specification
  - Test plan – including test matrix with physical, thermal, and mechanical test requirements
- Other Documents for Qualification Program:
  - Material Property Data Report
  - Statistical Analysis Report with Material Allowables

Knowledge gained from this program has aided in the completion of many sections of CMH-17 Volume 5

Ox/Ox Qualification

Material Specification: Complete

Process Specification: Revision Q1 2024

Test Plan: Complete

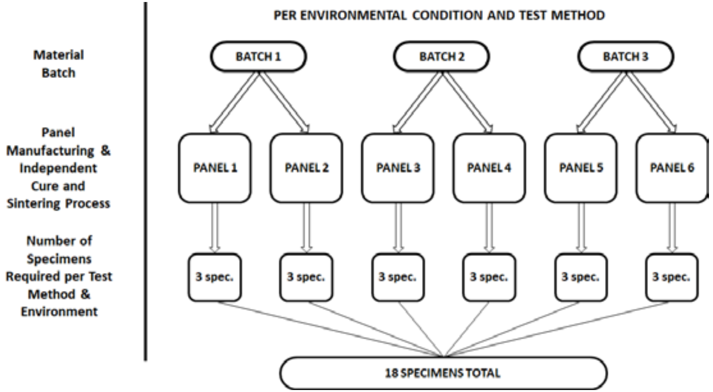
Panel Fabrication: Complete

Testing: Q1 2024

Statistical Analysis: Q2 2024

Test Reports: Q2 2024

Transition Data and Guidelines: Q2 2024

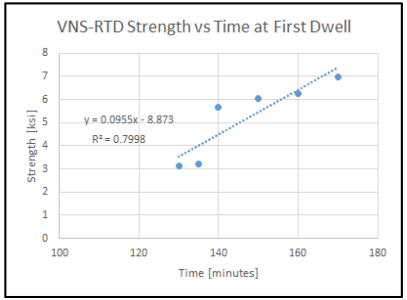


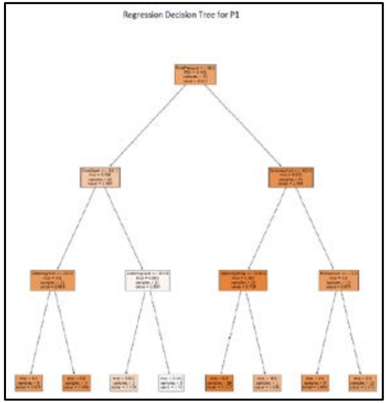
- Due to a high coefficient of variation observed for some properties, process-property data were evaluated and physical property acceptance limits were established – as a result, some data were excluded, requiring new data to be generated.
- Process parameter evaluations were performed and additional evaluations are planned to enable improved quality and reproducibility of test panels without changing material performance.
- More than five batches of prepreg and 100 panels have been produced, and more than 300 physical, 60 thermophysical, and 700 mechanical tests have been performed.

Process-Property Analysis and Acceptance limits

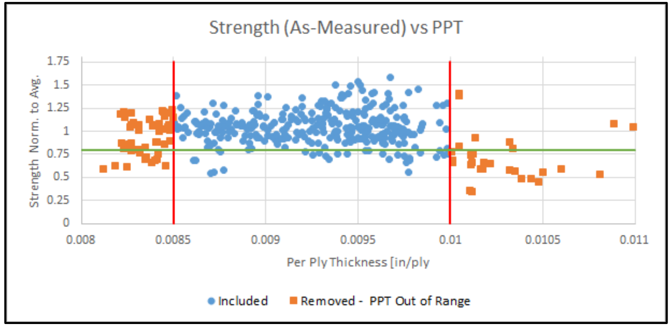
- Had previously performed linear and multivariate regression analysis on the qualification data to better understand process-property relationships to key-in on potential changes to the process specification.

Variables		
Processing	**Physical Testing**	**Mechanical Testing**
Min Vacuum During AC Cure ["Hg]	Density [g/cm3]	All Test Types (e.g., WT, FT, WC, FC, ILS)
Sintering Temperature [°F]	Porosity [% Vol]	All Properties (i.e., Strength and Modulus)
Sintering Hold Time [minutes]	Fiber Volume [% Vol]	All Test Temperatures (i.e., RTD and ETD)
Time at First Dwell [minutes]	Matrix Volume [% Vol]	
Time at Initiation of Full Pressure to Final Time of First Dwell [minutes]	Per Ply Thickness [in]	



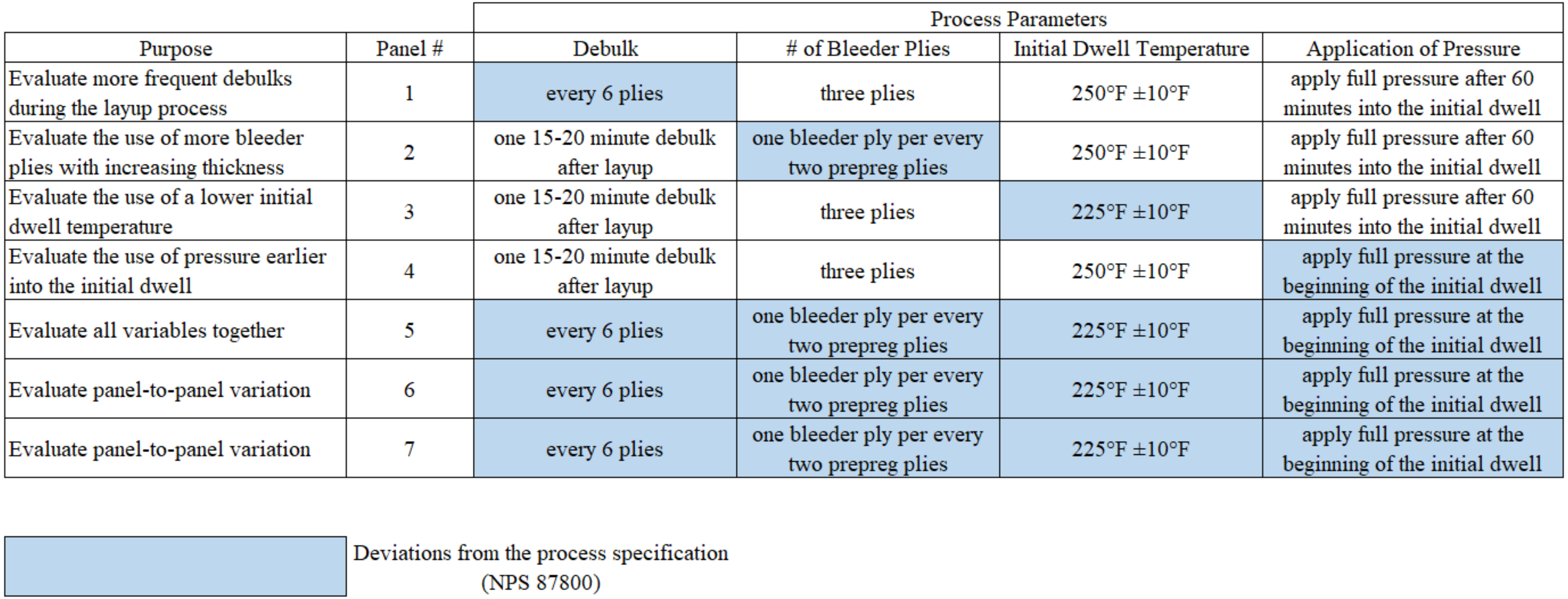


- Had previously performed analysis to determine the appropriate physical property acceptance limits for which all the data was filtered.
  - Porosity Acceptance Limit: ≤ 29%
  - Density Acceptance Limit: 2.6 - 2.8 g/cm^3
  - Per Ply Thickness Acceptance Limit: 0.0085 - 0.0100 in/ply



Process Parameter Evaluations

Process parameter evaluations were performed with a goal of improving quality and reproducibility of test panels without changing material performance, so the resulting changes to the process specification would be deemed only a “minor” change.



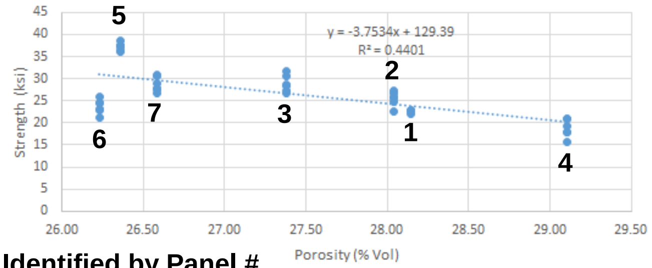
		Process Parameters			
Purpose	Panel #	Debulk	# of Bleeder Plies	Initial Dwell Temperature	Application of Pressure
Evaluate more frequent debulks during the layup process	1	every 6 plies	three plies	250°F ±10°F	apply full pressure after 60 minutes into the initial dwell
Evaluate the use of more bleeder plies with increasing thickness	2	one 15-20 minute debulk after layup	one bleeder ply per every two prepreg plies	250°F ±10°F	apply full pressure after 60 minutes into the initial dwell
Evaluate the use of a lower initial dwell temperature	3	one 15-20 minute debulk after layup	three plies	225°F ±10°F	apply full pressure after 60 minutes into the initial dwell
Evaluate the use of pressure earlier into the initial dwell	4	one 15-20 minute debulk after layup	three plies	250°F ±10°F	apply full pressure at the beginning of the initial dwell
Evaluate all variables together	5	every 6 plies	one bleeder ply per every two prepreg plies	225°F ±10°F	apply full pressure at the beginning of the initial dwell
Evaluate panel-to-panel variation	6	every 6 plies	one bleeder ply per every two prepreg plies	225°F ±10°F	apply full pressure at the beginning of the initial dwell
Evaluate panel-to-panel variation	7	every 6 plies	one bleeder ply per every two prepreg plies	225°F ±10°F	apply full pressure at the beginning of the initial dwell

🟦 Deviations from the process specification (NPS 87800)

Process Parameter Evaluations

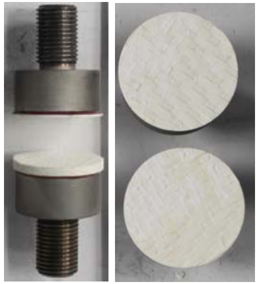
- Density, porosity, and flexural and interlaminar tensile strength were determined from each panel. Panels incorporating all process parameter changes performed the best but significant variation still exists.
- After conducting data and failure analyses, poor adhesion of plies at the interface where the last debulk was performed appears to be one possible reason for the lower strengths.
- Significant matrix bleed was observed after debulking, which may be an indication of too aggressive debulks.
- Additional changes will be made and a new panel will be fabricated:
    - Edge breathing dams to reduce matrix bleed
    - Reduce frequency of vacuum debulks and spread them more evenly across the layup
    - Measurements will be taken to determine the amount of matrix transfer to the poly backing – concerned debulks cause more matrix transfer to the poly

Flexure Strength vs Porosity

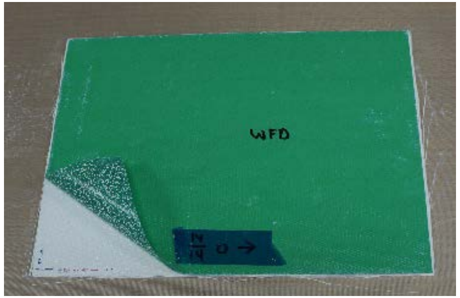


Identified by Panel #

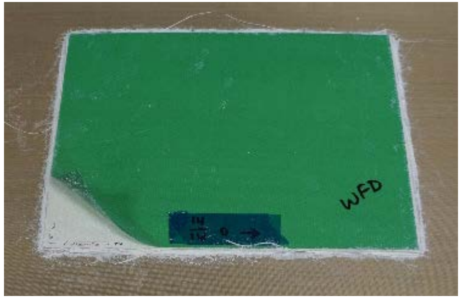
ILT Failure Images



Before Debulk (Panel 2)



Debulks Complete (Panel 1)



Statistics – Material Allowables Generation

- Material allowables were calculated in accordance with CMH-17 Volume 1 Chapter 8. Initial calculations were performed grouping the data by batch, which is typical for composites.
- For structured datasets, ANOVA must be used, which results in very low allowables because ANOVA doesn't handle less than 5 groups well.

- Greater panel-to-panel variability has been observed than batch-to-batch variability. In this case, CMH-17 allows for the substitution of multiple panels within a batch for multiple batches when calculating basis values using the ANOVA method.
- Grouping by panel (6 groups) results in more reasonable material allowables when using ANOVA.

Grouping by Material Batch

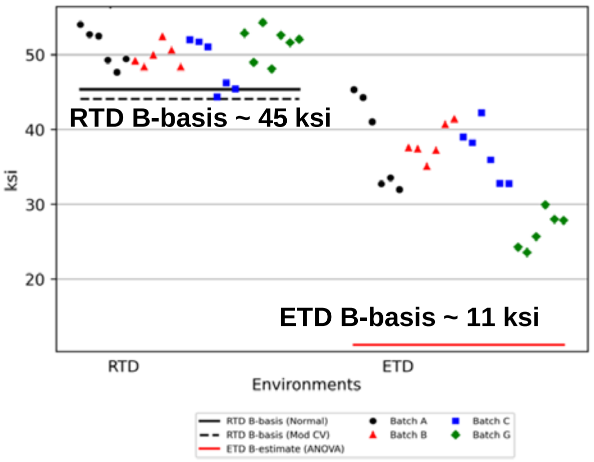


Figure 4-5: Batch plot for WT normalized strength

Grouping by Material Panel

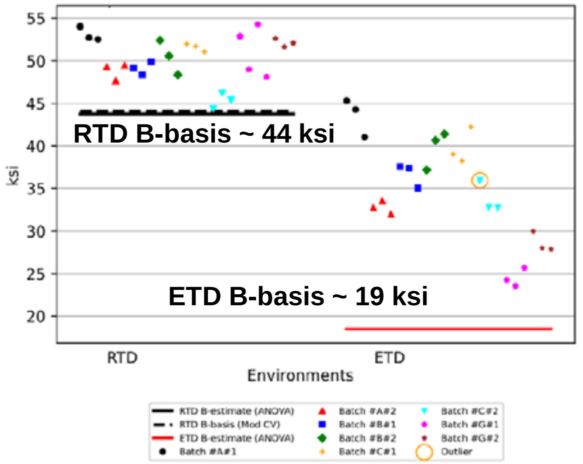


Figure 4-5: Batch plot for WT normalized strength

Ox/Ox Equivalency

Fiber Source:
3M

Weaver:
Textile Products Inc (TPI)

Prepreg Supplier:
Axiom Materials

Panel Fabricator:
NIAR

Nextel 610 Fibers
Alumina Fiber, 3K Denier, 5-Harness Satin Weave

AX-7800 Aluminum Silicate Slurry (Water Based)

Autoclave Cure and Sintering

1 Batch

- Documents Generated for Equivalency Program:
  - Test plan – including test matrix with physical, thermal, and mechanical test requirements
- Other Documents for Equivalency Program:
  - Material Property Data Report
  - Equivalency Statistical Analysis Report

Test Matrix:
Complete

Test Plan:
Complete

Manufacture of Prepreg:
Complete

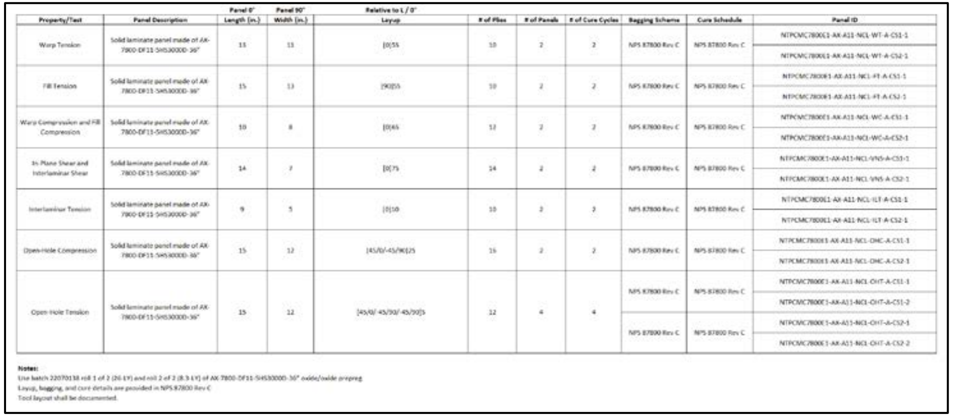
Panel Fabrication:
Q1 2024

Testing:
To Begin Q2 of 2024

Panel fabrication is pending outcome of the equivalency

Ox/Ox Equivalency

- Equivalency panel fabrication work details have been written and will be update after the process specification is revised.
- Equivalency test matrices include physical, thermophysical, and mechanical tests.
  - Physical: NDE, Per Ply Thickness, Fiber and Matrix Volume, Bulk Density, and Open Porosity
  - Thermophysical: Specific Heat and Thermal Diffusivity, Conductivity, and Expansion in X, Y, an Z Directions
  - Mechanical:
    - Tension: In-Plane (RTD and ETD), Interlaminar, and Open-Hole (RTD and ETD)
    - Compression: In-Plane (RTD and ETD) and Open-Hole
    - Shear: In-Plane and Interlaminar (RTD and ETD)



		Panel 0°	Panel 90°	Relative to L / 0°						
Property/Test	Panel Description	Length (in.)	Width (in.)	Layup	# of Plies	# of Panels	# of Cure Cycles	Bagging Scheme	Cure Schedule	Panel ID
Warp Tension	Solid laminate panel made of AX-7800-DF11-5HS3000D-36"	15	13	[0]5S	10	2	2	NPS 87800 Rev C	NPS 87800 Rev C	NTPCMC7800E1-AX-A11-NCL-WT-A-CS1-1 NTPCMC7800E1-AX-A11-NCL-WT-A-CS2-1
Fill Tension	Solid laminate panel made of AX-7800-DF11-5HS3000D-36"	15	13	[90]5S	10	2	2	NPS 87800 Rev C	NPS 87800 Rev C	NTPCMC7800E1-AX-A11-NCL-FT-A-CS1-1 NTPCMC7800E1-AX-A11-NCL-FT-A-CS2-1
Warp Compression and Fill Compression	Solid laminate panel made of AX-7800-DF11-5HS3000D-36"	10	8	[0]6S	12	2	2	NPS 87800 Rev C	NPS 87800 Rev C	NTPCMC7800E1-AX-A11-NCL-WC-A-CS1-1 NTPCMC7800E1-AX-A11-NCL-WC-A-CS2-1
In-Plane Shear and Interlaminar Shear	Solid laminate panel made of AX-7800-DF11-5HS3000D-36"	14	7	[0]7S	14	2	2	NPS 87800 Rev C	NPS 87800 Rev C	NTPCMC7800E1-AX-A11-NCL-VN5-A-CS1-1 NTPCMC7800E1-AX-A11-NCL-VN5-A-CS2-1
Interlaminar Tension	Solid laminate panel made of AX-7800-DF11-5HS3000D-36"	9	5	[0]10	10	2	2	NPS 87800 Rev C	NPS 87800 Rev C	NTPCMC7800E1-AX-A11-NCL-ILT-A-CS1-1 NTPCMC7800E1-AX-A11-NCL-ILT-A-CS2-1
Open-Hole Compression	Solid laminate panel made of AX-7800-DF11-5HS3000D-36"	15	12	[45/0/-45/90]2S	16	2	2	NPS 87800 Rev C	NPS 87800 Rev C	NTPCMC7800E1-AX-A11-NCL-OHC-A-CS1-1 NTPCMC7800E1-AX-A11-NCL-OHC-A-CS2-1
Open-Hole Tension	Solid laminate panel made of AX-7800-DF11-5HS3000D-36"	15	12	[45/0/-45/90/-45/90]S	12	4	4	NPS 87800 Rev C NPS 87800 Rev C	NPS 87800 Rev C NPS 87800 Rev C	NTPCMC7800E1-AX-A11-NCL-OHT-A-CS1-1 NTPCMC7800E1-AX-A11-NCL-OHT-A-CS1-2 NTPCMC7800E1-AX-A11-NCL-OHT-A-CS2-1 NTPCMC7800E1-AX-A11-NCL-OHT-A-CS2-2

Notes:
Use batch 22070138 roll 1 of 2 (26 LY) and roll 2 of 2 (8.3 LY) of AX 7800-DF11-5HS3000D-36" oxide/oxide prepreg
Layup, bagging, and cure details are provided in NPS 87800 Rev C
Tool layout shall be documented.

Durability and Long-Term Safety

Fiber Source:
3M

Weaver:
Textile Products Inc (TPI)

Prepreg Supplier:
Axiom Materials

Panel Fabricator:
NIAR

Nextel 610 Fibers
Alumina Fiber, 3K Denier, 5-Harness Satin Weave

AX-7800 Aluminum Silicate Slurry (Water Based)

3 Batches

Autoclave Cure and Sintering

- Documents Generated for Durability and Long-Term Safety Program:
  - Test plan – including test matrix with mechanical fatigue, long term thermal exposure, and high temperature creep test requirements

Test Matrix:
Complete

Test Plan:
Complete

Manufacture of Prepreg:
Complete

Panel Fabrication:
Q2 2024 to Q3 2024

Testing:
To Begin Q2 2024

Panel fabrication is pending outcome of the equivalency

Durability and Long-Term Safety

- Durability and long-term safety will be evaluated through mechanical fatigue, long-term thermal exposure, and creep testing.
- An initial set of mechanical fatigue evaluations will be performed to determine the final mechanical fatigue test matrix.

Table 1 Fatigue scoping trial test matrix with notional coupon counts

		Number of Batches x No of Panels x No of Specimens (see Note 1)				
		Projected Coupon Counts for Scoping Tests				
Layup	Test Type	Inclusion in Test Plan (see Note 2)	Appropriate R-Value (see Note 3)	Fatigue Frequency (see Note 4)	Stress Level Targets (see Note 5)	Elevated Temperature (see Note 6)
$[45/0/-45/90/-45/90]_S$	Unnotched Tension-Tension			1x1x9	1x1x6	1x1x3
$[45/0/-45/90/-45/90]_S$	Notched Tension-Tension			1x1x9	1x1x6	
$[45/0/-45/90/-45/90]_S$	Notched Tension-Compression			1x1x9	1x1x6	
$[0]_{7S}$	Interlaminar Shear (Double Notch Shear)	1x1x6	1x1x18		1x1x6	1x1x3
$[0]_{28}$	Interlaminar Shear (Short Beam Strength)	1x1x6			1x1x6	
$[0]_{10}$	Interlaminar Tension (Flatwise Tension)	1x1x3		1x1x9	1x1x6	
$[45/0/-45/90/-45/90]_S$	Fatigue After Impact Tension-Tension				1x1x6	
$[45/0/-45/90/-45/90]_S$	Fatigue After Impact Tension-Compression				1x1x6	

Notional Fatigue Test Matrix

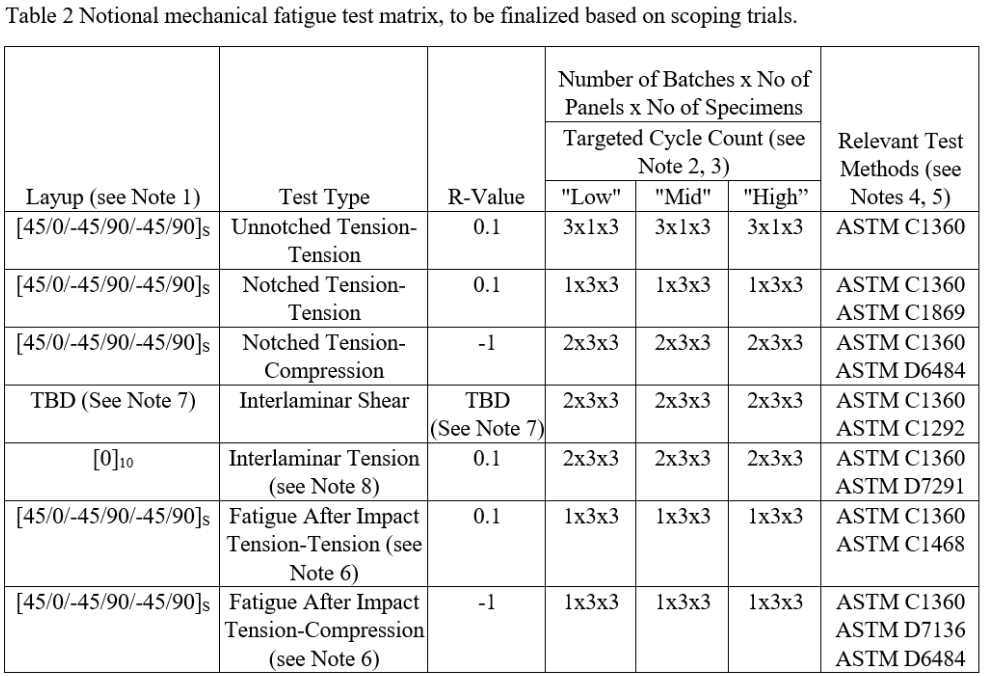


Table 2 Notional mechanical fatigue test matrix, to be finalized based on scoping trials.

Layup (see Note 1)	Test Type	R-Value	Number of Batches x No of Panels x No of Specimens			Relevant Test Methods (see Notes 4, 5)
			Targeted Cycle Count (see Note 2, 3)			
			"Low"	"Mid"	"High"	
$[45/0/-45/90/-45/90]_S$	Unnotched Tension-Tension	0.1	3x1x3	3x1x3	3x1x3	ASTM C1360
$[45/0/-45/90/-45/90]_S$	Notched Tension-Tension	0.1	1x3x3	1x3x3	1x3x3	ASTM C1360 ASTM C1869
$[45/0/-45/90/-45/90]_S$	Notched Tension-Compression	-1	2x3x3	2x3x3	2x3x3	ASTM C1360 ASTM D6484
TBD (See Note 7)	Interlaminar Shear	TBD (See Note 7)	2x3x3	2x3x3	2x3x3	ASTM C1360 ASTM C1292
$[0]_{10}$	Interlaminar Tension (see Note 8)	0.1	2x3x3	2x3x3	2x3x3	ASTM C1360 ASTM D7291
$[45/0/-45/90/-45/90]_S$	Fatigue After Impact Tension-Tension (see Note 6)	0.1	1x3x3	1x3x3	1x3x3	ASTM C1360 ASTM C1468
$[45/0/-45/90/-45/90]_S$	Fatigue After Impact Tension-Compression (see Note 6)	-1	1x3x3	1x3x3	1x3x3	ASTM C1360 ASTM D7136 ASTM D6484

- The target for “low” cycle fatigue is on the order of 1 x 10^4 to 5 x 10^4 cycles.
- The target for “mid” cycle fatigue is on the order of 5 x 10^4 to 2 x 10^5 cycles.
- The target for “high” cycle fatigue is on the order 2 x 10^5 to 1 x 10^6 cycles.
- Specimens which do not fail will be run for at least 10^6 cycles (runout), and residual strength tested.
- Stress levels to target low, mid, and high cycle fatigue stress will be identified during the scoping trials and better defined ranges will be established for low, mid, and high cycle failures.

Thermal Exposure Test Matrix

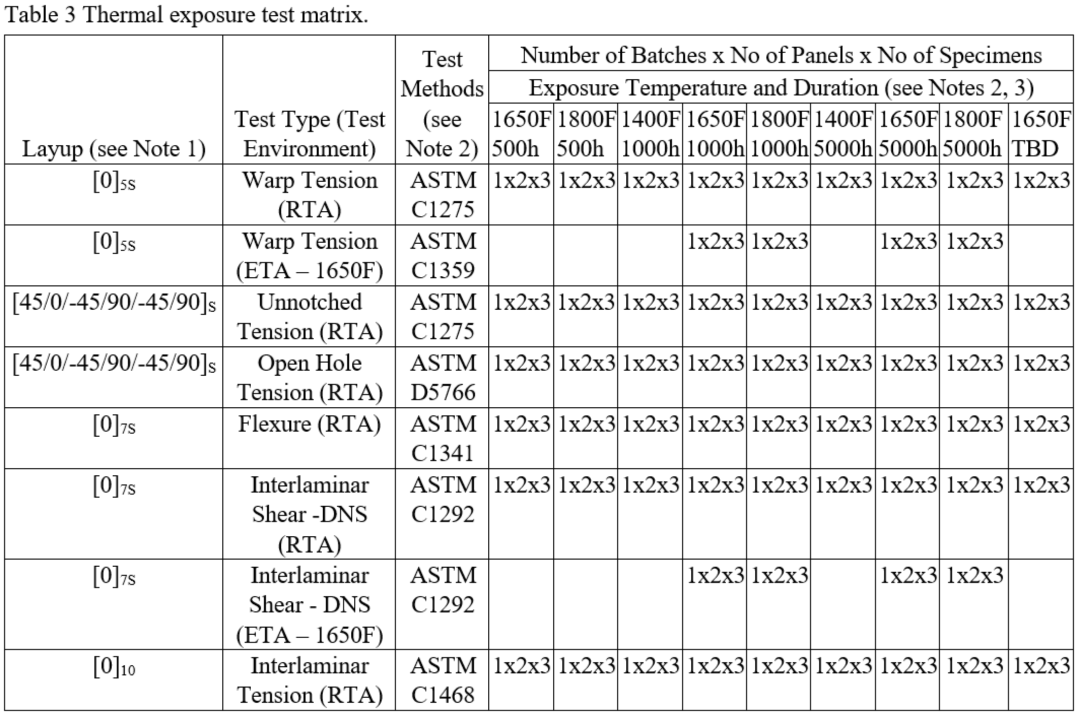


Table 3 Thermal exposure test matrix.

Layup (see Note 1)	Test Type (Test Environment)	Test Methods (see Note 2)	Number of Batches x No of Panels x No of Specimens								
			Exposure Temperature and Duration (see Notes 2, 3)								
			1650F 500h	1800F 500h	1400F 1000h	1650F 1000h	1800F 1000h	1400F 5000h	1650F 5000h	1800F 5000h	1650F TBD
$[0]_{5S}$	Warp Tension (RTA)	ASTM C1275	1x2x3	1x2x3	1x2x3	1x2x3	1x2x3	1x2x3	1x2x3	1x2x3	1x2x3
$[0]_{5S}$	Warp Tension (ETA – 1650F)	ASTM C1359				1x2x3	1x2x3		1x2x3	1x2x3	
$[45/0/-45/90/-45/90]_S$	Unnotched Tension (RTA)	ASTM C1275	1x2x3	1x2x3	1x2x3	1x2x3	1x2x3	1x2x3	1x2x3	1x2x3	1x2x3
$[45/0/-45/90/-45/90]_S$	Open Hole Tension (RTA)	ASTM D5766	1x2x3	1x2x3	1x2x3	1x2x3	1x2x3	1x2x3	1x2x3	1x2x3	1x2x3
$[0]_{7S}$	Flexure (RTA)	ASTM C1341	1x2x3	1x2x3	1x2x3	1x2x3	1x2x3	1x2x3	1x2x3	1x2x3	1x2x3
$[0]_{7S}$	Interlaminar Shear -DNS (RTA)	ASTM C1292	1x2x3	1x2x3	1x2x3	1x2x3	1x2x3	1x2x3	1x2x3	1x2x3	1x2x3
$[0]_{7S}$	Interlaminar Shear - DNS (ETA – 1650F)	ASTM C1292				1x2x3	1x2x3		1x2x3	1x2x3	
$[0]_{10}$	Interlaminar Tension (RTA)	ASTM C1468	1x2x3	1x2x3	1x2x3	1x2x3	1x2x3	1x2x3	1x2x3	1x2x3	1x2x3

- Mechanical tests will be performed statically for all test types.
- TBD: will notionally be tested after 10,000 hours, but specimens could be exposed for a longer period of time if the need arises.
- The weight of each specimen will be measured before and after exposure.
- Photographs of each failed specimen will be taken, and the failure mode will be recorded. A subset of coupons for each test type may have fracture surfaces analyzed.

High Temperature Creep Test Matrix

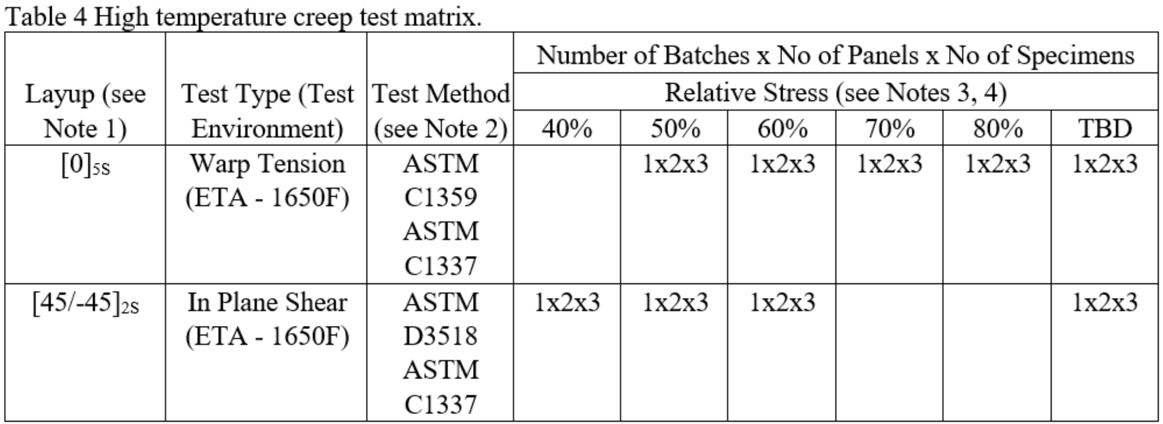


Table 4 High temperature creep test matrix.

Layup (see Note 1)	Test Type (Test Environment)	Test Method (see Note 2)	Number of Batches x No of Panels x No of Specimens					
			Relative Stress (see Notes 3, 4)					
			40%	50%	60%	70%	80%	TBD
$[0]_{5S}$	Warp Tension (ETA - 1650F)	ASTM C1359 ASTM C1337		1x2x3	1x2x3	1x2x3	1x2x3	1x2x3
$[45/-45]_{2S}$	In Plane Shear (ETA - 1650F)	ASTM D3518 ASTM C1337	1x2x3	1x2x3	1x2x3			1x2x3

- Testing will be conducted at 1650°F.
- Relative applied stress is defined as a percentage of either the ultimate stress or peak stress, as appropriate, as determined by static testing on the same batch of material.
- One set of coupons for each test type will be reserved for either testing at an additional stress level or testing at an identical stress level but a higher or lower temperature. This will be determined based on preliminary creep testing results.

SiC/SiC Qualification

Fiber Source:
Ube Industries Ltd.

Tyranno ZMI SiC Fiber

Fiber Coating Source:
IHI Corporation

Boron Nitride Coating

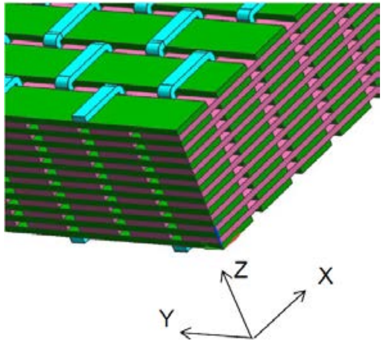
Weaver:
Shikibo Ltd.

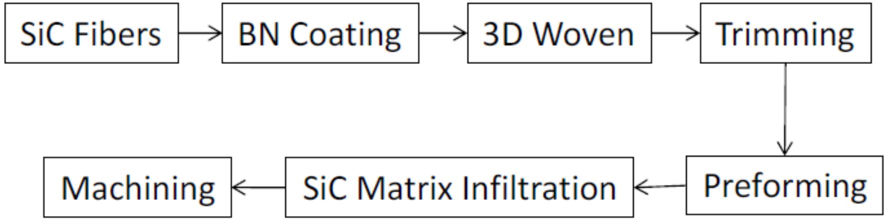
3D Orthogonal Woven Preform

Panel Fabricator:
IHI Corporation

Chemical Vapor Infiltration (CVI) and Polymer Infiltration and Pyrolysis (PIP) Hybrid Process

3 Batches




SiC Fibers → BN Coating → 3D Woven → Trimming → Preforming → SiC Matrix Infiltration → Machining

Fabrication and machining performed by IHI, thermophysical testing performed by JUTEM, and mechanical testing performed by Kiguchi Technics in Japan.

Watanabe, F., Manabe, T., “Engine Testing for the Demonstration of a 3D-Woven Based Ceramic Matrix Composite Turbine Vane Design Concept,” ASME Turbo Expo 2018: Turbomachinery Technical Conference and Exposition, Oslo, Norway, June 11-15, 2018

SiC/SiC Qualification

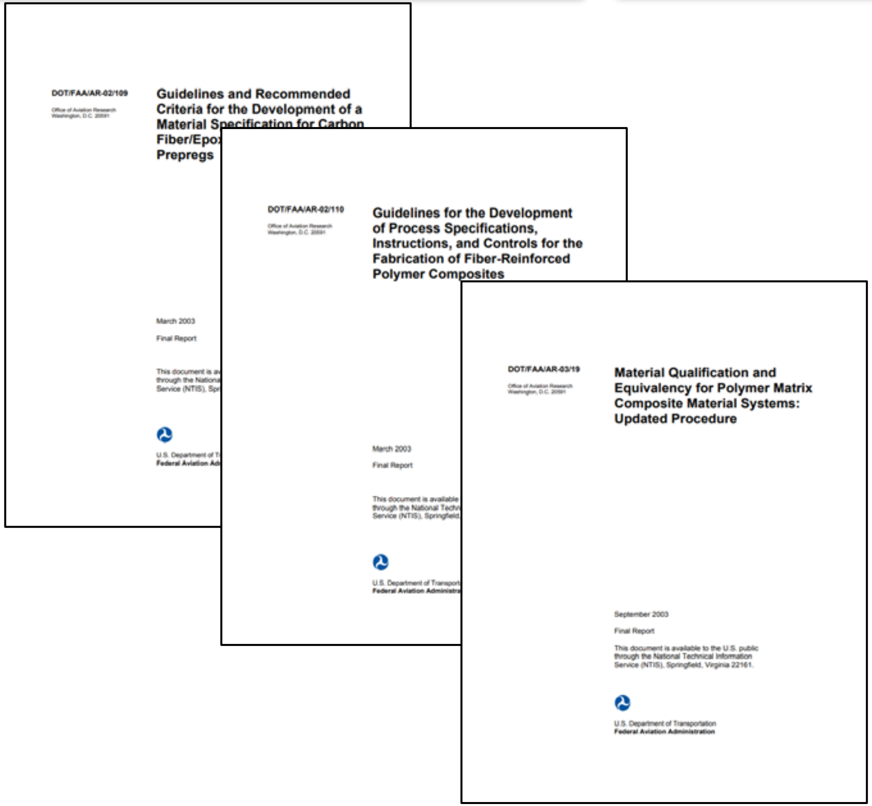
- Documents Generated for Qualification Program:
  - Material Specification (Restricted Distribution)
  - Process Specification (Restricted Distribution)
  - Test plan – including test matrix with physical, thermal, and mechanical test requirements (Restricted Distribution)
- Other Documents for Qualification Program:
  - Material Property Data Report (Restricted Distribution)
  - Statistical Analysis Report with Material Allowables (Restricted Distribution)

Material Specification: Complete → **Process Specification: Complete** → **Test Plan: Complete** → **Panel Fabrication: In Progress** → **Testing: In Progress**

Testing began in Q4 2022 with many RTD and ETD tests completed.

Documentation

- **Document framework development** thus far through reports similar to DOT/FAA/AR-02/109 and 110, and DOT/FAA/AR-03/19
  - Necessary to properly document everything learned during framework development
- **Develop standard guides** supporting ox/ox CMC testing for future test method standardization
  - Guides for
    - Specimen gripping
    - Specimen machining
    - Strain instrumentation
    - Heating and temperature distribution
  - Guidance already developed by NIAR as part of framework development will be supplemented by studies evaluating methodology precision and acceptability
  - Publish in ASTM standard guides or in CMH-17



DOT/FAA/AR-02/109
Office of Aviation Research
Washington, D.C. 20591

Guidelines and Recommended Criteria for the Development of a Material Specification for Carbon Fiber/Epox Prepregs

March 2003

Final Report

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Office of Aviation Research
Washington, D.C. 20591

Guidelines for the Development of Process Specifications, Instructions, and Controls for the Fabrication of Fiber-Reinforced Polymer Composites

March 2003

Final Report

This document is available
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U.S. Department of Transport
Federal Aviation Administra

DOT/FAA/AR-03/19
Office of Aviation Research
Washington, D.C. 20591

Material Qualification and Equivalency for Polymer Matrix Composite Material Systems: Updated Procedure

September 2003

Final Report

This document is available to the U.S. public through the National Technical Information Service (NTIS), Springfield, Virginia 22161.

U.S. Department of Transportation
Federal Aviation Administration

Questions?