Ceramic Matrix Composite Materials Guidelines for Aircraft Design

Federal Aviation Administration

Presented by:

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NIAR Wichita State University National Institute for Aviation Research

JAMS

Joint Centers of Excellence for Advanced Materials

A Center of Excellence AMTAS Advanced Materials in Transport Aircraft Structures

CECAM center of excellence for composites and advanced materials

JAMS Technical Review

September 29, 2021

Introduction

- **Project Title:** Ceramic Matrix Composite Materials Guidelines for Aircraft Design
- **Principal Investigators:** John Tomblin, Matt Opliger, Rachael Andrulonis
- **FAA Technical Monitor**: Curtis Davies
- **Other FAA Personnel:** 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
- **Source of matching contribution for the current award:** Kansas Aviation and Research Technology (KART)

Motivation and Key Issues

- Expanded use of Ceramic Matrix Composites (CMCs) in gas turbine engines and hypersonic applications
- CMCs require their own set of rules separate from more established PMCs
- No “fully approved” data in CMH-17
- Similar complexity to PMCs in terms of anisotropy, fiber architecture, high strength/stiffness fibers, and production process sensitivity and variability, they are also different in many ways such as:
    - Composite constituents
    - Degradation, damage, and failure mechanisms
    - High temperature life predictions
    - High temperature joining challenges
    - NDE challenges
    - Repairability

Objective and Scope

- 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.

Approach

- Generate Qualification Documents for CMCs and Perform Material Qualification
  - Material and Process Specifications
  - Test Plan
  - Statistical Analysis Report with B-Basis Allowables
- Generate Equivalency Documents for CMCs and Perform Material Equivalency
  - Test Plan
  - Equivalency Analysis Report
- Evaluate Durability and Long Term Safety of CMCs
  - Generate Test Plan
  - Perform fatigue, long term thermal exposure, and creep testing

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)

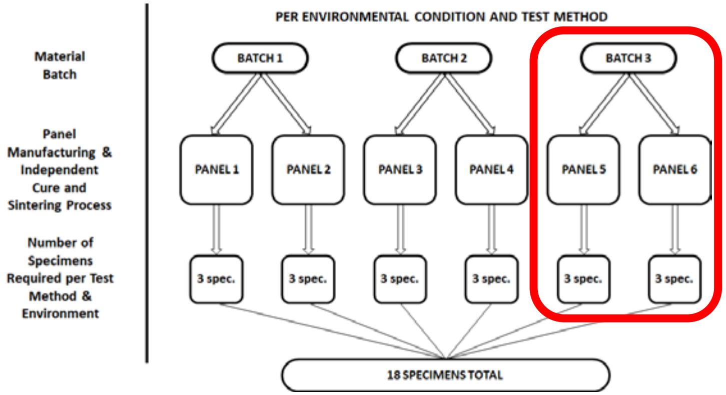
3 Batches

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

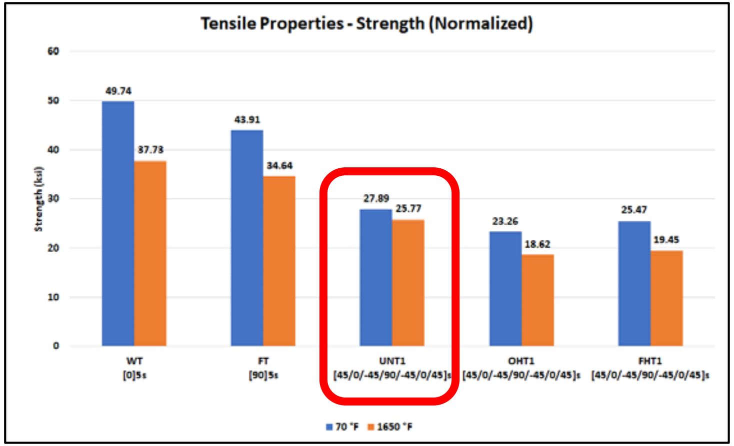
Ox/Ox Qualification

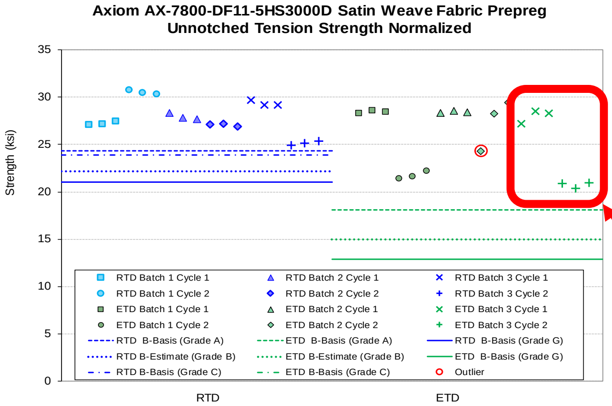
Material Specification: Complete → Process Specification: Complete → Test Plan: Complete → Panel Fabrication: Complete → **Testing: In progress, ETC Q4 2021** → **Statistical Analysis: In progress, ETC Q4 2021** → Test Reports: ETC Q1 2022 → Transition Data and Guidelines: ETC Q1 2022



Had quality issues with panels fabricated by AC&A from batch 3. Replacement panels were fabricated by Axiom Materials. Testing resumed once replacement panels were received earlier this year (testing was previously completed batch 1 and 2).

Ox/Ox Qualification





Data from batch 3 panels with quality issues.

Example of Statistical Approaches Being Evaluated

Unnotched Tension Strength Basis Values and Statistics					
		Normalized		As-measured	
	Env	RTD	ETD	RTD	ETD
Basis Statistics	Mean	27.891	25.767	27.690	25.675
	Stdev	1.793	3.481	2.067	3.974
	CV	6.427	13.510	7.465	15.478
	Mod CV	7.214	13.510	7.733	15.478
	Min	24.932	20.343	24.969	19.889
	Max	30.815	29.449	31.459	30.197
	No. Batches	3	3	3	3
	No. Panels	6	6	6	6
	No. Spec.	18	18	18	18
Grade A	Basis Values and Estimates (CMH17 by Batch)				
	B-Basis	24.352	18.111	23.609	17.878
	A-Estimate	21.844	11.694	20.717	10.646
	Method	Normal	Non-Parametric	Normal	Non-Parametric
Grade B	Basis Value Estimates (ANOVA By Panel)				
	B-Estimate	22.185	14.994	21.115	13.210
	A-Estimate	18.284	7.617	16.620	4.681
Grade C	Modified CV Basis Values and Estimates				
	B-Basis	23.919	NA	23.463	NA
	A-Estimate	21.109		20.473	
	Method	Normal		Normal	
Grade G	Generic Basis Values and Estimates				
	B-Basis	21.050	12.921	19.809	10.772
	A-Estimate	17.966	7.129	16.255	4.053

Various statistical approaches will be considered for determining equivalency and guidance will be developed.

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)
1 Batch

Autoclave Cure and Sintering

- Documents Generated for Equivalency Program:
  - Test plan – including test matrix with physical, thermal, and mechanical test requirements

Test Matrix: Complete → **Test Plan:** In progress, ETC Q4 2021 → **Manufacture of Prepreg:** ETC Q4 2021 → **Panel Fabrication:** ETC Q1 2022 → **Testing:** To Begin Q2 of 2022

Ox/Ox Equivalency

Equivalency Test Matrices

Property	Test Method	Min Replicates per Panel
NDI by Ultrasonic Through Transmission (C-Scan), Thermography, or Radiography (CT Scan)		1
Cured/Sintered Ply Thickness	ASTM D3171 (Method II)	All data from mechanical test specimens
Fiber Volume, % by Volume	ASTM D3171 (Method II)	3
Matrix Volume, % by Volume	ASTM D3171 (Method II)	3
Cured/Sintered Composite Density	ASTM C373	3
Void/Porous Content	ASTM C373	3
Specific Heat	ASTM E1269	3 (Total)
Thermal Conductivity (Diffusivity), Measured in x, y, and z directions	ASTM E1461	3
Thermal Expansion, Measured in x, y, and z directions	ASTM E228	3

Layup	Test Type and Direction	Property	Test Method	Number of Batches x No. of Panels x No. of Specimens	
				Test Temperature	
				RTD	ETD
$[0]_{5S}$	Warp Tension	Strength, Modulus, and Poisson's Ratio	ASTM C1275 (RTD) ASTM C1359 (ETD)	1x2x4	-
$[90]_{5S}$	Fill Tension	Strength and Modulus	ASTM C1275 (RTD) ASTM C1359 (ETD)	1x2x4	1x2x4
$[0]_{6S}$	Warp Compression	Strength and Modulus	ASTM C1358	1x2x4	1x2x4
$[90]_{6S}$	Fill Compression	Strength and Modulus	ASTM C1358	1x2x4	-
$[0]_{7S}$	In-Plane Shear (V-Notch Shear)	Strength and Modulus	ASTM D5379	1x2x4	
$[0]_{7S}$	Interlaminar Shear (Double Notch Shear)	Strength	ASTM C1292 (RTD) ASTM C1425 (ETD)	1x2x4	1x2x4
$[0]_{10}$	Interlaminar Tension (Trans-Thickness / Flatwise Tension)	Strength	ASTM C1468	1x2x4	
$[45/0/-45/90]_{2S}$	Open-Hole Compression	Strength	ASTM D6484	1x2x4	-
$[45/0/-45/90/-45/90]_{S}$	Open-Hole Tension	Strength	ASTM D5766	1x2x4	1x2x4

Ox/Ox Equivalency

Example of Statistical Approaches Being Evaluated

Estimates and Allowables Generated from Qualification Dataset

Unnotched Tension Strength Basis Values and Statistics					
		Normalized		As-measured	
	Env	RTD	ETD	RTD	ETD
Basis Statistics	Mean	27.891	25.767	27.690	25.675
	Stdev	1.793	3.481	2.067	3.974
	CV	6.427	13.510	7.465	15.478
	Mod CV	7.214	13.510	7.733	15.478
	Min	24.932	20.343	24.969	19.889
	Max	30.815	29.449	31.459	30.197
	No. Batches	3	3	3	3
	No. Panels	6	6	6	6
	No. Spec.	18	18	18	18
Grade A	Basis Values and Estimates (CMH17 by Batch)				
	B-Basis	24.352	18.111	23.609	17.878
	A-Estimate	21.844	11.694	20.717	10.646
	Method	Normal	Non-Parametric	Normal	Non-Parametric
Grade B	Basis Value Estimates (ANOVA By Panel)				
	B-Estimate	22.185	14.994	21.115	13.210
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Grade C	Modified CV Basis Values and Estimates				
	B-Basis	23.919	NA	23.463	NA
	A-Estimate	21.109		20.473	
	Method	Normal		Normal	
Grade G	Generic Basis Values and Estimates				
	B-Basis	21.050	12.921	19.809	10.772
	A-Estimate	17.966	7.129	16.255	4.053

Equivalency Criteria Determined from Analysis of Qualification Dataset

Unnotched Tension Strength Equivalency Criteria					
		Normalized		As-measured	
	Env	RTD	ETD	RTD	ETD
Grade A or Grade B	CMH17 Minimum Equivalency Criteria forStrength (n=8, alpha = 5%)				
	Mean	26.674	23.404	26.287	22.976
	Minimum	23.051	16.368	22.109	14.945
Grade C	CMH17 Mod CV Minimum Equivalency Criteria for Strength (n=8, alpha = 5%)				
	Mean	26.525	NA	26.236	NA
	Minimum	22.459		21.909	
Grade G	Generic Equivalency Criteria for Strength (n=8, alpha = 5%)				
	Acceptance Limit for Mean	23.823	18.128	23.004	16.813
	Maximum Sample Standard Deviation	4.185	7.860	4.822	9.118

Various statistical approaches will be considered for determining equivalency and guidance will be developed.

SiC/SiC Qualification

Fiber Source:
Ube Industries Ltd.

Tyranno ZMI SiC Fiber

Fiber Coating Source:
IHI Corporation

Boron Nitride Coating

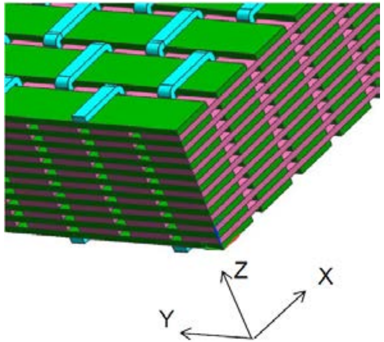
Weaver:
Shikibo Ltd.

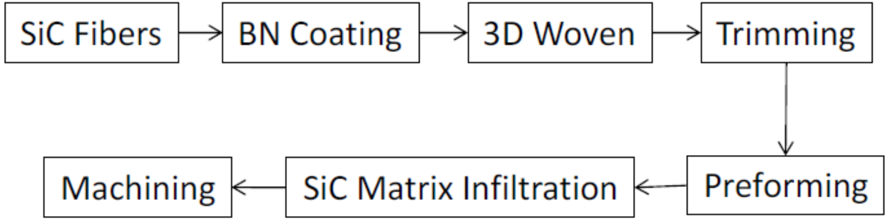
3D Orthogonal Woven Preform

Panel Fabricator:
IHI Corporation

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

3 Batches





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
    - Process Specification
    - Test plan – including test matrix with physical, thermal, and mechanical test requirements

Material Specification: Complete → **Process Specification:** Complete → **Test Plan:** Complete → **Panel Fabrication:** In Progress → **Testing:** To Begin Q2 of 2022

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:** In progress, ETC Q4 2021 → **Manufacture of Prepreg:** Q4 2021 – Q2 2022 → **Panel Fabrication:** ETC Q2 2022 → **Testing:** To Begin Q2 of 2022

Durability and Long Term Safety

Initial Evaluations for Scoping Final Fatigue Test Matrix

Table 1 Fatigue scoping trial test matrix with notional coupon counts

Layup	Test Type	Number of Batches x No of Panels x No of Specimens (see Note 1)				
		Projected Coupon Counts for Scoping Tests				
		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	

Durability and Long Term Safety

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 Targeted Cycle Count (see Note 2, 3) "Low"	 "Mid"	 "High"	Relevant Test Methods (see Notes 4, 5)
$[45/0/-45/90/-45/90]_S$	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 1×10^4 to 5×10^4 cycles.
- The target for “mid” cycle fatigue is on the order of 5×10^4 to 2×10^5 cycles.
- The target for “high” cycle fatigue is on the order 2×10^5 to 1×10^6 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.

Durability and Long Term Safety

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.

Durability and Long Term Safety

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.

Technical Publications

- FAA technical reports have been written with updates provided annually (2019 and 2020 reports submitted, 2021 report in-progress)
- Research has been presented annually at USACA conferences.
- Research has been presented at CMH-17 CMC coordination and working group meetings.