Ceramic Matrix Composite Materials Guidelines for Aircraft Design

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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
  - None destructive evaluation (NDE) challenges
  - Repairability

Partners and Objectives

Principal Investigators: John Tomblin, Matt Opliger, Rachael Andrulonis

FAA Technical Monitor: Ahmet Oztekin

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

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

NIAR Wichita State University National Institute for Aviation Research

AXIOM MATERIALS

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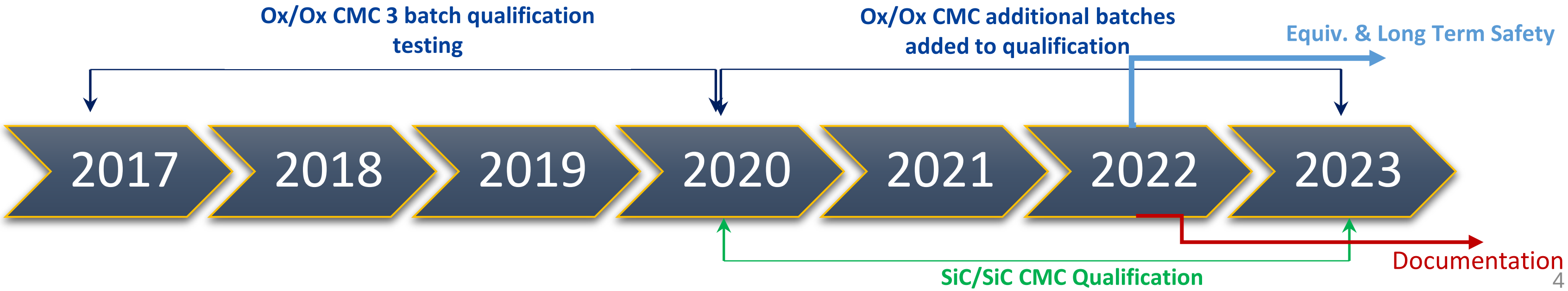
Approach

- **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
- **Documentation**
  - Document framework development and
  - Develop standard guides supporting Ox/Ox CMC testing for future test method standardization



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Ox/Ox Qualification Methodology

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

4 Batches
AX-7800 Aluminum Silicate Slurry (Water Based)

Autoclave Cure and Sintering

Qualification Documents Drafted

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

Lessons Learned

CMH-17 Volume 5:
- Fiber volume fraction
- Specific heat
- Coefficient of thermal expansion
- Thermal conductivity

Ox/Ox Qualification Tasks

Material Specification: Complete

Process Specification: Revision Q2 2023

Test Plan: Complete

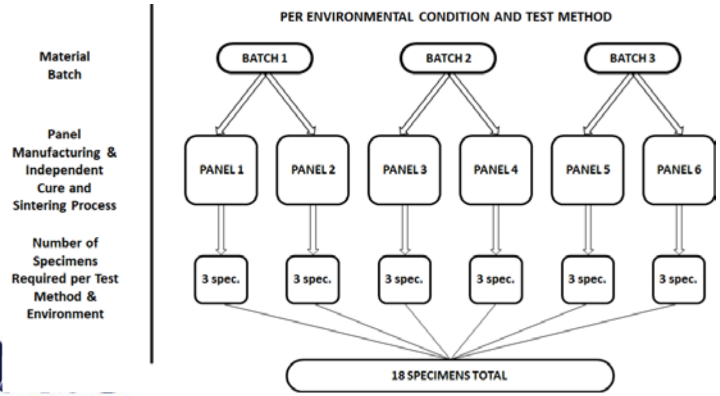
Panel Fabrication: Q2 2023

Testing: Q2 2023

Statistical Analysis: Q3 2023

Test Reports: Q3 2023

Transition Data and Guidelines: Q3 2023



- A fourth batch was later added and panels 7 and 8 were produced.
- A total of 24 specimens were tested per environmental condition and test method.

Ox/Ox Qualification – Properties Tested

Composite Physical Properties
Cured/Sintered Ply Thickness
Fiber Volume
Matrix Volume
Density
Porosity

Composite Thermophysical Properties
Specific Heat
Thermal Conductivity (Diffusivity), Measured in x, y, and z directions
Thermal Expansion, Measured in x, y, and z directions

Lamina Mechanical Properties

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 (RTD Only)	ASTM C1275 (RTD) ASTM C1359 (ETD)	3x2x3	3x2x3
$[90]_{5S}$	Fill Tension	Strength and Modulus	ASTM C1275 (RTD)	3x2x3	3x2x3
$[0]_{6S}$	Warp Compression	Strength and Modulus	ASTM C1358	3x2x3	3x2x3
$[90]_{6S}$	Fill Compression	Strength and Modulus	ASTM C1358	3x2x3	3x2x3
$[45/-45]_{2S}$	In-Plane Shear (+45/-45 Tension)	Strength and Modulus (RTD Only)	ASTM D3518	3x2x3	3x2x3
$[0]_{7S}$	In-Plane Shear (V-Notch Shear)	Strength and Modulus	ASTM D5379	3x2x3	
$[0]_{7S}$	Interlaminar Shear (Double Notch Shear)	Strength	ASTM C1292 (RTD)	3x2x3	3x2x3
$[0]_{28}$	Interlaminar Shear (Short-Beam Strength)	Strength	ASTM D2344	3x2x3	

Laminate and Design Guidance Mechanical Properties

Layup	Test Type and Direction	Property	Test Method	Number of Batches x No. of Panels x No. of Specimens Test Temperature RTD	 ETD
$[0]_{7S}$	Flexure	Strength and Modulus	ASTM C1341	3x2x3	
$[0]_{10}$	Interlaminar Tension (Trans-Thickness / Flatwise Tension)	Strength	C1468	3x2x3	
$[0/90]_{5}$	Interlaminar Tension (Trans-Thickness / Flatwise Tension)	Strength	C1468	1x1x6	
$[0/90]_{14}$	Interlaminar Shear (Short-Beam Strength)	Strength	ASTM D2344	1x1x6	
$[45/0/-45/90/-45/90]_{S}$	Unnotched Tension	Strength and Modulus	ASTM C1275 (RTD)	3x2x3	3x2x3
$[45/0/-45/90/-45/90]_{S}$	Unnotched Compression	Strength and Modulus	ASTM C1358	3x2x3	3x2x3
$[45/0/-45/90]_{2S}$	Open-Hole Compression	Strength	ASTM D6484	3x2x3	3x2x3
$[45/0/-45/90/-45/90]_{S}$	Open-Hole Tension	Strength	ASTM D5766	3x2x3	3x2x3
$[45/0/-45/90/-45/90]_{S}$	Filled-Hole Tension	Strength	ASTM D6742	3x2x3	3x2x3
$[45/0/-45/90/-45/90]_{S}$	Single Shear Bearing	Strength	ASTM D5961 (Procedure C)	3x2x3	
$[45/0/-45/90/-45/90]_{S}$	Double Shear Bearing	Strength	ASTM D5961 (Procedure A)	3x2x3	3x2x3
$[45/0/-45/90/-45/90]_{S}$	Tension After Impact	Strength	ASTM D7136 ASTM D5766	1x2x3	1x2x3

RTD = Room Temperature Dry
ETD = Elevated Temperature Dry (1650F/900C)

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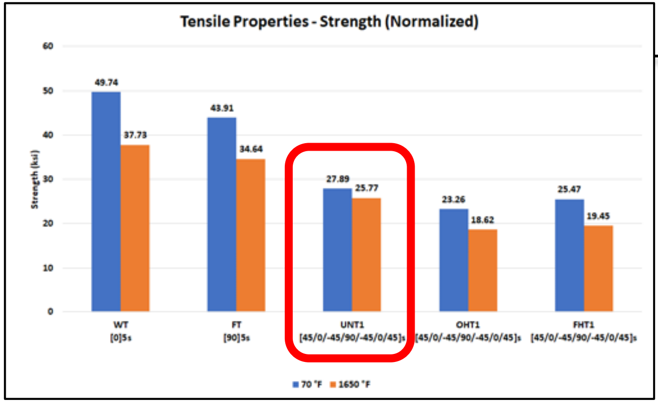
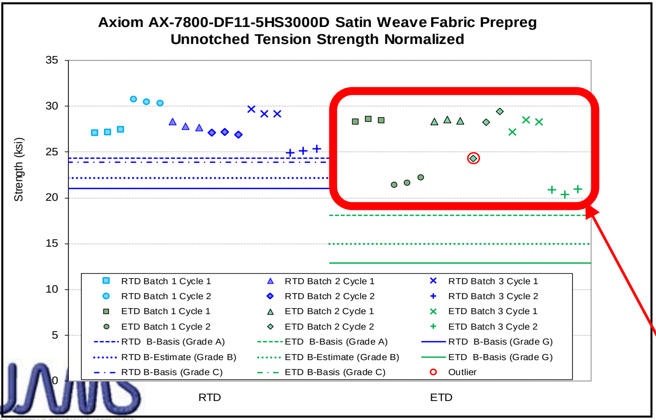
Example of Panel-to-Panel Variability and the Effects on Material Allowables





Example of Statistical Approaches Being Evaluated (First 3 Batches)

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

UNT1 Configuration:
In-Plane Tension
[45/0/-45/90/-45/0/45]s
Layup

- Panel-to-panel variability observed within the same material batch for ETD tests
- Coefficient of variation (CV) for ETD normalized data is 13.51%, resulting in a B-basis material allowable (CMH17 by Batch) that is 30% less than the mean

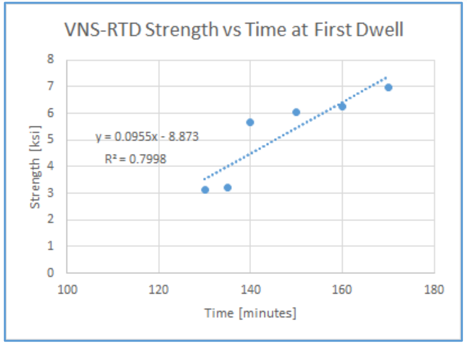
Greater panel-to-panel variability has been observed for other configurations and properties, resulting in very low material allowables

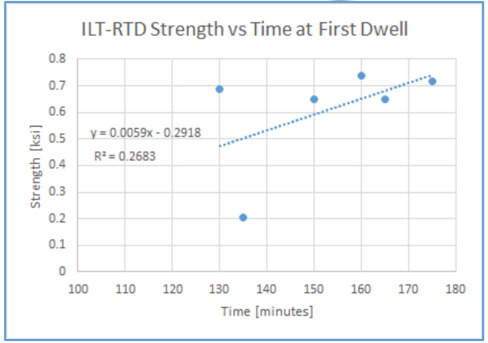
Panel-to-panel variability observed within the same material batch for ETD tests

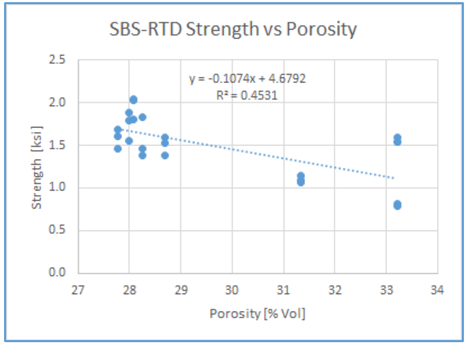
Process-Property Relationships – Linear Regression

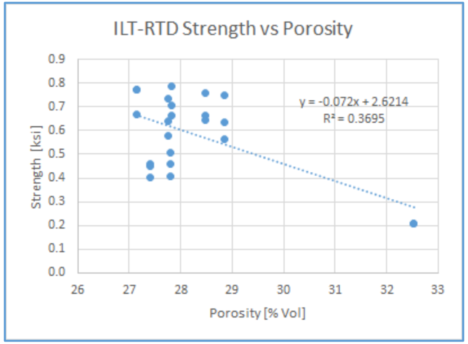
- Single parameter regression analysis was performed to evaluate process-property relationships
- Questions we hoped to answer:
  - Does any single or combination of processing variables correlate with any physical or mechanical variables (test properties)?
  - Does any single or combination of physical variables correlate with any mechanical variables (test properties)?
- Variables analyzed

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]	









Note: All processing data were taken from the cure and sintering runs corresponding to multiple panels. All physical test data except per ply thickness were determined on representative specimens from each panel.

VNS: In-Plane Shear (V-Notch/Iosipescu)

ILT: Interlaminar Tension

SBS: Interlaminar Shear (Short-Beam Strength)

Process-Property Relationships – Linear Regression

Linear Regression R^2 Value Tables for Strength and Modulus at Room Temperature

Strength at RTD Condition					
	R^2 Value				
Test	Density [g/cm^3]	Porosity [% Vol]	Fiber Volume [% Vol]	Matrix Volume [% Vol]	Per Ply Thickness [in]
Warp Tension	0.41	0.39	0.07	0.00	0.23
Fill Tension	0.06	0.12	0.23	0.04	0.17
Unnotched Tension 1	0.23	0.16	0.00	0.03	0.04
Open-Hole Tension 1	0.13	0.15	0.01	0.01	0.08
Filled-Hole Tension 1	0.56	0.49	0.09	0.03	0.20
Warp Compression	0.72	0.70	0.18	0.02	0.43
Fill Compression	0.00	0.00	0.28	0.36	0.10
Unnotched Compression 1	0.82	0.75	0.44	0.01	0.43
Open-Hole Compression 1	0.17	0.38	0.07	0.40	0.01
In-Plane Shear (+/-45 Tension)	0.76	0.60	0.03	0.02	0.32
In-Plane Shear (V-Notch/Iosipescu)	0.06	0.07	0.15	0.52	0.07
Interlaminar Shear (Double-Notch)	0.01	0.02	0.00	0.03	0.00
Interlaminar Shear (Short-Beam)	0.46	0.45	0.29	0.00	0.37
Interlaminar Tension	0.43	0.37	0.13	0.02	0.21
Flexure	0.13	0.16	0.06	0.38	0.47
Single-Shear Bearing	0.73	0.80	0.12	0.08	0.31
Double-Shear Bearing	0.00	0.03	0.15	0.42	0.09

Modulus at RTD Condition					
	R^2 Value				
Test	Density [g/cm^3]	Porosity [% Vol]	Fiber Volume [% Vol]	Matrix Volume [% Vol]	Per Ply Thickness [in]
Warp Tension	0.15	0.09	0.79	0.54	0.78
Fill Tension	0.08	0.16	0.14	0.01	0.12
Unnotched Tension 1	0.43	0.29	0.22	0.03	0.40
Warp Compression	0.57	0.55	0.56	0.18	0.67
Fill Compression	0.17	0.16	0.07	0.00	0.14
Unnotched Compression 1	0.55	0.46	0.19	0.00	0.15
In-Plane Shear (+/-45 Tension)	0.37	0.39	0.45	0.16	0.80
In-Plane Shear (V-Notch/Iosipescu)	0.18	0.22	0.01	0.23	0.00
Flexure	0.32	0.40	0.26	0.05	0.00

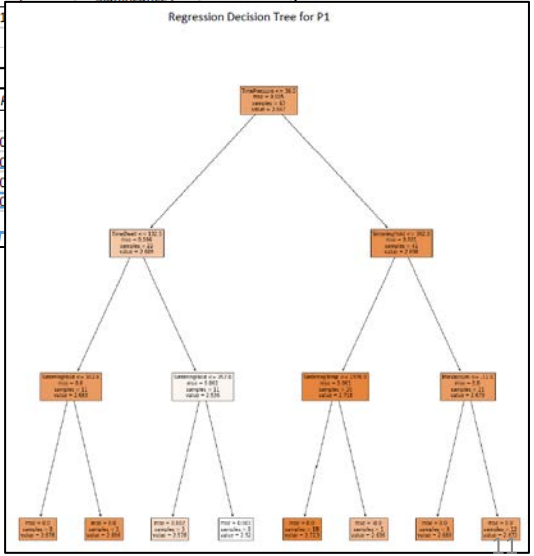
- Correlations were not strong for properties at elevated temperature so they are not shown
- Strongest correlation is with density and porosity for most strength properties at room temperature
- Fiber volume and per ply thickness have much higher correlation with most modulus properties than strength properties at room temperature

Process-Property Relationships – Multivariate Regression

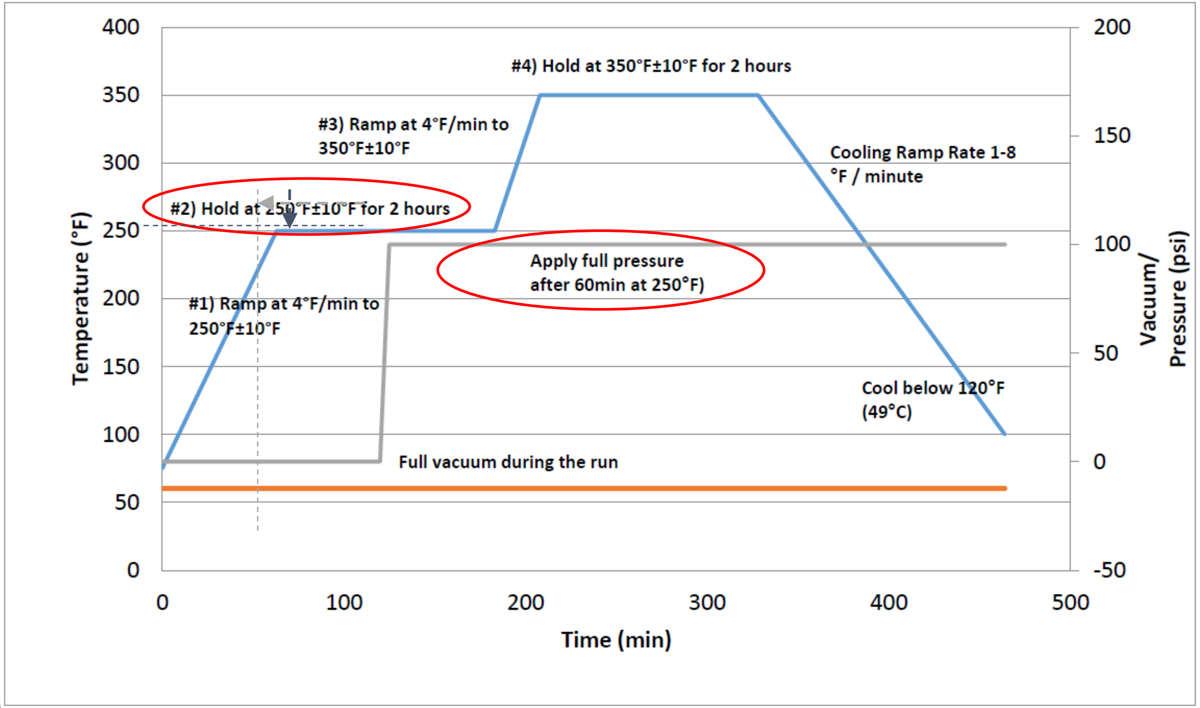
- Single parameter regression analysis does show some correlation among multiple parameters so a multivariate regression analysis was performed for each test to see if certain parameters in combination with one another show greater correlation with mechanical properties
- A regression decision tree was used to model the data
- The following processing parameters had a statistically significant effect (P-value ≤ 0.05) on the density, porosity, fiber volume, matrix volume, and per ply thickness
  - Sintering hold time
  - Duration of full pressure at initial dwell prior to ramping to the final dwell
- The same physical properties were found to have a statistically significant effect (P-value ≤ 0.05) on most mechanical properties with matrix dominant properties being more significantly affected

REGRESSION P1	DENSITY [G/CM3]				
SUMMARY OUTPUT					
Regression Statistics					
Multiple R	0.689480987				
R Square	0.475384031				
Adjusted R Square	0.429365087				
Standard Error	0.051765437				
Observations	63				
ANOVA					
	df	*SS*	*MS*	*F*	*Significance F*
Regression	5	0.13840689	0.027681378		
Residual	57	0.152740647	0.00267966		
Total	62	0.291147537			
	Coefficients	*Standard Error*	*t Stat*		
Intercept	2.883881303	1.472581481	1.958384877		
X Variable 1	0.000169744	0.000242998	0.698541291		
X Variable 2	0.00016714	0.000762835	0.21910367		
X Variable 3	-0.00183725	0.000608168	-3.020959339		
X Variable 4	-0.000218546	0.000355747	-0.61433034[illegible]		
X Variable 5	0.003774568	0.000803987	4.69481[illegible]02		

Processing	
VAR1	Min Vacuum During AC Cure ["Hg]
VAR2	Sintering Temperature [°F]
VAR3	Sintering Hold Time [minutes]
VAR4	Time at First Dwell [minutes]
VAR5	Time at Initiation of Full Pressure to Final Time of First Dwell [minutes]



Process Parameter Evaluations



Autoclave Cure Cycle from Process Specification NPS 87800

Investigate the following parameters:

1. Debulk – one 15-20 minute debulk after layup in NPS 87800 but Axiom now recommends debulk at least every 6 plies
2. Bleeder plies – three plies in NPS 87800 but Axiom now recommends one bleeder ply per every two prepreg plies
3. Initial dwell temperature – hold at 250°F ±10°F in NPS 87800 but Axiom now recommends 225°F ±10°F as a result of rheology data collected after the NPS was issued
4. Pressure – apply full pressure after 60 minutes into the initial dwell in NPS 87800 but Axiom now recommends applying full pressure at the beginning of the initial dwell

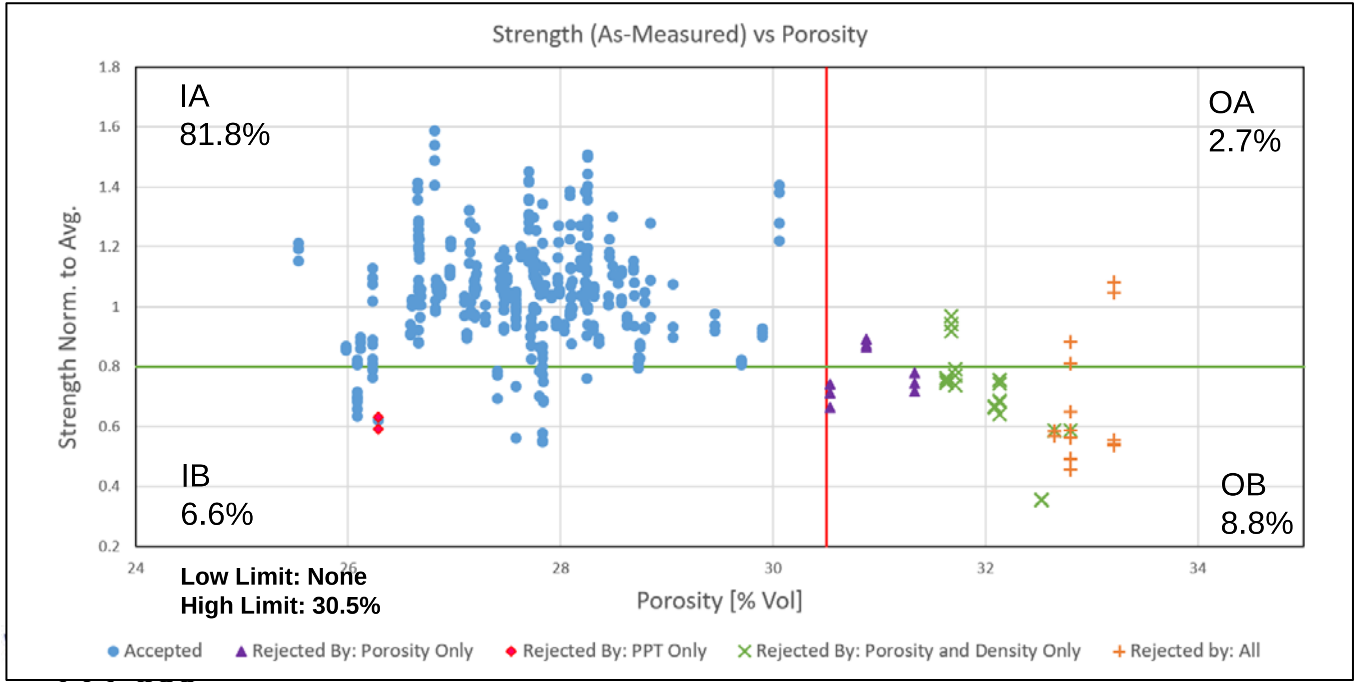
Physical Property Acceptance Limits

- **No current acceptance limits -** Axiom has typical values for many physical properties but doesn't have acceptance limits for composite physical properties from a robust dataset for this material and panel fabrication process.

- **Acceptance Limit Investigation**
  - The qualification data from all four material batches was aggregated and normalized to the mean for each property then plotted against porosity, density, and per ply thickness.
  - Limits were analyzed with the goal of optimizing the limits such that as much "good data" falls within the bounds and as much "bad data" falls outside of the bounds.
  - A program was written to determine the ratio of accepted-to-rejected strength data over a range of limits to guide initial acceptance limits. The goal was to find the highest ratio of accepted-to-rejected strength data for initial acceptance limits.
  - Limits were further optimized by looking at the data graphically.

Physical Property Acceptance Limits – Porosity

407 Total Data Points



88.5% of all data are inside of limits (IA + IB), including 81.8% that are above (IA) and 6.6% that are below (IB) 80% of the mean

11.5% of all data are outside of limits (OA + OB), including 2.7% that are above (OA) and 8.8% that are below (OB) 80% of the mean

The average CV across all strength properties is roughly 10% so 80% of the mean corresponds to a 2 x CV deviation from the mean.

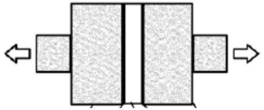
Evaluation of the Microstructure

- **Background** - *There are cases where specimens with similar measured bulk porosity have different mechanical properties.*

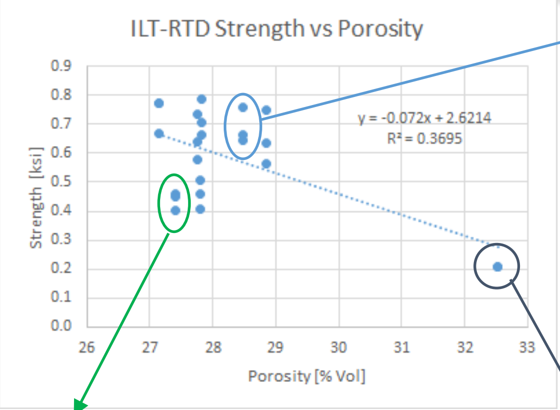
- **Methodology**
  - X-ray CT was utilized to determine if pore size, location, or distribution is different and if there is any correlation with mechanical properties.
  - Select panel remnants were submitted for X-ray CT inspection, which was performed from each batch and cure/sintering cycle to better understand the microstructure of these panels.
  - X-ray CT scans were reviewed to look for differences between the panels, and some analysis was performed to determine if the features in the microstructure correlate with panel quality.

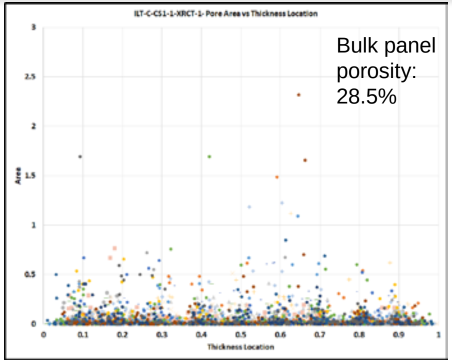
Evaluation of the Microstructure – ILT

Interlaminar (Through-Thickness) Tension

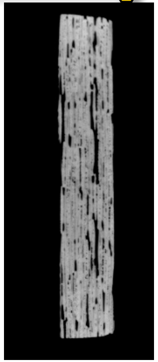


Interested in finding long interconnected pores/voids in a single plane





Analysis of 18 in-plane images



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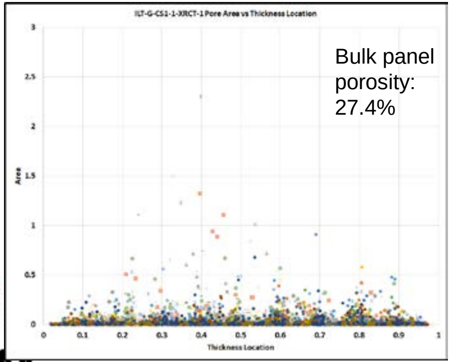
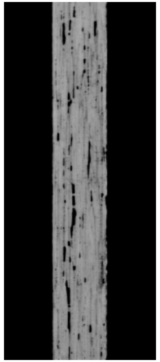


Analysis of 25 in-plane images



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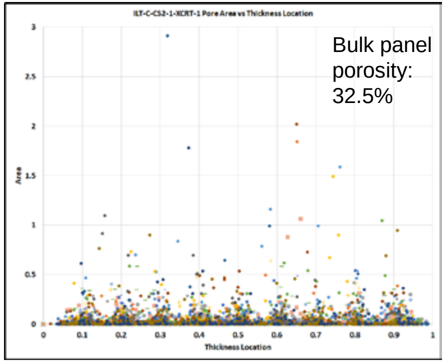
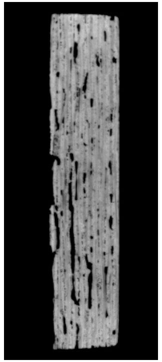
Notes:

X-ray CT scans are from off-cuts from the panels and are not actual test specimens

Images are not representative – were selected to show long interconnected pores/voids in a single plane



Analysis of 17 in-plane images



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Ox/Ox Equivalency

Methodology

1 Batch

- **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

Tasks

Test Matrix: Complete → **Test Plan:** Complete → **Manufacture of Prepreg:** Complete → **Panel Fabrication:** Q2 2023 → **Testing:** To Begin Q3 of 2023 → **Statistical Analysis**

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

Prepreg has been received. *Panel fabrication is pending revisions to the process specification.*

Ox/Ox 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 – Statistical Approaches

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
	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

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.

Durability and Long Term Safety

Methodology

- **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

3 Batches
AX-7800 Aluminum Silicate Slurry (Water Based)

Autoclave Cure and Sintering

Tasks

1. **Test Matrix:** Complete
2. **Test Plan:** Complete
3. **Manufacture of Prepreg:** Complete
4. **Panel Fabrication:** Q3 2023 to Q4 2023
5. **Testing:** To Begin Q4 2023
6. Statistical Analysis

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

Prepreg has been received. *Panel fabrication is pending revisions to the process specification.*

Durability and Long Term Safety

Initial Evaluations for Scoping Final Fatigue Test Matrix

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

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 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.

Durability and Long Term Safety

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

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

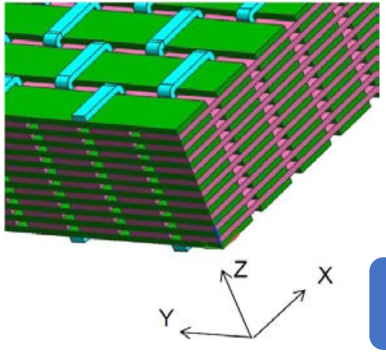
- Testing will be conducted at 1650°F.
- Relative applied stress is defined as a percentage of either the ultimate stress or peak stress, as appropriate, as determined by static testing on the same batch of material.
- One set of coupons for each test type will be reserved for either testing at an additional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

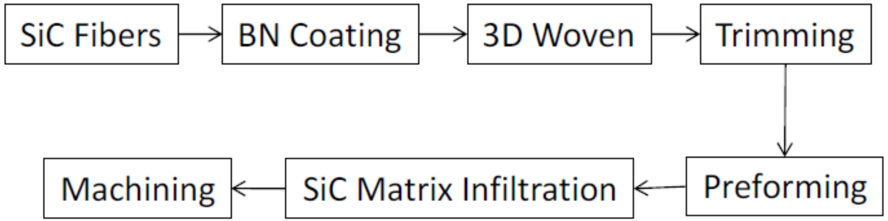
SiC/SiC Qualification

Methodology

Fiber Source:	Fiber Coating Source:	Weaver:	Panel Fabricator:
Ube Industries Ltd.	IHI Corporation	Shikibo Ltd.	IHI Corporation 3 Batches
Tyranno ZMI SiC Fiber	**Boron Nitride Coating**	**3D Orthogonal Woven Preform**	**Chemical Vapor Infiltration (CVI) and Polymer Infiltration and Pyrolysis (PIP) Hybrid Process**

SiC/SiC Processing & Fabrication





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

JAMS CECAM

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 Tasks

Documents Generated for Qualification Program:

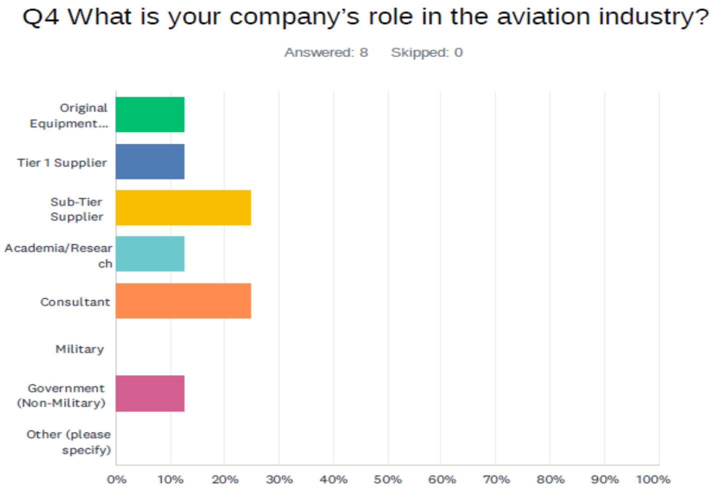
- Material Specification
- Process Specification
- Test plan – including test matrix with physical, thermal, and mechanical test requirements

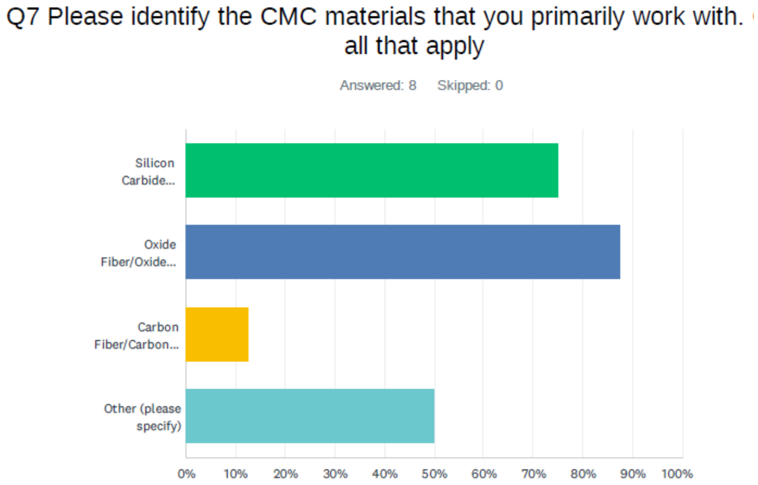
Material Specification: Complete → **Process Specification:** Complete → **Test Plan:** Complete → **Panel Fabrication:** In Progress → **Testing:** In Progress

Testing began in Q4 2022 with RTD and ETD Tension and In-Plane Shear tests.

Future Tasks Survey

- A survey was distributed to solicit input on further work to support the CMC qualification framework development.
- Responses came from a broad distribution of backgrounds and with expertise in materials relevant to commercial aviation

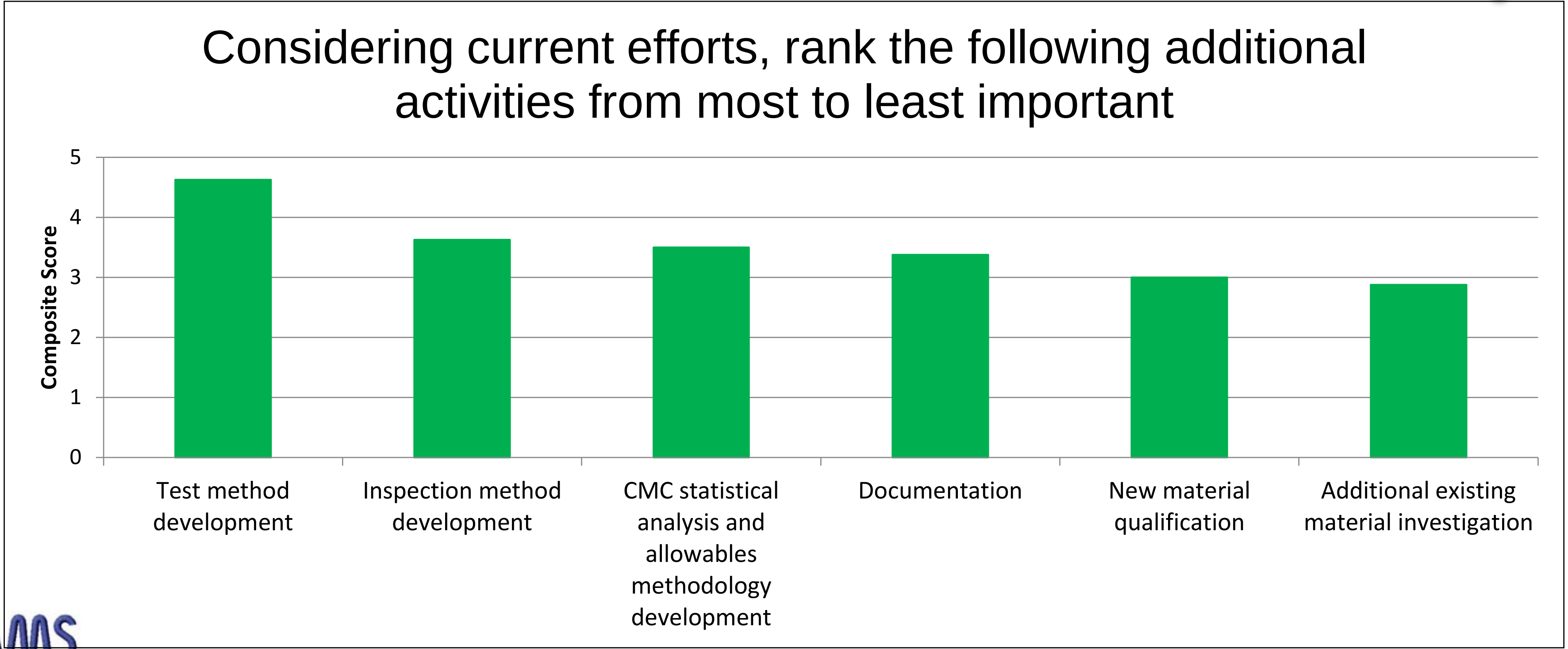




Key Survey Question

- Considering current efforts on developing a CMC qualification framework, rank the following from most to least important:
  - Documentation: Document recommended framework through FAA reports
  - Test method development: including testing, heating, and instrumentation methods
  - Inspection method development: including NDE to support framework development
  - Statistical analysis and allowables methodology development
  - New material qualification: validate the framework development that has occurred on a different CMC material and process
  - Additional existing material investigation: including effects of aging, freeze/thaw, and protective coatings

Survey Response



Current CMC Test Methods Status

- ASTM C28.07 publishes and maintains a basic set of test methods, with goals to develop additional standards when resources allow
- In the absence of CMC-specific methods for testing, heating, and instrumentation, PMC methods are generally substituted, sometimes with significant modification
- Common CMC data needs that use modified PMC methods
  - Precursor or sol-gel flow
  - Prepreg tack, drape
  - Water absorption, density, porosity
  - Short beam strength, in-plane shear ±45°, filled hole tension, bearing, bearing fatigue, compression after impact, tension after impact, fastener pull through, curved beam strength,
  - Elevated temperature tests: compression, open hole compression, interlaminar tension, fatigue, open hole tension, flexure

ASTM C28.07 Future Standards Goals

- C28.07 has identified individual standards to develop, but is not broadly addressing common issues of standardized specimen preparation, instrumentation, or heating

Mechanical Properties	Flats-Bars	Tubes/Rods
Compression Properties (ultimate, fracture, PropL)	Modified for HT Tests	*RT in draft*
Shear Properties (ultimate, fracture, PropL) Interlaminar, Translaminar	•Improved for Interlaminar •Improved for Translaminar	New for Torsion
Transthickness Tensile Properties (ultimate, fracture, PropL)	Improved for HT	Needed???
Fracture Toughness / Crack Growth Resistance/	•*RT Mode I in Draft*	Needed???
Strain Energy Release Rate/	•New for Mode II & Mode III	
Interlaminar and Translaminar	•New for Translaminar	
(Mode 1, 2, 3, Mixed)	•New HT for All	
Open Hole Tensile Strength Properties	C1869	New for Tubes
Open Hole Compression Strength Properties	NEW base D6484	New for Tubes
Notch Tensile Strength Properties	NEW	New for Tubes
Notch Compression Strength Properties	NEW	New for Tubes
Pin Bearing Strength Properties	NEW base D5961	New for Tubes
Torsion Shear Joint Strength	*RT in Draft*	NA
Single Filament Tensile	C1557 Improved,	NA
Dry/Impreg. Tensile Tow Tests	NEW	NA

ASTM C28.07 would be the most appropriate place for standard guides to Ox/Ox testing.

•Source: Steve Gonczy

General CMC Testing Challenges

- **Specimen alignment and gripping** –CMCs are in general stiffer and more brittle than metals and usually require alignment fixtures. Gripping needs to be done with hydraulic grips with metallic or polymer inserts to mitigate surface roughness effects and distribute forces more evenly
- **Specimen shape and machining** –Typically notches or dog bones are required to insure high stress regions within the gage section; machining CMCs can be difficult especially if sharp notches are required
- **Specimen size** – Due to cost and effort of production, coupons are often sized as small as possible, which can impact results if specimen architecture/repeat units are not compatible with the size
- **Strain instrumentation** – Since CMCs are more brittle and matrix cracking occurs at relatively low strain (0.03 to 0.1%), more sensitive strain measures are required –offset strain techniques for nonlinearity parameters will have much smaller offset strains (0.005% instead of 0.2% as in metals) which still may not be adequate
- **Material class variability** – CMC material systems have a very wide range of material behavior characteristics ($E_f >> E_m$, $E_f = E_m$, $E_f < E_m$; 2D & 3D weaves; very low to very high K's, CTEs, CMEs; various tow sizes and FAW) so standard test methods and specimen designs that work for one material system won't work for all/others.
- **High temperature environment** – Furnace design, heating rates, temperature distributions, and interface with grips and instrumentation can vary widely.

•Sources: Greg Morscher and Carl Rousseau

Documentation Next Steps

- **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

Publications

Publication Type	Date	Publication
Conference Presentation	Jan-17	R. Andrulonis, “CMC Qualification Research at NIAR,” United States Advanced Ceramics Association (USACA), Cocoa Beach FL, January 2017.
Conference Presentation	Jan-18	R. Andrulonis, “CMC Qualification Research at NIAR,” United States Advanced Ceramics Association (USACA), Cocoa Beach FL, January 2018.
Conference Presentation	Jan-19	M. Opliger, “CMC Qualification Research at NIAR,” United States Advanced Ceramics Association (USACA), Cocoa Beach FL, January 2019.
FAA Technical Reports	Dec-19	FAA Annual Report, “Ceramic Matrix Composites (CMC) Characterization and Qualification Guidelines for Aircraft Design and Certification,” December 2019 (submitted).
Conference Presentation	Jan-20	M. Opliger, “CMC Qualification Research at NIAR,” United States Advanced Ceramics Association (USACA), Cocoa Beach FL, January 2020.
FAA Technical Reports	Dec-20	FAA Annual Report, “Ceramic Matrix Composites (CMC) Characterization and Qualification Guidelines for Aircraft Design and Certification,” December 2020 (submitted).
Conference Presentation	Mar-21	R. Andrulonis, “CMC Qualification Research,” SAE Aero Tech conference, March 2021.
FAA Technical Reports	Dec-21	FAA Annual Report, “Ceramic Matrix Composites (CMC) Characterization and Qualification Guidelines for Aircraft Design and Certification,” December 2021 (submitted).
Conference Presentation	Jan-22	M. Opliger, R. Andrulonis, “CMC Qualification Research at NIAR,” United States Advanced Ceramics Association (USACA), Cocoa Beach FL, January 2022.
Conference Presentation	Mar-22	M. Opliger, “CMC Qualification Research at NIAR,” ESA Virtual Conference, March 2022.
Conference Presentation	Jan-23	M. Opliger, R. Andrulonis, “Oxide-Oxide Process Property Relationships,” United States Advanced Ceramics Association (USACA), St Augustine FL, January 2023.
FAA Technical Reports	May-23	Processing Property Relationship of Oxide/Oxide Composites
FAA Technical Reports	Sep-23	Oxide/Oxide Ceramic Matrix Composites Qualification Summary and Lessons Learned
FAA Technical Reports	TBD	Durability and Long Term Safety

JAMS
CECAM

Questions/Comments:

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