

NATIONAL INSTITUTE FOR AVIATION RESEARCH

WICHITA STATE UNIVERSITY

Qualification Framework for Oxide/Oxide and SiC/SiC Ceramic Matrix Composites

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JOINT ADVANCED MATERIALS & STRUCTURES CENTER OF EXCELLENCE



Federal Aviation Administration

RESEARCH TEAM & OBJECTIVES





APPROACH



- Generate Qualification Documents for CMCs and Perform Material Qualification to Establish Qualification Guidance
 - Material and Process Specifications
 - Test Plan
 - Statistical Analysis Report with B-Basis Allowables or Estimates
- Generate Equivalency Documents for CMCs and Perform Material Equivalency to Establish Equivalency Guidance
 - 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
- Generate Technical Reports
- Generate Guidance Documents



SCHEDULE





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



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Ox/Ox Qualification







3 Batches

- 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







- 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.



NOUSTRIAL MODERNIZATION

DX/DX MATERIAL AND PROCESS SPECIFICATIONS



Titles:

- NCAMP Material Specification NMS 780 Rev A: Autoclave Cure, Slurry Processed Axiom Oxide/Oxide Prepregs
- NCAMP Process Specification NPS 87800 Rev B: Fabrication of NMS 780 Qualification, Equivalency, and Acceptance Test Panels

Objectives:

- Define technical requirements and quality assurance requirements for material procurement
- Define materials, test laminate fabrication, and quality assurance requirements for laminate fabrication

Status: Draft material and process specifications authored ahead of qualification activity. Specifications to be finalized at the conclusion of data analysis.





OX/OX QUALIFICATION – PROCESS ISSUES AND EVALUATIONS

- 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.
- 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/cm³
 - Per Ply Thickness Acceptance Limit: 0.0085 0.0100 in/ply
- Performed a limited number of process-parameter evaluations to understand process sensitivities and aid in revising the process specification by altering the following: **Debulks Complete (Panel 1)**
 - Frequency of Vacuum Debulks
 - Number of Bleeder Plies
 - Initial Dwell Temperature
 - Time Pressure Applied
 - Edge Dams















Title: Processing-Property Relationship of Oxide/Oxide Composites

Objectives:

- Understand the effects of processing variables and physical properties on the variability of mechanical properties
- Define physical property acceptance limits reducing the variability of mechanical property data

Status: Report drafted, reviewed by FAA personnel, revision in work

		R2 Value												
	Test - RTD Strength		Porosity [% Vol]	Fiber Volume [% Vol]	Matrix Volume [% Vol]	Per Ply Thickness [in]	Min Vacuum During AC Cure ["Hg]	Sintering Temperature [°F]	Sintering Hold Time [min]					
	Warp Tension	0.41	0.39	0.07	0.00	0.23	0.01	0.00	0.09					
	Warp Compression	0.72	0.70	0.35	0.00	0.43	0.00	0.25	0.00					
	Fill Tension	0.06	0.12	0.16	0.01	0.17	0.28	0.11	0.12					
	Fill Compression	0.29	0.56	0.67	0.52	0.10	0.62	0.17	0.01					
- 1	n-Plane Shear (+/-45 Tension)	0.76	0.60	0.04	0.03	0.32	0.87	0.23	0.17					
	Unnotched Tension	0.23	0.16	0.01	0.09	0.04	0.13	0.34	0.05					
	Unnotched Compression	0.82	0.75	0.60	0.01	0.43	0.26	0.00	0.01					
	Open-Hole Tension	0.08	0.04	0.12	0.10	0.24	0.20	0.23	0.00					
	Open-Hole Compression	0.17	0.38	0.04	0.34	0.01	0.02	0.17	0.03					
	Filled-Hole Tension	0.56	0.49	0.17	0.01	0.20	0.01	0.04	0.23					
Int	erlaminar Shear (Double Notch)	0.01	0.02	0.00	0.05	0.00	0.14	0.19	0.15					
	Single-Shear Bearing	0.73	0.80	0.29	0.01	0.31	0.29	0.33	0.11					
	Double-Shear Bearing	0.00	0.03	0.13	0.43	0.09	0.18	0.22	0.00					
	In-Plane Shear (V-Notch)	0.06	0.07	0.10	0.54	0.07	0.02	0.08	0.06					
In	Interlaminar Shear (Short-Beam)		0.45	0.39	0.10	0.37	0.00	0.12	0.00					
	Interlaminar Tension	Interlaminar Tension 0.43 (0.33	0.01	0.21	0.08	0.38	0.13					
	Flexure	0.13	0.16	0.02	0.35	0.47	0.00	0.00	0.05					
Correlation Color Scale														
	0.1 0.2	0.3	0.4	0.	5	0.6	0.7 0	.8 0.9						

Example correlation table for physical properties and mechanical strength properties used to generate acceptance limits





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DX/DX QUAL. 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.



DUSTRIAL MODERNIZATION

- 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 ANDVA method.
 - Grouping by panel (6 groups) results in 55 more reasonable 50 material allowables when using ANOVA, but this could result 40 ۱ ·ږ 35 in an overestimation of the b-basis value 30 if batch-to-batch 25 variability is not 20 significantly smaller than panel-to-panel variability, so caution should be taken.





Properties – Structured vs Unstructured



<u>Unstructured / Single-Point</u>

- Warp Tension (WT) RTD
- Warp Compression (WC) RTD, ETD
- Fill Compression (FC) RTD
- Interlaminar Shear (SBS) RTD
- Filled-Hole Tension 1 (FHT1) RTD, ETD (Mod CV)
- Double-Shear Bearing 1 (DSB1) RTD (2% Offset)



B-estimate is greater than 50% of the mean



*The CMH-17 Statistics Working Group is looking at Bayesian Methods for determining allowables – expected to be better approach when multiple sources of variation are present.

DX/DX QUALIFICATION – PATH FORWARD

- WICHITA STATE UNIVERSITY
- Data generated to date has mostly come from four batches of material for each property and condition.
- For structured datasets, ANOVA is being used.
 - Allowables are unreasonably low in many cases because of significant panel-to-panel variability and only four material batches in the dataset.
- The CMH-17 Joint Statistics and CMC Data Review Working Groups have been briefed on the qualification program and the datasets. They gave the following key recommendations:
 - Only publish allowables for properties with reasonable allowables and classify the others a screening data. Don't publish unreasonable allowables. Engines with CMCs have been certified without allowables for every property.
 - Historical data may be useful in determining typical process variability, which can be helpful in making engineering judgements for allowables.
- For datasets requiring ANOVA, "screening" values will be published. A fifth batch of material will not be tested.



DX/DX DATA REPORTS AND STATISTICAL ANALYSIS

Titles:

- Axiom AX-7800-DF11-5HS3000D Qualification Material Property Data Report
- Axiom AX-7800-DF11-5HS3000D Qualification Statistical Analysis Report

Objectives:

• Publish material property data and statistical analysis results for oxide/oxide CMC qualification framework material

Status:

• Reports to be compiled at the conclusion of qualification testing and data analysis

Data generated as part of this qualification program is currently being balloted for inclusion into CMH-17 Volume 5 Rev. B







Ox/Ox Equivalency



DX/DX EQUIVALENCY





- 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





DX/DX 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.
 - <u>Physical:</u> NDE, Per Ply Thickness, Fiber and Matrix Volume, Bulk Density, and Open Porosity
 - <u>Thermophysical:</u> Specific Heat and Thermal Diffusivity, Conductivity, and Expansion in X, Y, an Z Directions
 - <u>Mechanical:</u>

USTRIAL MODERNIZATION

- 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)

More panels may be needed for equivalency if process variability is the key issue. Plans have been made to allow for specimens to be extracted from more than two panels for some properties where significant panelto-panel variation has been observed.



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Durability and Long-Term Safety



DURABILITY AND LONG-TERM SAFETY





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Program:

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Test Report

- 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









Panel fabrication is pending outcome of the equivalency

Other Documents for Durability and Long-Term Safety



DURABILITY AND LONG-TERM SAFETY: TEST PROGRAM AND REPORT



Title: Ceramic Matrix Composite (CMC) Materials Guidelines for Aircraft Design and Certification: Durability and Long-Term Safety of CMCs

Objectives:

- Perform testing to characterize durability and long-term safety of an oxide/oxide CMC
 - Room temperature fatigue
 - Thermal exposure
 - High temperature creep
- Report material properties and provide guidelines for future durability and long-term safety testing

Status: Test plan drafted, test program awaiting completion of base qualification framework activity

		F														
		l li		Fatiqu	Fatique			Test	Numbe	hes x N	s x No of Panels x No of Specimens					
							Μ	ethods	Expos	ure Tem	oerature	e and D	urati	on (see l	Notes 2,	3)
				Lavun (see Not	e 1) Env	Type (Test	N	(see ote 2)	1650F180 500b 500	0F14001	1650F	1800F	1400)F1650F 0b5000b	1800F 5000h	1650F TBD
				[0] _{5S}	Wa	rp Tension	A	STM	1x2x3 1x2	x3 1x2x3	3 1x2x3	1x2x3	1x2x	3 1x2x3	1x2x3	1x2x3
						(PTA)	C	1275							<u>ل</u>	
		Therma	I F	xnnsure				Num	ber of Ba	tches x	No of				x2x3	
Ļ								Pane	els x No o	of Speci	mens				x2x3	1x2x3
				Test Type s Unnotched Tension- Tension s Notched Tension- Tension s Notched Tension- Tension				Targ	geted Cycle Count (see		Relevant Test					
						R-Value 0.1 0.1 -1				Note 2)		Methods (see			x2x3	1x2x3
	Lay	up (see Note	1)					"Lov	v" "Mid	" "H	gh"	Notes 3, 4)				1 x 2 x 2
	[45/0	/-45/90/-45/9	90]s					1x3x	3 1x3x	3 1x	3x3	AS	ГМ	21360	1 x2x3	18283
															x2x3	1x2x3
	[45/0	/-45/90/-45/9	90]s					1x3x	3 1x3x3	3 1x	3x3	ASTM C1360 ASTM C1869 3 ASTM C1360		ГМ C1360		
														C1869		
	[45/0	/-45/90/-45/9	90]s					2x3x	$x^3 = 2x^3x^3$	3 2x	3x3			x2x3		
	TBD (See Note 6) [0] ₁₀ [45/0/-45/90/-45/90] _s		Compression								AS	ΓM]	'M D6484			
			Interlaminar Shear Interlaminar Tension (see Note 7) Fatigue After Impact		TBD (See Note 6 0.1		2x3x	3 2x3x3	3 2x	3x3	3 ASTM C1360 ASTM C1292		x2x3	1x2x3		
													STM C1292			
							2x3x	$x^3 = 2x^3x^3$	3 2x3x3		ASTM C1360					
											ASTM D7291		_			
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					Number	of Batche	es x	No of	Panels x	No of S	specim	nens		01260	_	
re	ep	Туре (Т	Fest	Test Method		Relativ	ve S	stress	(see Note	s 3, 4)				CI360		
te i		Environme	nt)	(see Note 2)	40%	50%	6	0%	70%	80%	Т	BD		D/130		
)] _{5S}		Warp Tensi	on	ASTM		1x2x3	1x	2x3	1x2x3	1x2x3	3 1x	2x3		00484	-	
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				ASTM C1227												In .
				C1337												I∐-\
															1	



Planner

SiC/SiC Qualification



ISTRIAL MODERNIZATION





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)



Guidance Documents



GUIDANCE ON QUALIFICATION PROCEDURE



Title: Material Qualification and Equivalency for Oxide/Oxide Ceramic Matrix Composite Material Systems DOT/FAA/AR-03/19 Material Qualification and Office of Aviation Researce Washington, D.C. 20591 Equivalency for Polymer Matrix **Composite Material Systems: Updated Procedure Objectives**: Provide guidance for performing oxide/oxide CMC material gualification and equivalency similar to DOT/FAA/AR-03/19 SPECIMEN SELECTION METHODOLOGY AND TRACEABILITY PER ENVIRONMENTAL CONDITION AND TEST METHO Applicable material forms and batch requirements Material Batcl CMC-appropriate physical and mechanical test methods and environments ۲ Panel Manufacturing & Independent Cure PANEL PANEL 2 PANEL : PANEL PANEL (

- CMC laminate and specimen manufacturing, machining, inspection ۲
- Statistical analysis relevant for types of variability common in oxide/oxide CMCs ullet

Status:

USTRIAL MODERNIZATION MATERIALS & MANUFACTUR

- Report has been revised to provide recommendations for qualification and equivalency with added content discussing topics for which the approach has not yet been validated in CMCs (e.g., equivalency and acceptance testing)
- Revised document with FAA for review



Example specimen selection methodology





GUIDANCE ON WRITING MATERIAL SPECIFICATIONS



Title: Guidelines and Recommended Criteria for the Development of a Material Specification for Oxide/Oxide Ceramic Matrix Composite Prepregs

Objectives:

- Provide guidance for writing specifications to control the procurement of oxide/oxide CMC pre-impregnated material similar to DOT/FAA/AR-02/109
 - Visual, physical, chemical, and mechanical property requirements
 - Storage and handling requirements, defect limit definition
 - Quality assurance and receiving inspection

Status:

DUSTRIAL MODERNIZATION

- Initial draft completed and shared with the FAA. Report is now being revised based on discussions from Feb. 2025 meeting.
- Comments are welcome on either the initial draft or the revision, once completed.





GUIDANCE ON WRITING PROCESS SPECIFICATIONS



Title: Guidelines for the Development of Process Specifications, Instructions, and Controls for the Fabrication of Oxide/Oxide Ceramic Matrix Composites

Objectives:

- Provide guidance for writing specifications to control the processing of oxide/oxide CMCs similar to DOT/FAA/AR-02/110
 - Characterization and control of oxide/oxide CMC key material attributes
 - Oxide/oxide CMC material storage and handling
 - Defining procedures for control of tooling, equipment, layup, consolidation, sinter, machining, and heat cleaning
 - Process monitoring and inspection

Status:

- Initial draft completed and shared with the FAA. Report is now being revised based on discussions from Feb. 2025 meeting.
- Comments are welcome on either the initial draft or the revision, once completed.







GUIDE FOR COUPON HEATING, GRIPPING, INSTRUMENTATION, AND TESTING WICHITA STATE UNIVERSIT

Title: Standard Guide for Heating, Gripping, and Instrumentation of Oxide/Oxide Continuous Fiber Advanced Ceramics

Objectives:

- Provide guidance in the form of an industry-standard guide for heating, gripping, and instrumentation of oxide/oxide CMCs relevant to a range of test methods
 - Temperature methods, control, uniformity, and thermal surveys
 - Specimen gripping pressure, use of tabs, alignment tools
 - Temperature instrumentation: contact and non-contact methods
 - Strain instrumentation: appropriate use of extensometers, strain gages, visual techniques

Status: Many processes developed during material qualification framework activity. Additional testing and documentation needs to be identified after finalization of current guidance.



High temperature tension test with strain and temperature instrumentation





NEW AND REVISED CMC-SPECIFIC TEST METHODS



Test Method Description of Effort Type **Objectives:** Separate different test options (round bar in a voke, rectangular bar in grips, etc.) into reportable Types Leverage lessons learned from CMC qualification framework expansion activities to improve or author new CMC-specific test methods Require reporting of test type Tension Revision Add US dimension and unit options throughout (RT and ET are separate standards) Revise specimen drawings to contain achievable tolerances, update drawing GD&T to state of the art Recommend strain ranges for modulus and require reporting of range, include quidance on using strain gages Separate different test options (edge loaded vs face loaded) into reportable Types Require reporting of test type Revise current standardized test methods (tension, Add US dimensions and unit options throughout Revision Compression Revise specimen drawings to achievable tolerances, update drawing GD&T to state of the art compression, fracture toughness) to: Recommend strain ranges for modulus and require reporting of range, include quidance on using strain Improve usability Standardize an asymmetric four-point bend (4APB) ILS test method Provide more specific configuration selection guidance Currently standardized PMC test methods do not work well for CMCs or at elevated temperatures Interlaminar Shear New New standard to be applicable at RT and elevated temperature Reduce the frequency of unnecessary dispositioning NASA LaRC and GE have internal test methods that can be used as a baseline Standardize a thick-plate. 3-pt bend ILT test method Author new, CMC-specific standardized test methods for properties essential to CMC design Current PMC test method (flatwise tension) does not work at elevated temperature Interlaminar Tension New New standard to be applicable at RT and elevated temperature GE has an internal test method that can be used as a baseline New standard that is an adaptation of a current PMC standard Interlaminar shear, interlaminar tension, open-hole Specimen geometries will be obtainable for CMCs **Open-Hole Compression** New compression, compression creep, flexure creep, bearing Will include quidance on gripping testing low strain to failure materials Will include quidance on elevated temperature testing New standard based on a framework of the tension creep standards (PMC and CMC) Defined specimen geometries based on load configuration (R-value) and meetable tolerances Compression Creep New Guidance on fixturing and instrumenting Applicable to RT and elevated temperature Initial effort focusing on one revision and one new New standard based on a framework of the tension creep standards (PMC and CMC) Defined specimen acometries based on load configuration (R-value) and meetable tolerances standard Flexure Creep New Guidance on fixturing and instrumenting Key: Applicable to RT and elevated temperature Revision of the room temperature tension standard New standard that is an adaptation of a current PMC standard Specimen tolerances will be obtainable for CMCs In-work Bearing New New standard for interlaminar tension Will include guidance on testing low strain to failure materials Will include guidance on elevated temperature testing Planned Using GE Aerospace standardized method as a foundation Rewrite compliance equation used in data reduction to be accurate for thick mach Fracture Toughness Revision Add load introduction method using a harness and through-width machined holes

Status:

QUESTIONS?



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