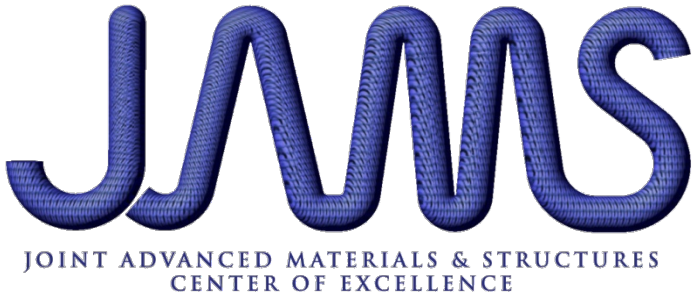

Review of Advanced Materials - Characterization and Qualification Guidelines

John Tomblin, Wichita State University
Rachael Andrulonis, Wichita State University

JAMS 2017 Technical Review
March 21, 2017

WSU Projects

- **Advanced Fiber Reinforced Polymer Composite Materials** Characterization and Qualification Guidelines for Aircraft Design and Certification
- **Composite Repair** Materials Characterization and Qualification Guidelines for Aircraft Design and Certification
- **Ceramic Matrix Composite (CMC)** Materials Characterization and Qualification Guidelines for Aircraft Design and Certification
- **Polymer-Based Additive Manufacturing** Characterization and Qualification Guidelines for Aircraft Design and Certification
- **Bond Process** Qualification Protocol
- **Adhesive Characterization** Guidelines for Aircraft Design and Certification

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Overview

- **Shared Goals**
  - Develop the qualification framework for a unique material form.
  - Generate shareable databases and guidelines to benefit industry and regulatory authorities.

- **Timeline**
  - Projects in varying stages of development
  - All projects will have initial qualification framework and test data in early 2018

Technical Approach

- Develop a framework to advance selected material forms into the aerospace industry.
- Utilize the experience and framework of the NCAMP composite program as an example of process sensitive material characterization.
- Assess the validity of the qualification framework with equivalency testing.

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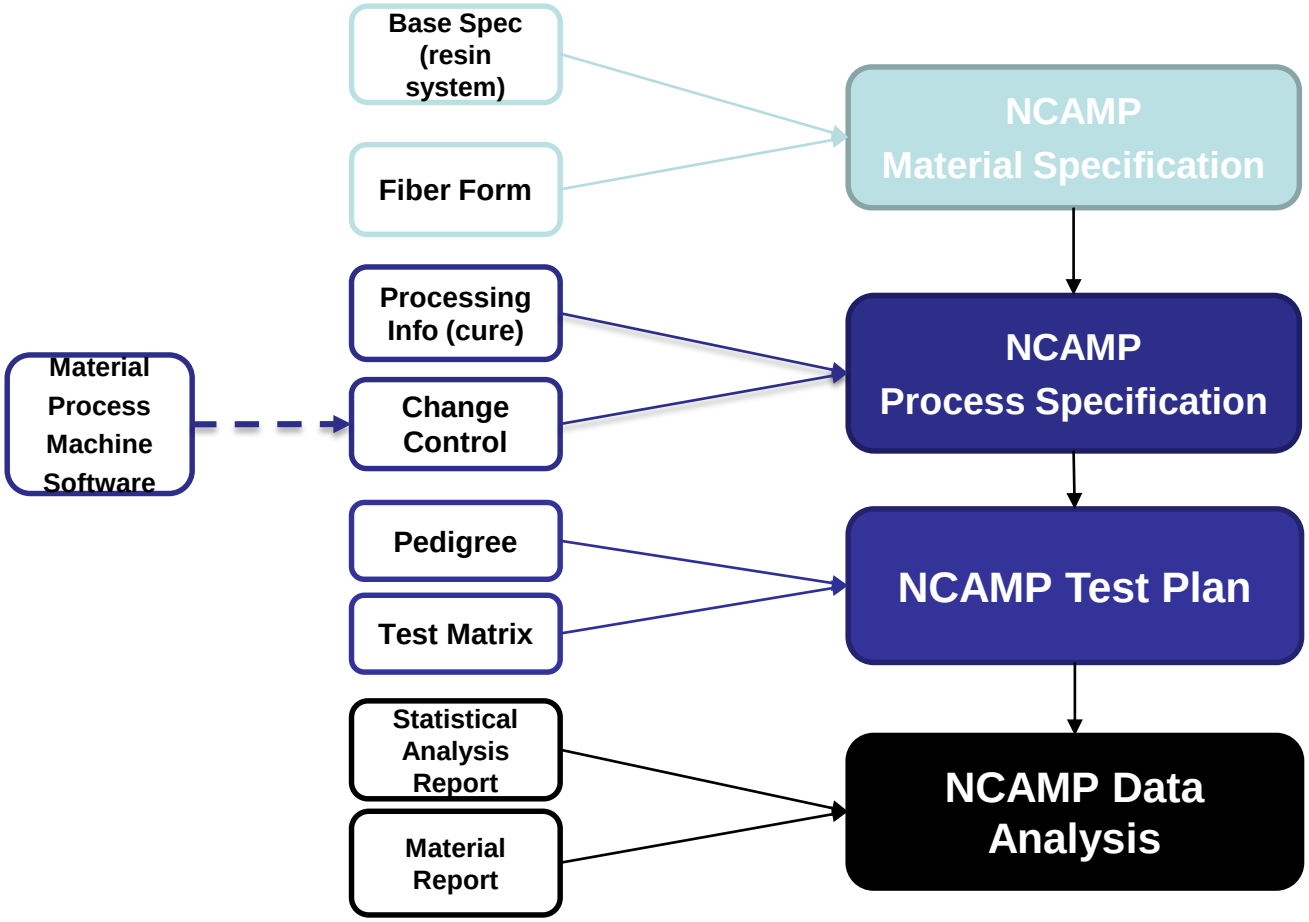
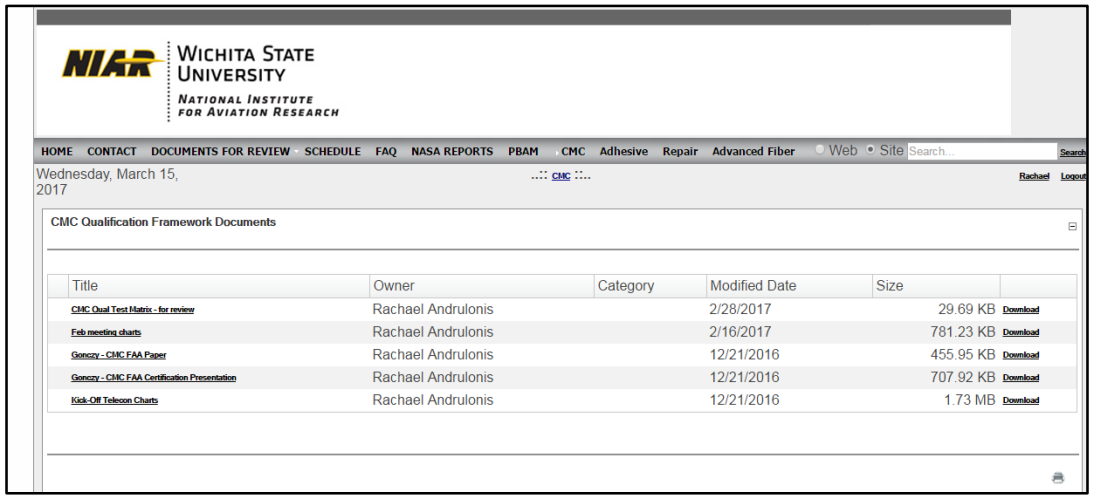
Qualification Documentation



On-Line Portal

- All members of each Steering Committee have access to this site
- Meeting charts
- Documents for review
- Other relevant resources



Example: http://www.niar.wichita.edu/ncampportal/CMC/tabid/177/Default.aspx

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ADVANCED FIBER MATERIALS

Survey Overview

- **Objective**: To generate industry driven input on the development of a qualification framework for an advanced PMC material system.

- Administered to the composites community through Survey Monkey (Oct - Nov) to 143 responses received.

- The survey included multiple parts:
  1. Current PMC material forms and processes
  2. Future/planned material forms and processes
  3. Applications and parts
  4. Factors affecting the decision making process when considering new PMC materials
  5. Individual and company interest in serving on steering committee or contributing to CMH-17

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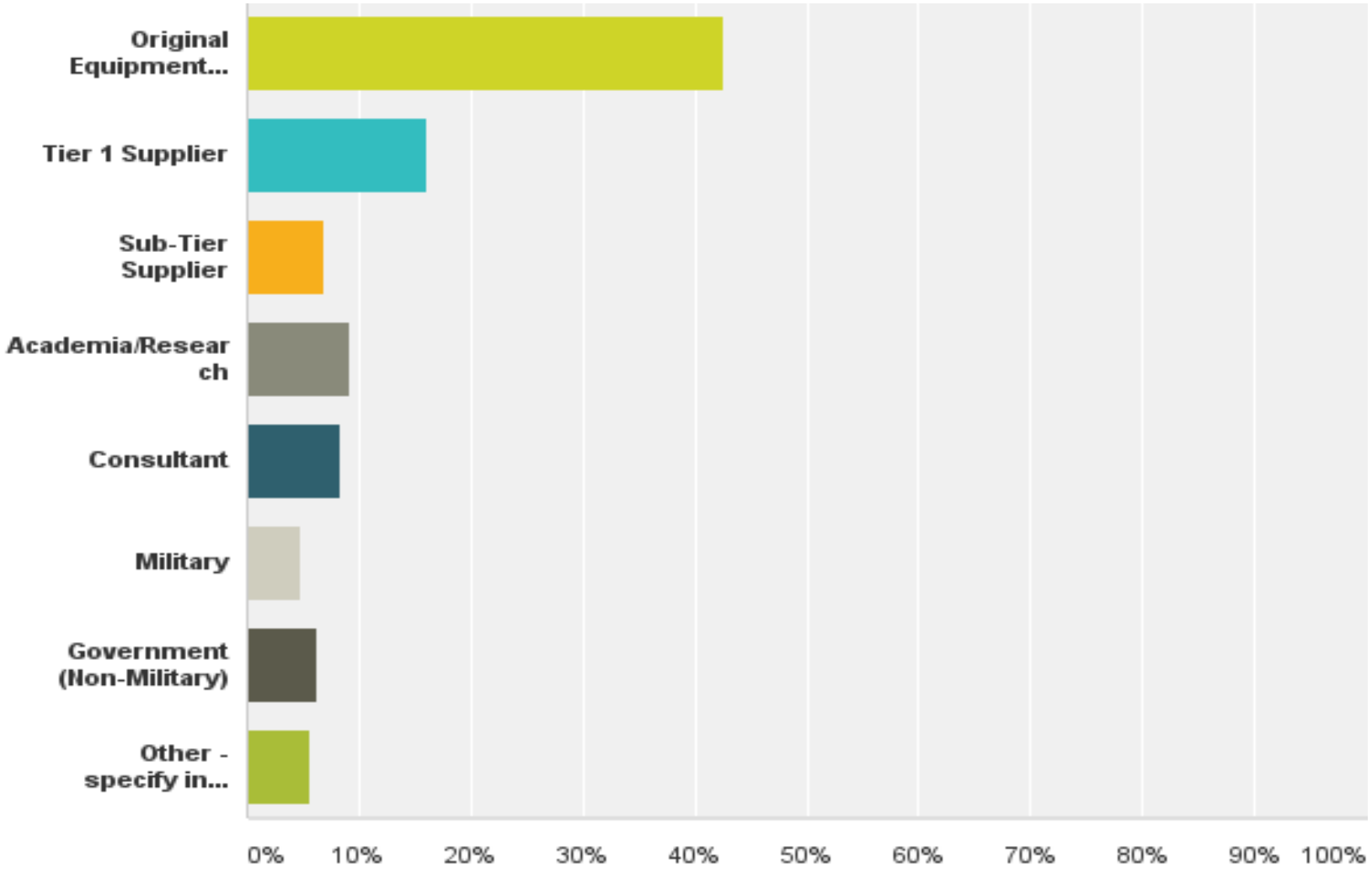
Results: Organization Type



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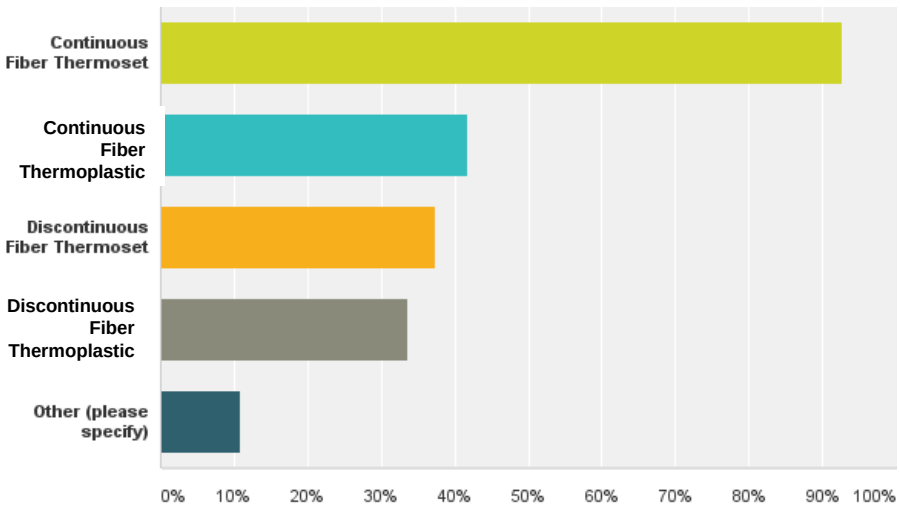
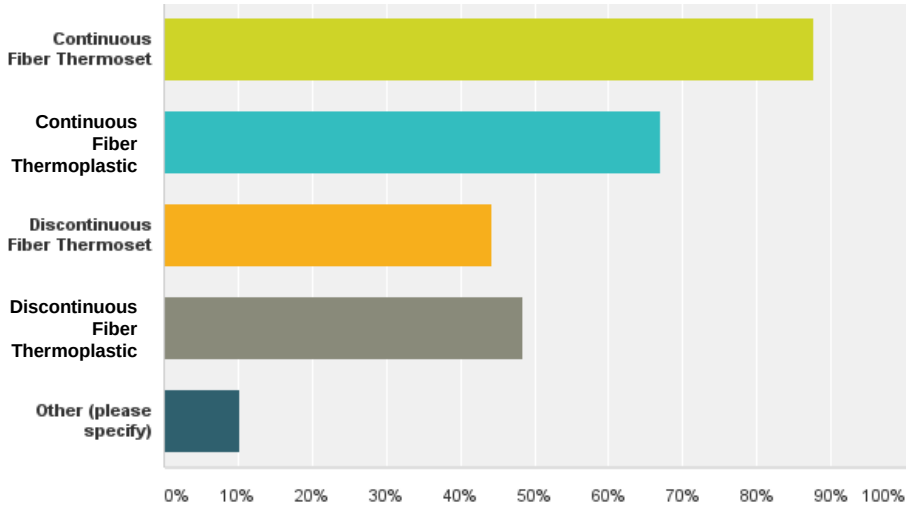
Results: PMC Material Forms



Currently In Use



5 – 10 years

PMC Form	NOW [%]	5-10 Years [%]	Change
Continuous Fiber Thermoset	93	88	-5%
Continuous Fiber Thermoplastic	42	67	60%
Discontinuous Fiber Thermoset	37	44	19%
Discontinuous Fiber Thermoplastic	34	48	44%
Other	11	10	-5%

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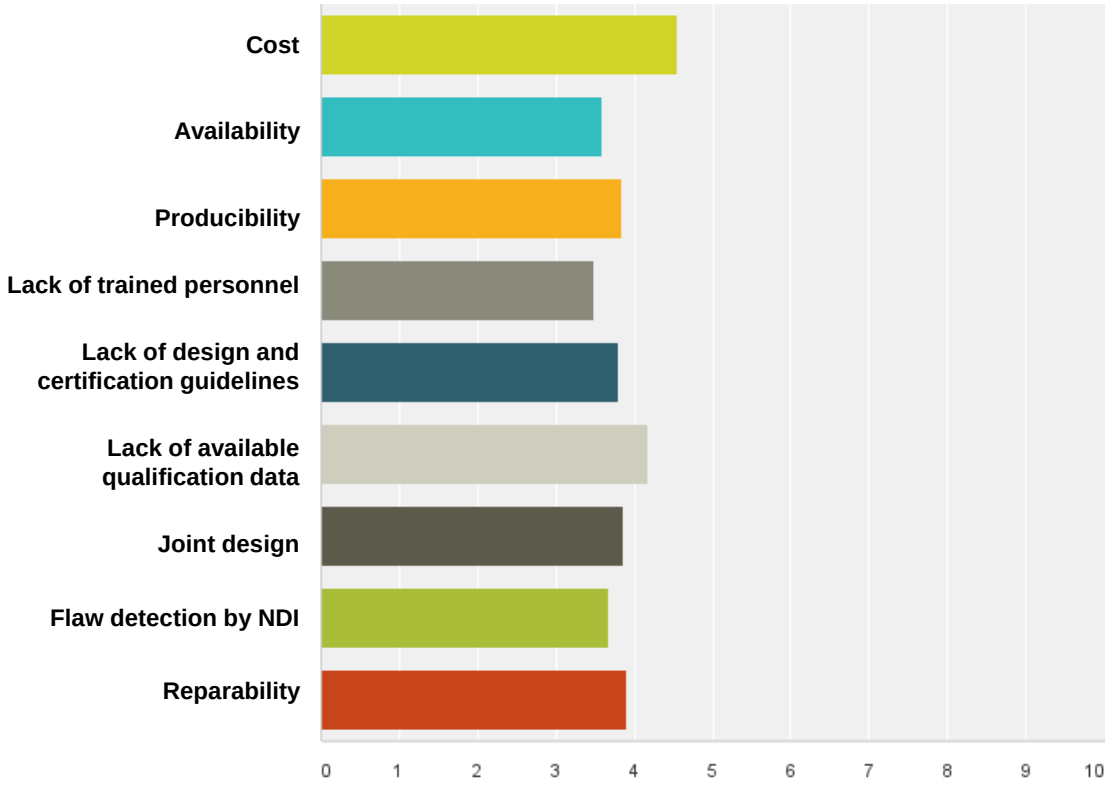
Challenges



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Advanced Fiber Current Status

- **Material Selection**
  - Thermoplastic composite based on survey results
  - Initial phase: UD Tape Thermoplastic
  - Secondary phase: Chopped Fiber Thermoplastic
  - Material Partner: Tencate - TC1225 (PAEK) UD Carbon
    - Polyarlyetherketone (PAEK) resin
    - Available commercially
    - Low melting point that makes for easier processing
    - Can be cured in an oven or autoclave
    - Can be used in melt overlay scenarios which make it more versatile for co-cure situations
    - Good smoke, toxicity, and flammability results.
- **Next Steps**
  - Steering Committee
  - Draft of test matrix and test plan

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REPAIR MATERIALS

Repair Survey Overview

- **Objective**: To seek industry experts' guidance regarding
  - Repair material and technology development - existing repair procedures and manuals, training curriculum and technology transfer, repair records keeping and new repair processing technologies
  - Identify a repair composite system(s) suitable for the initial qualification methodology framework.

- Administered to the repair community through Survey Monkey (Nov-Dec 2016).

- Results included information on:
  - Composite parts that are most commonly damaged
  - Most commonly used materials and processes for repair
  - Successes, challenges and lessons learned encountered during repair

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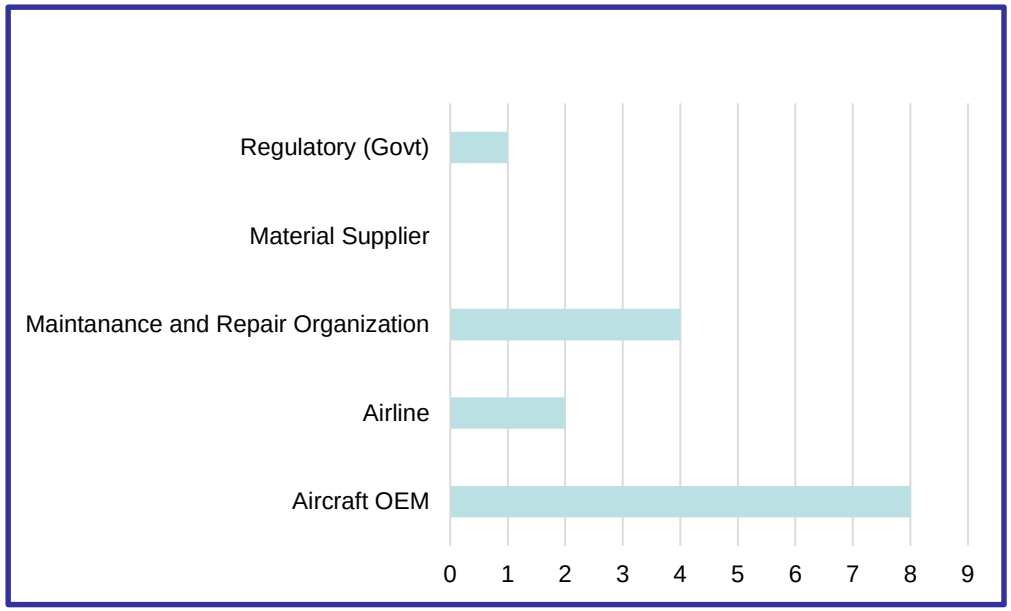
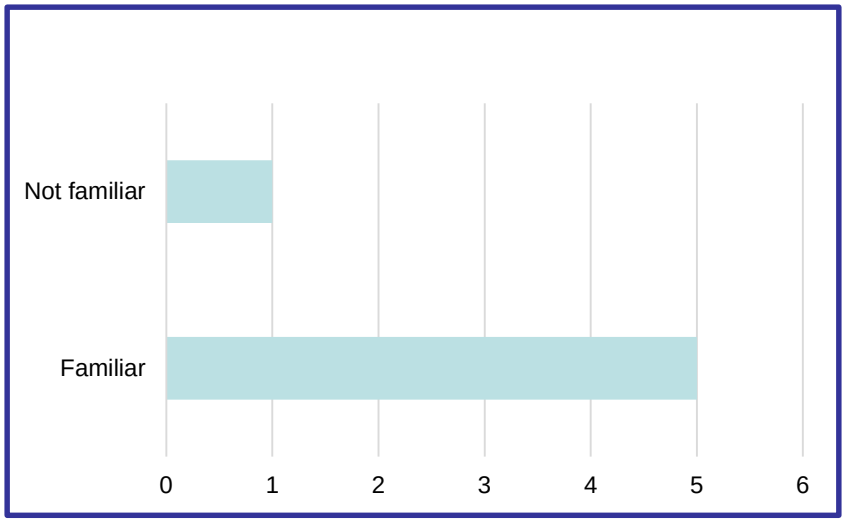
Repair Survey Results

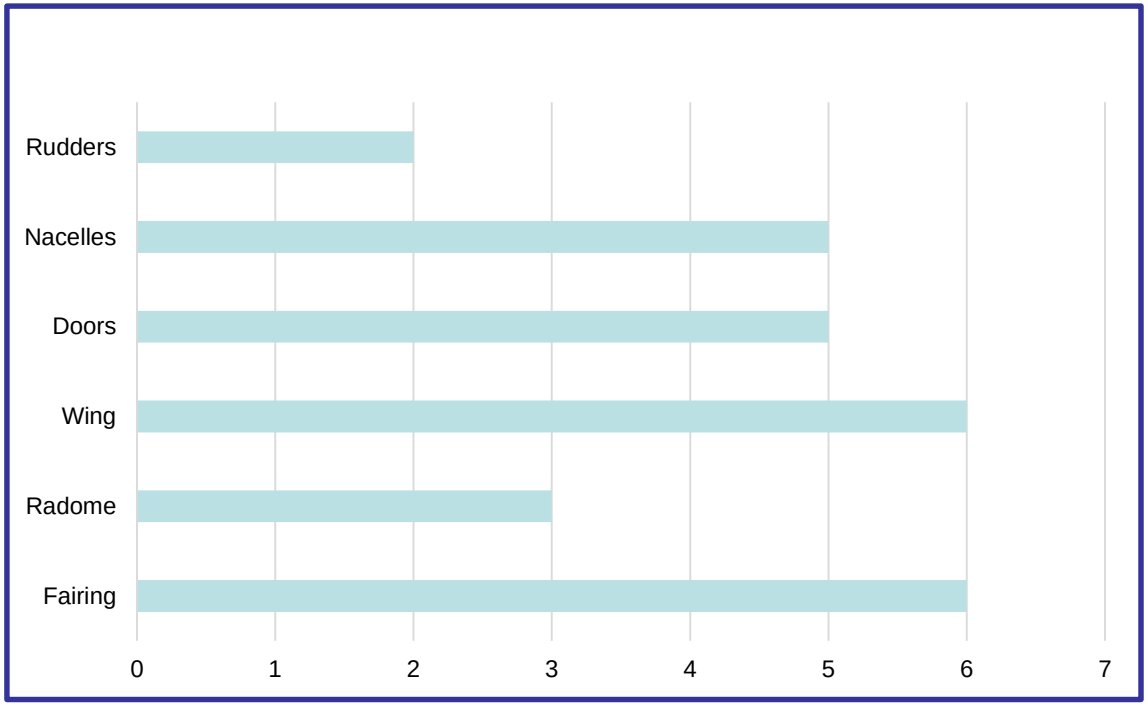


Organization



Familiar with CMH-17 and NCAMP

Parts Most Frequently Damaged



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Most Commonly Used Systems for Repair

- Most commonly used systems used for repair: include the parent materials used for the base structure, wet lay-up systems or out-of autoclave prepreg systems.

- **Parent materials**:
  - Mostly 350◦F carbon fiber/epoxy or fiberglass/epoxy prepreg,
  - Mostly fabric with some unidirectional tape: AS4/8552, AS4/3501-5, T300/934, T300/970, T300/F593, G30/F593, etc.
  - Fiberglass/epoxy prepregs are typically used for fairings, radomes and interior structures.

- **Repair Processes**:
  - Most of the repairs conducted are autoclave, oven or heat blanket cured.

Commonly Used Materials

- **Wet lay-up systems**:
  - Laminating resins LY5052, EA9396, Epocast 52 A/B, EA9390
  - Dry carbon fabric Plain weave 193 gsm, 5HS, 8HS.
  - Dry glass fabrics AMSC9084 grade III and grade VIII
  - Specifically: Epocast 52 A/B + dry carbon fabric G0904 (plain weave 193 gsm) (AMS2980 – CACRC qualified)

- **Prepregs and adhesive films**:
  - Cytec 5320-1 OOA
  - Hexply M20/40%/G904 + EA9695 05NW (AMS 3970 – CACRC qualified)
  - Hexply M20/40%/G904 + FM300-2M
  - Hexply M20/34%/134 or M20/34%/194 + FM300-2M (AMS6885 – CACRC qualification in progress)
  - Hexply F155/ 3K-70PW
  - Hexply F263/ 3K-70PW
  - Hexply F155/ 7781 Fiberglass Prepreg

Lessons Learned

- Preparation of bonding surface
- Removal of moisture
- Oil/contamination
- Insufficient drying of original parts
- Water tightness
- Control of thermal curing
- Repair inspectability after the repair application
- Importance of strict adherence to the SRM instructions and technician training to ensure quality repairs.
- Other challenges include repair material supply and storage, OEM approvals of repair materials for particular applications, and OEM proprietary test data

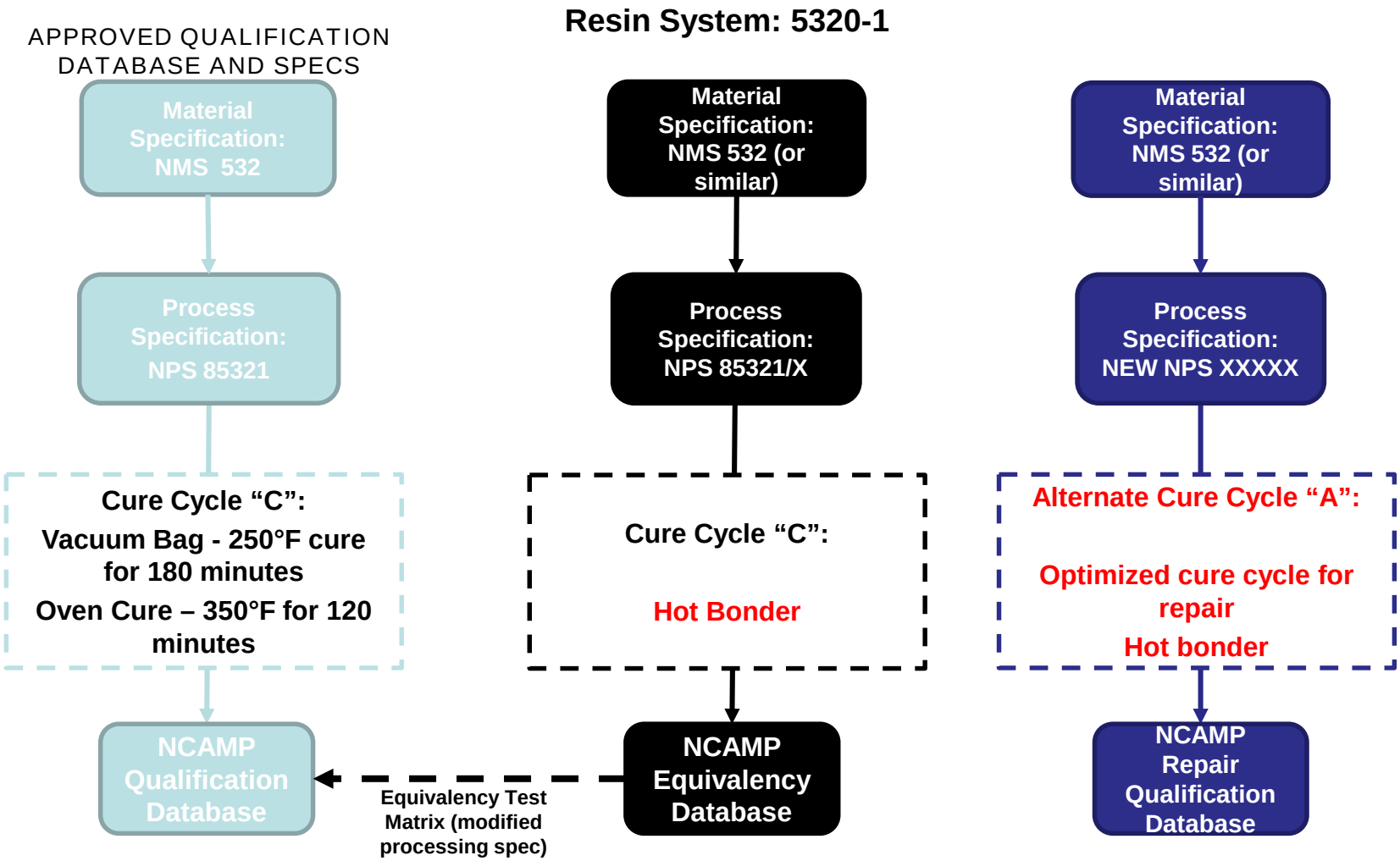
Industry Needs

- Results showed a strong need for the standardization of composite repair.
- The need for new materials, particularly lower temperature cure materials.
- Data
- Better documentation, training, and information to be included in the repair manuals.
- Several recommendations for improvements to existing repair procedures were suggested including:
  - More closely following current procedures, supplying build data, better NDT procedures and standards, better surface preparation and inspection procedures, more comprehensive process details, repair technology transfer, repairmen minimum training requirements and more extensive use of travelers.

Repair – Current Status

- Steering Committee
  - Established based on survey results
  - Monthly meetings began in January
- Material Partner
  - Solvay – 5320-1 / T650 PW
- Qualification Framework
  - Process Spec – initial draft complete
  - Material Spec – already approved, possible modifications
  - Test Plan – in development

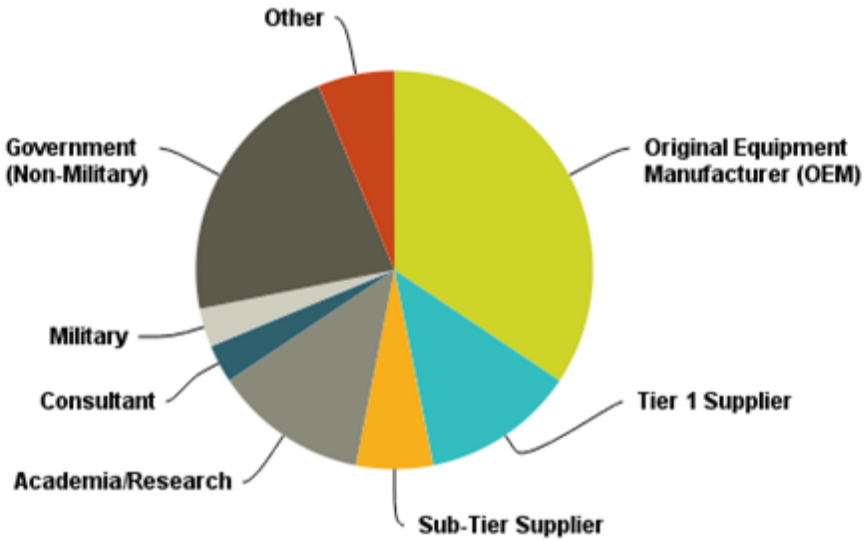
Current Approach



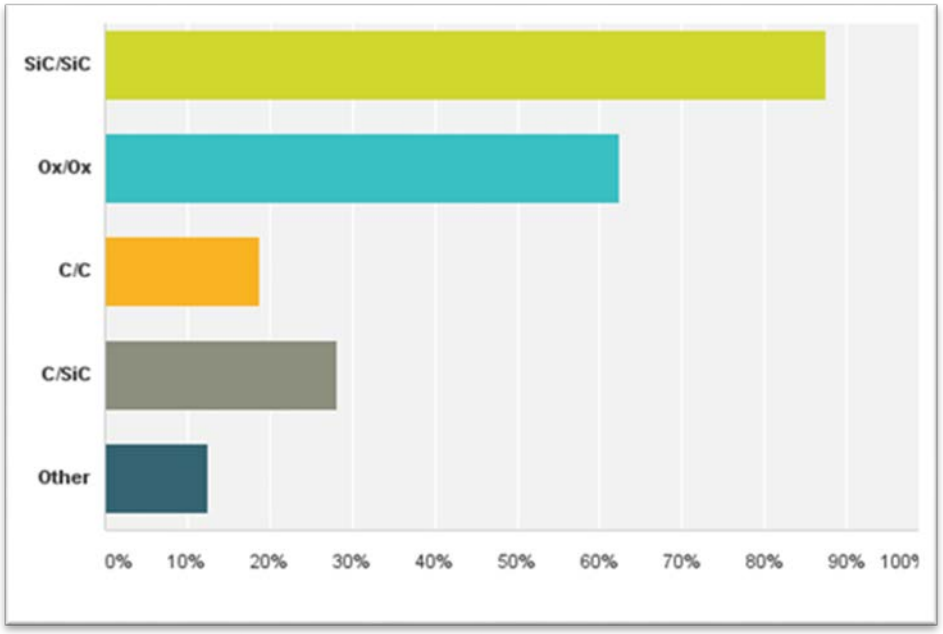
CERAMIC MATRIX COMPOSITE MATERIALS

Survey Results

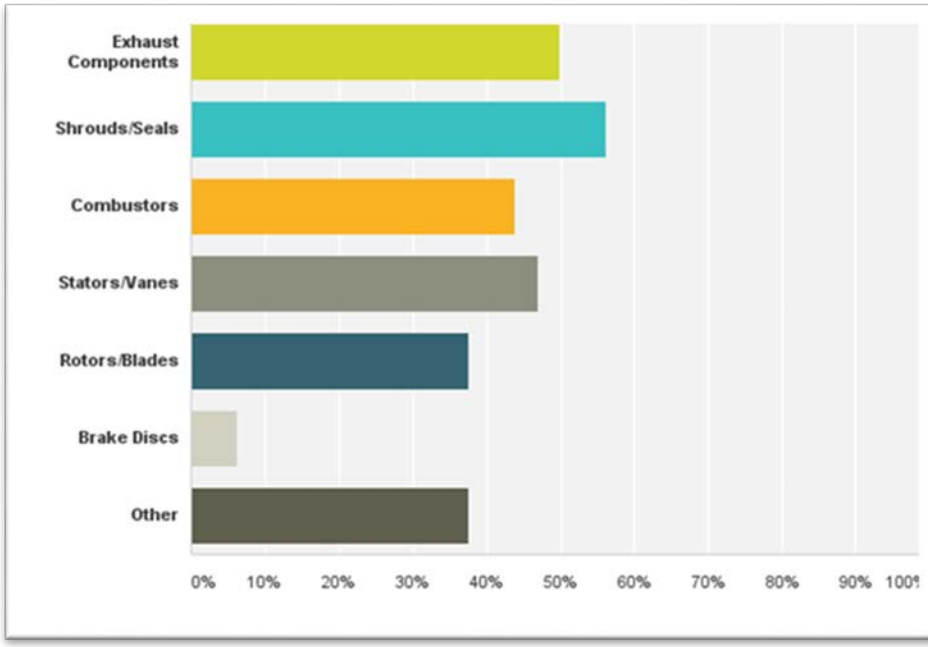
- **Objective**: Generate industry input on issues for qualification and material selection
- Administered by Survey Monkey (Oct – Nov 2016)
- **Responses**: 32 responses received (21%)



CMC Survey Results



CMC Materials Currently Used



Parts Made With CMC Materials

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Survey Responses

- Challenges related to CMC Materials
  - Highest rated: cost (4.38) and lack of design and certification guidelines (4.03)
  - Other noted challenges:
    - Reproducibility of parts with consistent properties
    - Stability of materials and processes over time and the cost to evaluate stability
    - Current state of CMC materials is such that only limited applications exist
    - Limited design database and lack of lifetime testing presents challenges to trustworthiness and safety of the design

CMC Materials – Current Status

- Steering Committee
  - Monthly meetings
  - Input on test matrix

- Coordination
  - CMH-17 CMC Coordination Group
  - USACA, ASME, AFRL, NASA inputs
  - ASTM C28

CMC Materials – Current Status

- **Material Partner**: Axiom
  - CMC Oxide/Oxide Prepreg – specific form TBD
  - Ox/Ox composites becoming mainstream material option for high temp composites
  - Cost reductions are required to keep market competitive with Ti and other high temp alloys
  - 3M – Nextel Ceramic Fibers
  - Axiom – prepreg developer
  - Composite Horizons Inc – parts designer

Figure 2: Typical OxOx CMC supply chain from fiber to component

SOURCE: Ox/Ox CMCs – Enabling Widespread Industry Adoption

CMC Materials – Current Status

- **Qualification Framework Documents**
  - Test Matrix
    - Initial draft reviewed
    - Input and comments
  - Test Plan being drafted

  - Test Matrix includes:
    - Composite Physical and Thermal Properties
    - Lamina Mechanical – Fabric
    - Laminate and Design Guidance
    - Advanced Design Guidance

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Composite Physical and Thermal Test Matrix

Property	Test Method	Min Replicates per Panel	Comments
NDT by Ultrasonic Through Transmission (C-Scan), Thermography, or Radiography	MIL-HDBK-787 MIL-HDBK-731 MIL-HDBK-733 CMH 17 V5 Sec. 3.7	1	NDT on flat cured/consolidated composite test panels. Chosen methodology should consider the CMC constituents being evaluated.
Cured/Consolidated Ply Thickness	ASTM D3171 (Method II)	Determined for all mechanical test specimens	To be used for determining normalized mechanical properties.
Fiber Volume, % by Volume	ASTM D3171 (Method II)	3	Requires knowledge of the fiber and cured/consolidated composite density and weight of the fiber in a single unconsolidated ply (fiber areal weight).
Matrix Volume, % by Volume	ASTM D3171 (Method II)	3	Assumes zero void content, which will lead to gross error due to the highly porous nature of CMCs; however, the equations can be modified using the actual void content found by optical microscopy. Requires knowledge of the fiber, matrix, and cured/consolidated composite density along with the void content and weight of the fiber in a single unconsolidated ply (fiber areal weight).
Cured Matrix Density	ASTM C373	3 (total per batch)	Density of monolithic ceramic processed with composite test panels.
Cured/Consolidated Composite Density	ASTM C373	3	Density of composite taken from composite test panels.
Void Content	Optical Microscopy CMH-17 V5 Sec. 3.6	3	Determined by optical microscopy of polished cross-section.
Specific Heat	ASTM E1269	3 (total per batch)	The temperature range of interest must be defined.
Thermal Conductivity (Diffusivity)	ASTM E1461	3 (total per batch)	Test temperatures must be defined. Directions need to be determined based on the thermo-physical differences/likeness between the fiber and the matrix as well as the fiber form (i.e. 1-D, 2-D weave, or 3-D weave). Directions to consider: in-plane (fiber direction) and out-of-plane (non-fiber direction).
Thermal Expansion	ASTM E228	3 (total per batch)	The temperature range of interest must be defined. Directions need to be determined based on the thermo-physical differences/likeness between the fiber and the matrix as well as the fiber form (i.e. 1-D, 2-D weave, or 3-D weave). Directions to consider: in-plane (fiber direction) and out-of-plane (non-fiber direction).
SEM	CMH-17 V5 Sec. 3.6	3 (total per batch)	Surface topography and composition.

Lamina Mechanical Test Matrix for 2-D and 3-D Fabrics

Layup	Test Type and Direction	Property	Test Method	Number of Batches x No. of Panels x No. of Specimens Test Temperature RTD	Number of Batches x No. of Panels x No. of Specimens Test Temperature ETD	Comments
[0]	Warp In-Plane Tension	Strength, Modulus, and Poisson's Ratio (RTD Only)	ASTM C1275 (RTD) ASTM C1359 (ETD)	3x2x3	3x2x3	A number of differently shaped specimens are discussed for various fiber forms and constituents. A contoured specimen is generally preferred, but the stress concentration at the radii and can be problematic for 1-D CMCs. A straight sided specimen may be more desirable for 1-D CMCs. Poisson's Ratio may be difficult to determine at high temperatures due to limitations of strain instrumentation.
[90]	Fill In-Plane Tension	Strength and Modulus	ASTM C1275 (RTD) ASTM C1359 (ETD)	3x2x3	3x2x3	A number of differently shaped specimens are discussed for various fiber forms and constituents. A contoured specimen is generally preferred, but the stress concentration at the radii and can be problematic for 1-D CMCs. A straight sided specimen may be more desirable for 1-D CMCs.
[0]	Warp In-Plane Compression	Strength and Modulus	ASTM C1358	3x2x3	3x2x3	A straight sided specimen is generally preferred, but a contoured specimen has been used successfully. For a straight sided specimen, consider using specimen detailed in SACMA SRM 1 with face supported fixture detailed in SACMA SRM1 or ASTM D695. For a contoured specimen, consider using specimen detailed in ASTM C1358 or ASTM D695 with face supported fixture detailed in SACMA SRM1 or ASTM D695. Back-to-back strain gages should be used on first two specimens from the RTD condition to assess bending (see note 2).
[90]	Fill In-Plane Compression	Strength and Modulus	ASTM C1358	3x2x3	3x2x3	A straight sided specimen is generally preferred, but a contoured specimen has been used successfully. For a straight sided specimen, consider using specimen detailed in SACMA SRM 1 with face supported fixture detailed in SACMA SRM1 or ASTM D695. For a contoured specimen, consider using specimen detailed in ASTM C1358 or ASTM D695 with face supported fixture detailed in SACMA SRM1 or ASTM D695. Back-to-back strain gages should be used on first two specimens from the RTD condition to assess bending (see note 2).
[+45/-45]	In-Plane Shear (+45/-45 Tension)	Strength and Modulus	ASTM D3518	3x2x3	3x2x3	A simple test method but poor for measuring ultimate shear strength because fibers align with length/tensile axis as the specimen is loaded. Offset strength, strength at defined strains, and modulus may be difficult to determine at high temperatures due to limitations of strain instrumentation.
[0]	In-Plane Shear (Iosipescu Shear)	Strength and Modulus	ASTM D5379	3x2x3		Provides the best shear response of the standardized methods but not suitable for high temperatures because fixture moves on greased rod/bearing. Can be used to supplement data obtained from ASTM D3518 since this data is obtained from a mixed stress state.
[0]	Interlaminar Shear (Double Notch Shear)	Strength	ASTM C1292 (RTD) ASTM C1425 (ETD)	3x2x3	3x2x3	The face supported fixture detailed in ASTM D695 is utilized to stabilize the specimen. Notched specimens are more difficult to machine and failures are sensitive to notch quality.
[0]	Interlaminar Shear (Short-Beam Strength)	Strength	ASTM D2344	3x2x3		ASTM D2234 is very simple and inexpensive, great quality control test, but the stress state is mixed. Can be used to supplement data obtained from ASTM C1292 since notched specimens are more difficult to machine and failures are sensitive to notch quality.

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Laminate and Design Guidance

Layup	Test Type and Direction	Property	Test Method	Number of Batches x No. of Panels x No. of Specimens — Test Temperature: RTD	Number of Batches x No. of Panels x No. of Specimens — Test Temperature: ETD	Comments
[0]	Flexure	Strength and Modulus	ASTM C1341	3x2x3	3x2x3	A good test for material development, quality control, and material flexural specifications. A number of factors lead to ambiguity in using flexure results for CMC material design data. Uni-axial tensile and compressive tests are recommended for material design data based on uniformly stressed test condition. Flexural stress is calculated using elastic beam theory with the assumption that the material is homogeneous and linearly elastic. This only holds true when the principal fiber direction is transverse to the length of the beam. Four-point loading geometries are preferred. Modulus may be difficult to determine at high temperatures due to limitations of strain/displacement instrumentation.
[0]	Trans-Thickness (Flatwise) Tension	Strength	C1468	3x2x3		May be difficult to obtain valid failure modes for 3-D woven fabrics. More test development is necessary for testing at high temperatures.
[+45/0/-45/90]	Unnotched In-Plane Tension	Strength and Modulus	ASTM C1275 (RTD) ASTM C1359 (ETD)	3x2x3	3x2x3	A number of differently shaped specimens are discussed for various fiber forms and constituents. A contoured specimen is generally preferred, but the stress concentration at the radii and can be problematic for 1-D CMCs. A straight sided specimen may be more desirable for 1-D CMCs.
[+45/0/-45/90]	Unnotched In-Plane Compression	Strength and Modulus	ASTM C1358	3x2x3	3x2x3	A straight sided specimen is generally preferred, but a contoured specimen has been used successfully. For a straight sided specimen, consider using specimen detailed in SACMA SRM 1 with face supported fixture detailed in SACMA SRM1 or ASTM D695. For a contoured specimen, consider using specimen detailed in ASTM C1358 or ASTM D695 with face supported fixture detailed in SACMA SRM1 or ASTM D695. Back-to-back strain gages should be used on first two specimens from the RTD condition to assess bending (see note 2).
[+45/0/-45/90]	Notched / Open-Hole Compression	Strength	ASTM D6484	3x2x3	3x2x3	Assumption is made that an open hole is critical for CMC notched compression testing.
[+45/0/-45/90]	Notched / Filled-Hole Tension	Strength	ASTM D6742	3x2x3	3x2x3	Assumption is made that a filled hole is critical for CMC notched tension testing, as opposed to a open hole due to fastener torque/preload.
[+45/0/-45/90]	Single Shear Bearing	Strength	ASTM D5961 (Procedure C)	3x2x3	3x2x3	Procedure C is for a single-piece configuration, fastened to a robust fixture.
[+45/0/-45/90]	Tension or Compression After Impact	Strength	ASTM D7136 & ASTM D5766 or ASTM D7137	3x2x3	3x2x3	Tension after impact strength may be more appropriate for CMCs than compression after impact due to the tension sensitive nature of CMCs and their specific applications. Consider using ASTM D5766 (open-hole tension) for guidance for performing the tension test. Modifications to ASTM D7136 may have to be made for CMCs (e.g. specimen geometry, impactor geometry, and impact energy).

Advanced Design Guidance for 2-D and 3-D Fabrics

Layup	Test Type and Direction	Property	Test Method	Number of Batches x No. of Panels x No. of Specimens Test Temperature RTD	Number of Batches x No. of Panels x No. of Specimens Test Temperature ETD	Comments
Any	Fracture Toughness		ASTM D5528 ASTM D6671 ASTM E1922	1x2x3		Considerations may need to be made by the end user for specific applications. There may not be a consensus for a standard approach. Probably outside of the scope of the effort.
Any	Creep		ASTM C1291 ASTM C1337		1x2x3	Considerations may need to be made by the end user for specific applications. There may not be a consensus for a standard approach. Probably outside of the scope of the effort.
Any	Fatigue, In-Plane Tension		ASTM C1360	1x2x9		Considerations may need to be made by the end user for specific applications. There may not be a consensus for a standard approach. Probably outside of the scope of the effort. If performed, would recommend 6 replicates at 3 stress levels.
Any	Fatigue, Thermal & Static, In-Plane Tension		-	1x2x3		Thermal fatigue of specimens followed by determination of static in-plane tensile strength. Considerations may need to be made by the end user for specific applications. There may not be a consensus for a standard approach. Probably outside of the scope of the effort.

POLYMER BASED ADDITIVE MANUFACTURED MATERIALS

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Project Overview

- **Initial meeting/workshop**: St. Paul – August 2016
  - Material for initial qualification was selected: ULTEM 9085
  - Discussion on specifications and overall plan
- **Collaboration** with America Makes/AFRL/rp+m program
  - Shared resources
  - Deliverables and overall reporting are not changing
- **Material Partner**: Stratasys - Certified ULTEM 9085
  - Polyetherimide high performance thermoplastic
  - Good smoke and toxicity rating
  - Ability to withstand high temperatures – common use in aerospace and automotive duct work
- Controlled builds with fixed process
- Integrated test matrix

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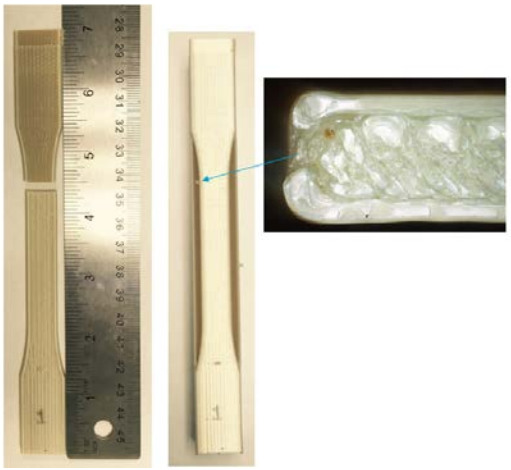
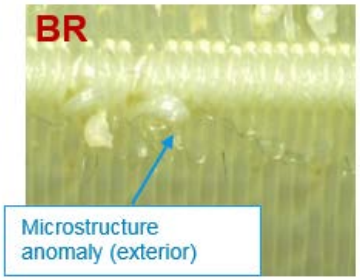
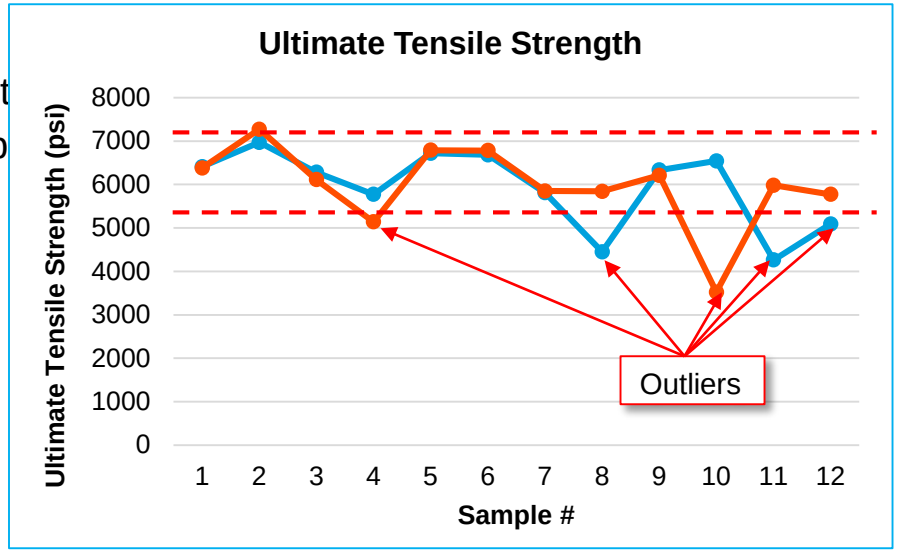
America Makes Program - High Performance Additive Manufactured Thermoplastics (HPAMT)

- Objective: Create dataset to expand and validate previous FDM data
- Enable industry members to use their own design allowable methodologies
- Led by RP+M (project management and build location)
  - Stratasys - certified material supply, material and process specs, printing location
  - Zodiak – printing location
- FDM Process Variability Analysis
  - Machine parameters
  - Failure analysis/identify data spread
  - Correlate data trends to machine and build paramet
  - Control data variability through machine and build p



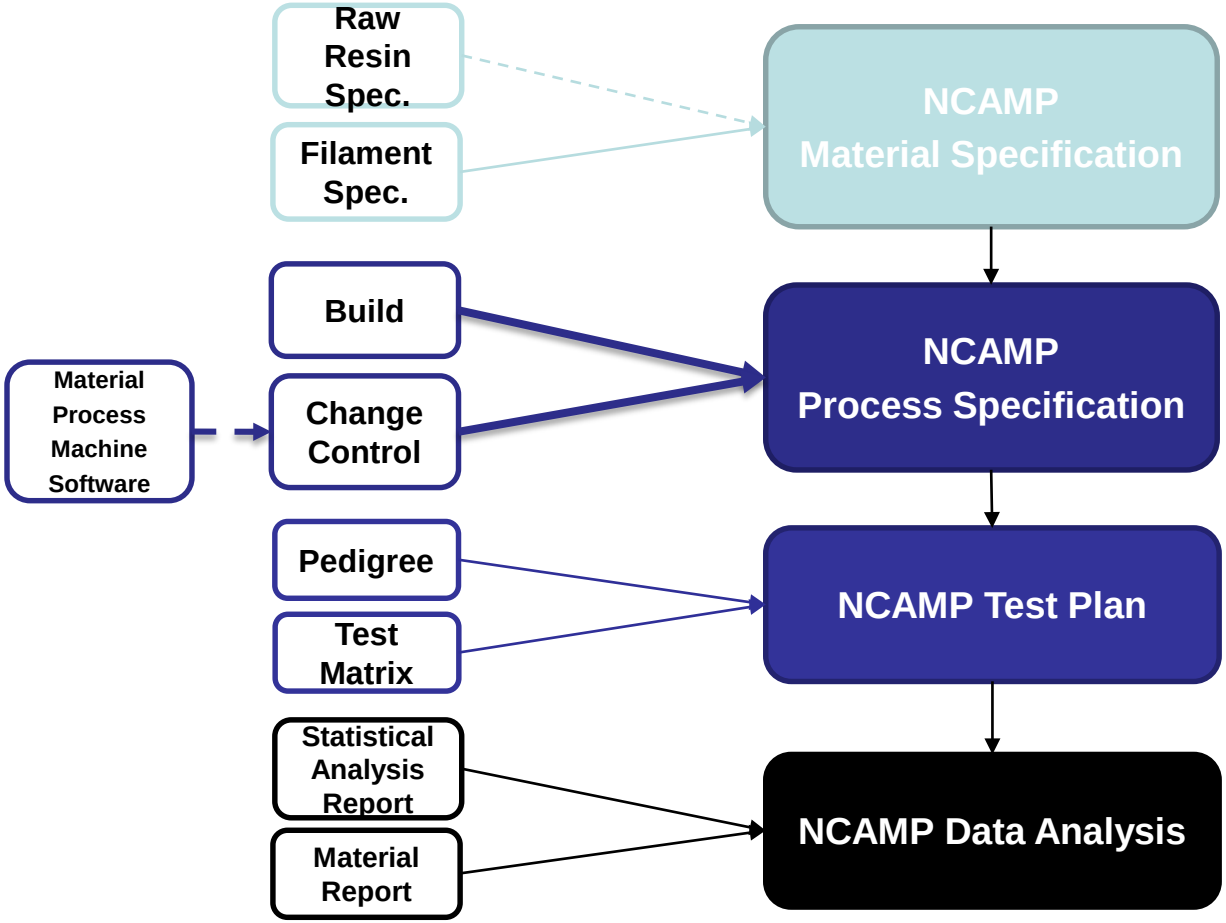




Current Steering Committee Activity

- **Test Matrix**
  - Qualification matrix reviewed in February
- **Test Plan**: Material Property Data Acquisition and Qualification Test Plan
  - Initial draft posted in December
  - Several comments received
  - Updated version posted last week
- **Other Upcoming Activity**:
  - Specification Templates
  - Equivalency Test Plan
  - Coordination with SAE AMS AM - Polymers

NCAMP DOCUMENTATION



STATUS

- Currently working with Stratasys to finalize Material and Process Specs (this week)
- Once content is stable, NCAMP templates to be developed

- Test Plan finalized, Equivalency plan being drafted
- Dimensional studies currently being conducted
- Site Inspections (Qualification) – week of March 27th
- Qualification builds to begin after inspections

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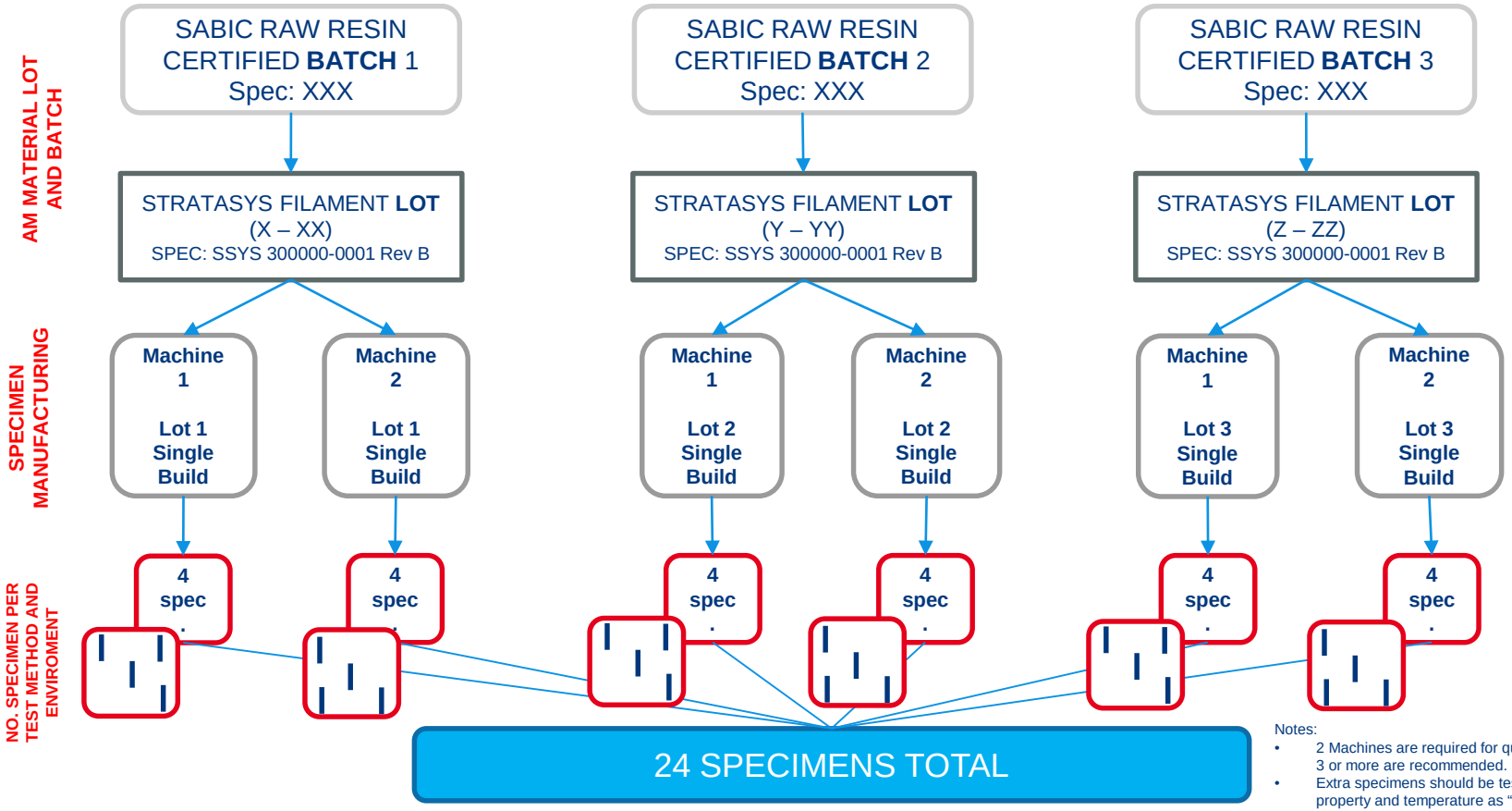
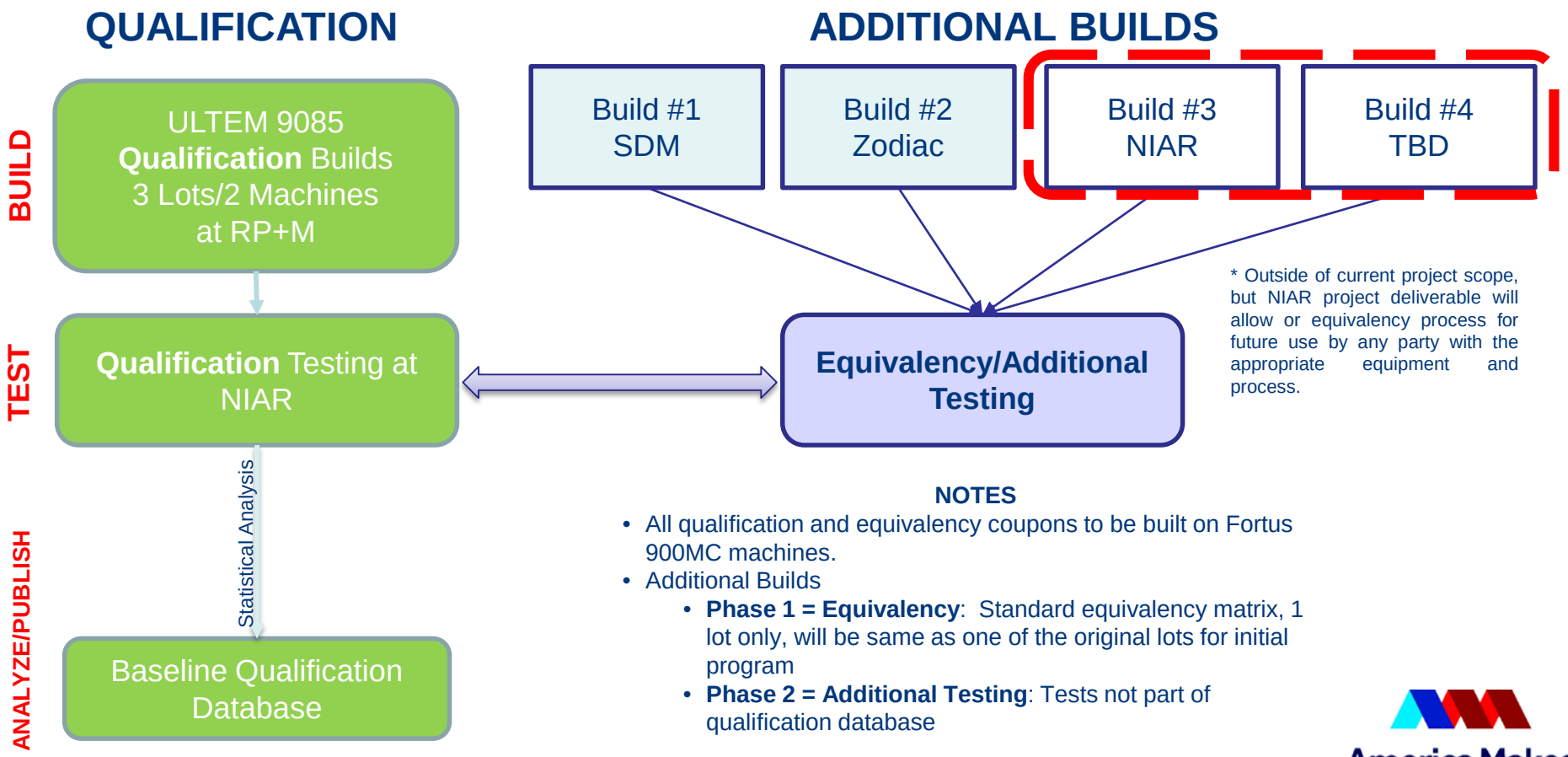


Notes:

- 2 Machines are required for qualification however 3 or more are recommended.
- Extra specimens should be tested for each property and temperature as “spares” to ensure desired quantity (min of 3 specimens).



NOTES

- All qualification and equivalency coupons to be built on Fortus 900MC machines.
- Additional Builds
  - **Phase 1 = Equivalency**: Standard equivalency matrix, 1 lot only, will be same as one of the original lots for initial program
  - **Phase 2 = Additional Testing**: Tests not part of qualification database

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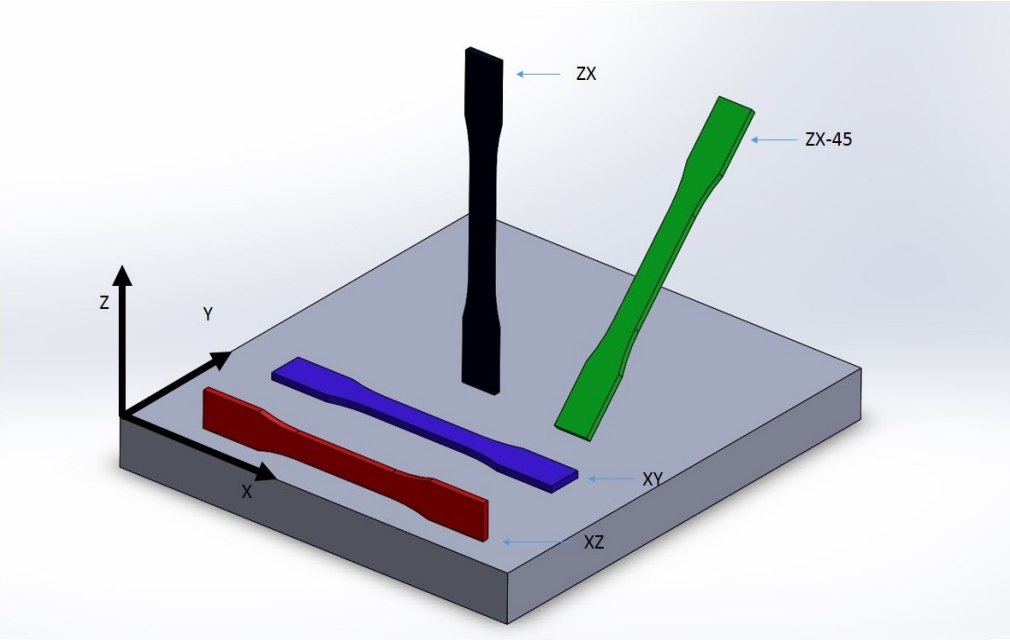
Build Orientation Investigation



ADHESIVE MATERIALS

Please contact Rachael if you are interested in more information:

rachael@niar.wichita.edu

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Questions?

Don’t forget to fill out the feedback form in your packet or online at www.surveymonkey.com/r/jamsfeedback

Thank you.

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