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Qualification of Additively Manufactured Material Extrusion Thermoplastic and Lessons Learned

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16. Abstract

Wichita State University (WSU) - National Institute for Aviation Research (NIAR) completed an initial additive manufacturing research project under the Federal Aviation Administration's (FAA) Joint Advanced Materials and Structures (JAMS) program between 2016 and 2019. The project focused on the development of a framework for the qualification of polymer-based additive manufactured (PBAM) materials including guidelines and recommendations for their characterization, testing, design and utilization. Through this initiative, a complete qualification of ULTEM 9085 printed on a Stratasys Fortus 900mc was created along with material and process specifications. This report documents the qualification program along with complementary research and lessons learned.

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Acronyms

Acronym	Definition
AFRL	Air Force Research Lab
AIR	Authorized Inspection Representative
AM	Additive Manufacturing
ASTM	American Society for Testing and Materials
CECAM	The Center for Excellence for Composites and Advanced Materials
CMH-17	Composite Materials Handbook-17
COE	Center of Excellence
COV	Coefficient of Variance
CTD	Cold Temperature Dry
ETW	Elevated Temperature Wet
FAA	Federal Aviation Administration
FDM	Fused Deposition Modeling
FHC	Filled-hole compression
FHT	Filled-hole Tension
FST	Flame Smoke Toxicity
GD&T	Geometric dimensioning and tolerances
JAMS	Joint Advanced Materials and Structures
LMCO	Lockheed Martin Company
MEX	Material Extrusion
MOU	Memorandum of Understanding
NASA	National Aeronautics and Space Administration
NCAMP	National Center for Advanced Material Performance
NIAR	National Institute for Aviation Research
OHC	Open-hole Compression
OHT	Open-hole tension
PBAM	Polymer-Based Additive Manufacturing
PBF	Powder Bed Fusion
PCD	Process control document
PMC	Polymer-matrix Composite
RP+M	Rapid Prototype and Manufacturing
SAE	Society of Automobile Engineers

SDM	Stratasys Direct Manufacturing
SDO	Standards Development Organization
SSB	Single Shear Bearing
SSYS	Stratasys
UTS	Ultimate Tensile Strength
VIPS	V-notch Iosipescu
WSU	Wichita State University

Executive summary

Wichita State University (WSU) - National Institute for Aviation Research (NIAR) completed an initial additive manufacturing research project under the Federal Aviation Administration's (FAA) Joint Advanced Materials and Structures (JAMS) program between 2016 and 2019. The project focused on the development of a framework for the qualification of polymer-based additive manufactured (PBAM) materials including guidelines and recommendations for their characterization, testing, design and utilization. Through this initiative, a complete qualification of ULTEM 9085 printed on a Stratasys Fortus 900mc was created, along with material and process specifications.

This qualification is a significant first step in safely qualifying PBAM parts by creating a baseline database and qualification framework that has been recognized by the additive manufacturing (AM) industry (both commercial and Department of Defense). The results of this research program address a key barrier to the adoption of PBAM technology. Based on the lessons learned from these activities, tasks were created and executed in subsequent years to address the gaps that still exist to further expand the framework. Continuing to update the framework will provide additional opportunities for polymer AM to be implemented within the aviation industry. The results of this work are currently being added as new content to the CMH-17 AM Volume, ASTM standards, and SAE specifications with the intent to further proliferate the learnings from the work performed by the Center of Excellence for Composites and Advanced Materials (CECAM) JAMS.

1 Introduction

The Center for Excellence for Composites and Advanced Materials (CECAM) at Wichita State University (WSU) - National Institute for Aviation Research (NIAR) is supporting the Federal Aviation Administration's (FAA) Joint Advanced Materials and Structures (JAMS) initiative through research aligned to certification efficiency goals and offering quality assurance guidelines specifically to the qualification of polymer-based additive manufacturing (PBAM). Local and national aviation leaders have expressed interest demonstrated by their level of investment in the development and implementation of additive manufacturing (AM) materials and technologies.

Regardless of research funding, the broad adoption of AM is still not realized due to a few key barriers. According to feedback and surveys conducted and compiled by Wohlers Associates (2021) and America Makes, the National Additive Manufacturing Innovation Institute (2018, June) reports that standards and qualification guidelines are two of the top reasons AM is not currently pervasively used. Some question if AM is disruptive enough to change how materials and processes can be qualified due to its inherent complexity. Others struggle to apply what they have seen done in previous qualification on either metals or composites to their AM materials, processes, and ultimately parts and designs.

AM is categorized as an advanced material/process because the material is being manufactured at the same time as the component. This differs from traditional materials because you cannot define the material without defining the process. It is well established that AM is a combination of material-driven variations as well as process intensive, making it more difficult to predict part performance. Having to account for increased levels of variability and the potential for anisotropic behaviors has left both applicants and certifying bodies with gaps in understanding AM behavior in both commercial and federal space.

CECAM is leveraging the knowledge residing within the National Center for Advanced Material Performance (NCAMP) to fill in the gaps in understanding for qualifying process intensive material systems such as additive, by creating a framework for AM initially focused on polymeric materials. The goal of the framework consisted of establishing qualification templates, performing a qualification of selected material, developing statistical guidelines, demonstrating equivalency on additional machines, and reporting on additional recommendations and guidelines. Follow-on research is ongoing and includes:

- Validating and expanding the framework through equivalency studies; and

- Supporting complementary initiatives building on the result of the framework creation, such as a new AM chapter in CMH-17, development and release Society of Automobile Engineers (SAE) specifications, and a drive to adopt newly developed characterization standards through American Society for Testing and Materials (ASTM).

2 Polymer-based additive manufacturing program history and overview

The focus of the work for PBAM on developing a qualification framework can be split into three main areas that are critical to the successful completion of a qualification program for AM materials. Figure 1 outlines the sub-elements that comprise those three focus areas.

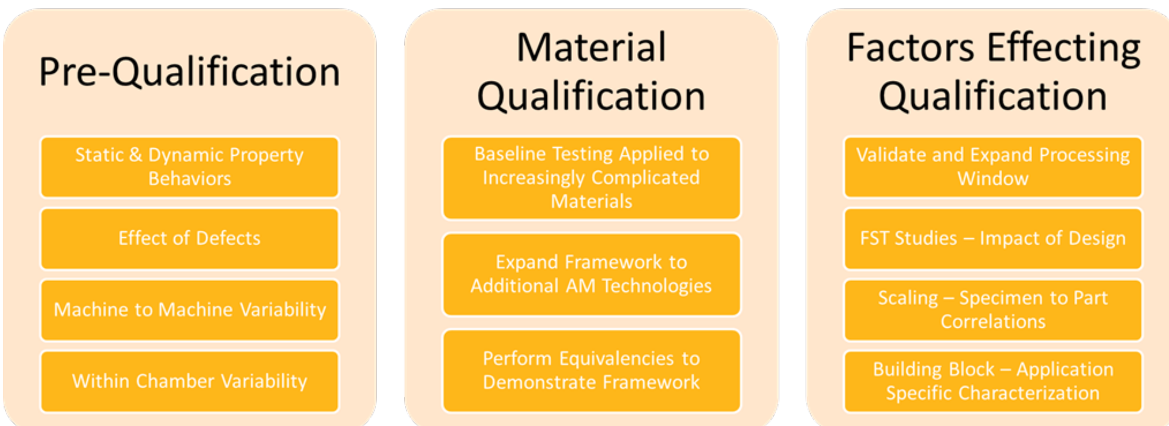

Figure 1. Gating and non-gating components of the PBAM qualification framework

The first focus area, “Pre-Qualification,” was developed to scope the expectations of what a stable and repeatable process should look like. Tasks performed at the beginning of the program, dating back to 2016, looked into static properties and how those could be applied to AM. The rest of the pre-qualification work was leveraged through a partnership with America Makes and Stratasys Inc (SSYS). An entire qualification program was attempted previously, only to show that across a fleet of machines, there was no consistency due to a lack of documented process control as well as a defect phenomenon that was undiagnosed and uncontrolled, leading to wide distributions in data. The first block shown in Figure 1 was meant as a gate to ensure that time and money from an applicant and test house are not wasted on a dataset that is unusable. Additionally, a set of documents and tools were created to ensure appropriate levels of data pedigree were captured up front to enable data analysis on the back end of the qualification.

The next area, “Material Qualification,” is the actual qualification of an AM material with a specific fabrication process. Having observed several attempts at qualifying a process intensive material from both composites and metals AM perspective, the team decided to limit the material, chosen by an industry steering committee, to be processed on a single version controlled and locked machine. The intent of the framework was to steadily increase in complexity and additional qualifications to demonstrate the versatility of the framework. Based on industry feedback, ULTEM 9085, produced by SABIC and sold by Stratasys, was selected and was to be run on a Stratasys Fortus 900mc machine.

ULTEM is a polyetherimide material and is amorphous by nature. It also is a commercially off-the-shelf material that for AM, is merely converted from one feedstock to another. This greatly simplified the requirements on the material specification side to control certain aspects of the filamentization process without having to worry about other feedstock additives or constituents. The framework can be expanded in the future to look at a more complex material like a reinforced/filled semi-crystalline material but on the same machine (Fortus 900mc). The introduction of a compounding step into the material supply chain was used as a stepping stone to eventually get to higher performance materials with higher criticality applications in mind.

The third focus area can be performed at any stage and does not need to be repeated for new materials on the same machine or similar technology. This area was important for understanding the application limitations of the material and process going through qualification. This focus area investigated the processing windows of AM machines that can produce a wide variety of geometries and parts in a single build. For the ULTEM qualification, over 300 inputs and variables were analyzed and documented to ensure a narrow, repeatable performance band could be achieved. Additionally, it was important to understand how the building block from specimen to final structure behaved in the AM space. It was determined that some properties were best used to demonstrate control and repeatability of the process but did not necessarily contribute to the creation of data that can be used as design values. In some instances, accurate property generation to allow for component level interpretation is still ongoing.

By following the three focus areas and the detailed sub-elements outlined, there is a higher level of confidence that any material and printer combination can be successful in conducting a qualification program. Additionally, improvements to testing methods, statistical methods, and printing approaches have all been documented and are being implemented by the appropriate standards development organizations (SDOs).

3 Qualification of ULTEM 9085/Fortus 900mc

The qualification of ULTEM 9085 was a multi-year, multi-task plan that encompassed the creation of the framework and ongoing tasks surrounding the learnings associated with performing the first PBAM qualification. The following sections outline the tasks that were accomplished throughout the qualification program.

3.1 Task 1: Establish industry/government steering committee

A one-day workshop was conducted on Wednesday, August 24, 2016 in conjunction with the CMH-17 Polymer Matrix Composite (PMC) Coordination meeting in St. Paul, Minnesota. The workshop included overview presentations, followed by discussions on the best path to developing a qualification methodology and initial database for PBAM materials. Near-term tasks to create templates for material and process specifications and a final test plan were completed. A steering committee was assembled to guide the development of the PBAM qualification framework based on guidance from the FAA as well as lessons learned from previous AM-specific qualification efforts. The steering committee was made up of workshop attendees and others who expressed interest but were not able to attend. A 30-member group including members from industry, government, and academia was created. One critical set of members for that group included America Makes (leading projects) and SAE (potential developer of specifications). At that time, America Makes was at the beginning of a Phase 2 project to characterize ULTEM 9085 with Air Force Research Laboratory (AFRL), Rapid Prototype and Manufacturing (RP+M), Stratasys, Inc. (SSYS), and Zodiak. At the workshop, the steering committee agreed to coordinate the two efforts to maximize the resources and data available. A memorandum of understanding (MOU) was established between the prime, RP+M, of the America Makes project, and NIAR, structuring a mechanism where the two project groups could work together. A partnership between SSYS, RP+M, and NIAR remains intact to ensure the successful continued execution of CECAM activities.

In addition to the combining of efforts for technical execution, this task also saw the creation and kickoff of an SAE AM polymer working group that agreed to use the material and process specifications created by the CECAM initiative as their first polymer AM publication. A NIAR representative was appointed cochair of this committee and has seen the development of four specification documents, two of which have been published (AMS AM Additive Manufacturing Non-Metallic, 2019; AMS AM Additive Manufacturing Non-Metallic, 2022).

Research relevant to this program was compiled based on applicability to the specifications, test plan (coupon and build matrices), and qualification procedures. PBAM research was discovered that had been conducted by NIST, America Makes, and various research institutions in the open literature. A separate area of the NCAMP portal was created for this effort (https://ncamp.niar.wichita.edu/) and limited to members of the steering committee to capture findings of the literature review. NIAR and the America Makes team engaged hundreds of industry and government experts to ensure the PBAM aerospace community remained involved and updated on the development of the methodology. Regular meetings with these communities of experts have been conducted dating back to November 2016.

3.2 Task 2: Development of qualification program

The steering committee selected ULTEM 9085 at the kickoff meeting due to its wide use in the aerospace industry and repeatable build results. ULTEM 9085 is a polyetherimide high-performance thermoplastic material with application acceptable strength-to-weight ratio and flame, smoke, and toxicity (FST) rating. This material is often used in aerospace, automotive, and other industrial applications where a high-strength, high-temperature thermoplastic material is needed. As this material is one of the only high-performance thermoplastic materials available for material extrusion, also known as fused deposition modeling (FDM), a complete database of material properties would further enable use in various commercial and government applications.

Using the PMC qualification documents as a template, material and process specifications were drafted in collaboration with SSYS. Figure 2 shows the documentation hierarchy that was employed based on the PMC approach. Draft specifications were created, extensively reviewed, and eventually approved by the steering committee. A test plan was also developed to include build and test matrices capable of generating adequate data to determine variability and effects of process parameters. The test plan included mechanical property testing bound by part-realistic applications.

Three certified lots were determined to be required to confidently assess the true variability of the feedstock material. A multi-month effort resulted in the completion of a material line audit and the procurement of multiple certified lots of material from SSYS. In addition to auditing material production, RP+M underwent a thorough NCAMP audit to validate their quality management systems and adherence to the process control documents and process specifications required to produce specimens. The use of an authorized inspection representative (AIR) per NCAMP protocols was leveraged and validated as a viable method for coupon inspection and conformity before shipment to NCAMP for testing. The audit determined that two of RP+M's Fortus 900mcs would be utilized for the qualification effort.

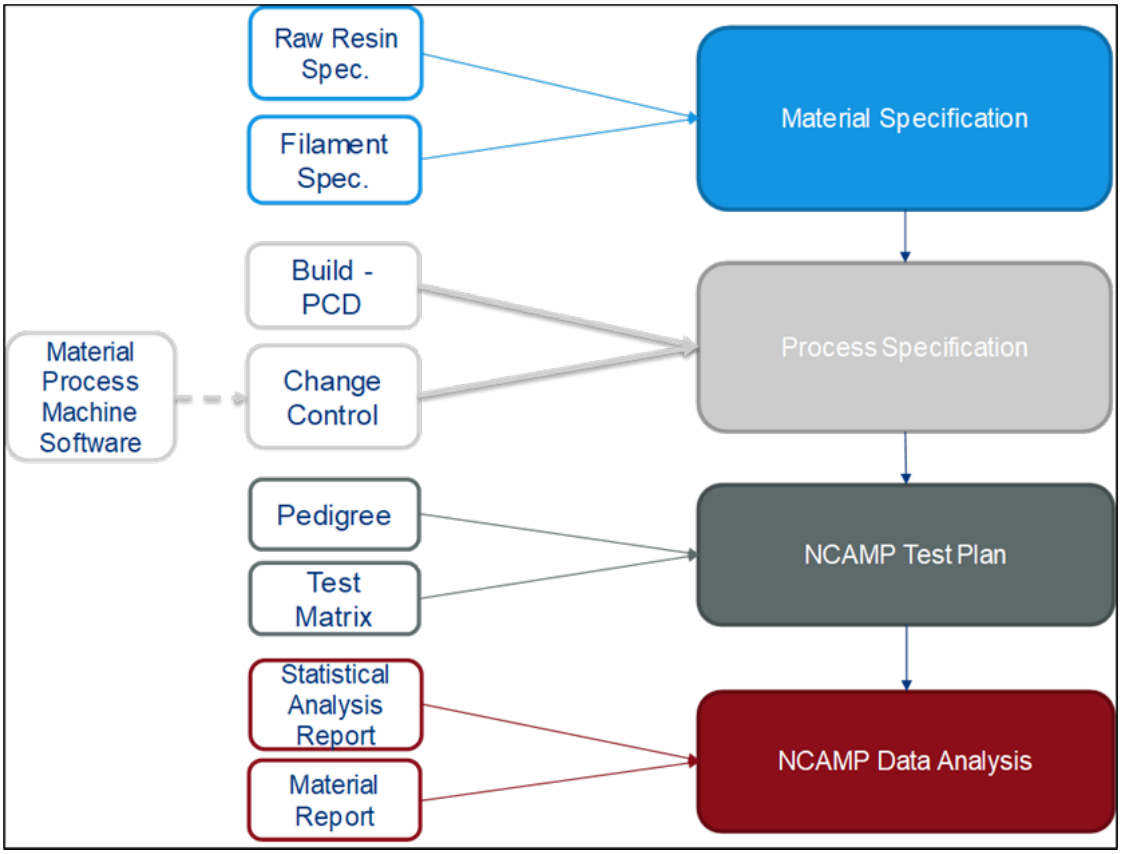

Figure 2. Documentation hierarchy employed based on the PMC approach

The full test plan can be found on the NCAMP PBAM portal (2019). Broadly speaking, the test plan documents all the data pedigree. Four orientations for specimen builds (XY, XZ, ZX, and ZX-45) were included in the test plan. The plan considered the different toolpath configurations that could exist within a part in order to capture the appropriate levels of isotropic behaviors of the material and process.

Specimen location was designated based on the thermal characteristics of the Fortus 900mc. The Fortus 900mc has a documented temperature gradient within the build chamber. Some early adopters limited their builds to only occur in certain quintant areas of the chamber based on their understanding of this gradient. By including all five areas of the chamber in the study, a comparison could be made to determine if the temperature gradient affected mechanical performance. Section 4 reports the results based on capturing this level of process nuances.

Combining all the build variables, a 3x2x4 matrix was created for each orientation and each environmental testing condition: three material lots, two machines, and four specimens. Figure 3 graphically depicts the test matrix methodology. The coupons were tested in accordance with the test matrix developed using current NCAMP protocols.

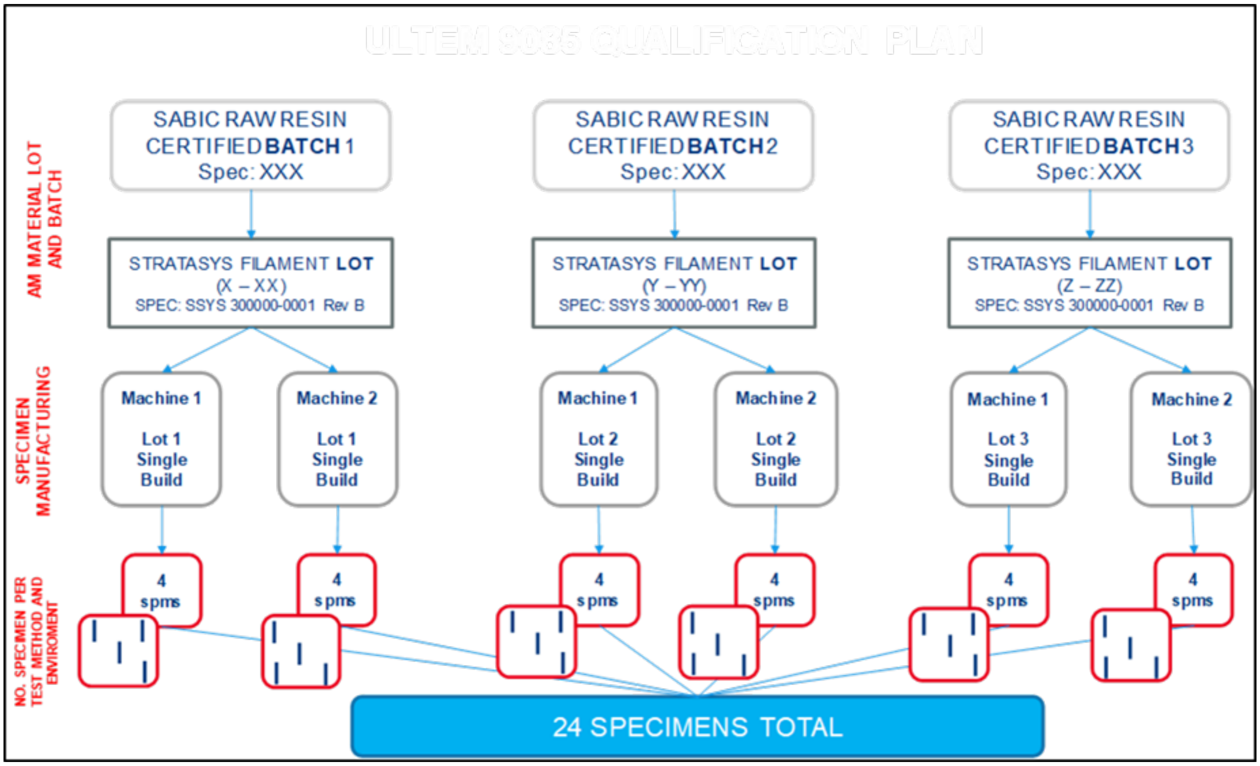

Figure 3. Test matrix methodology

3.3 Task 3: Perform equivalency on selected material

The qualification flow chart in Figure 4 shows the breakdown of qualification efforts as well as the demonstration of equivalency to validate the baseline database and show that other machines can produce specimens of statistical equivalence.

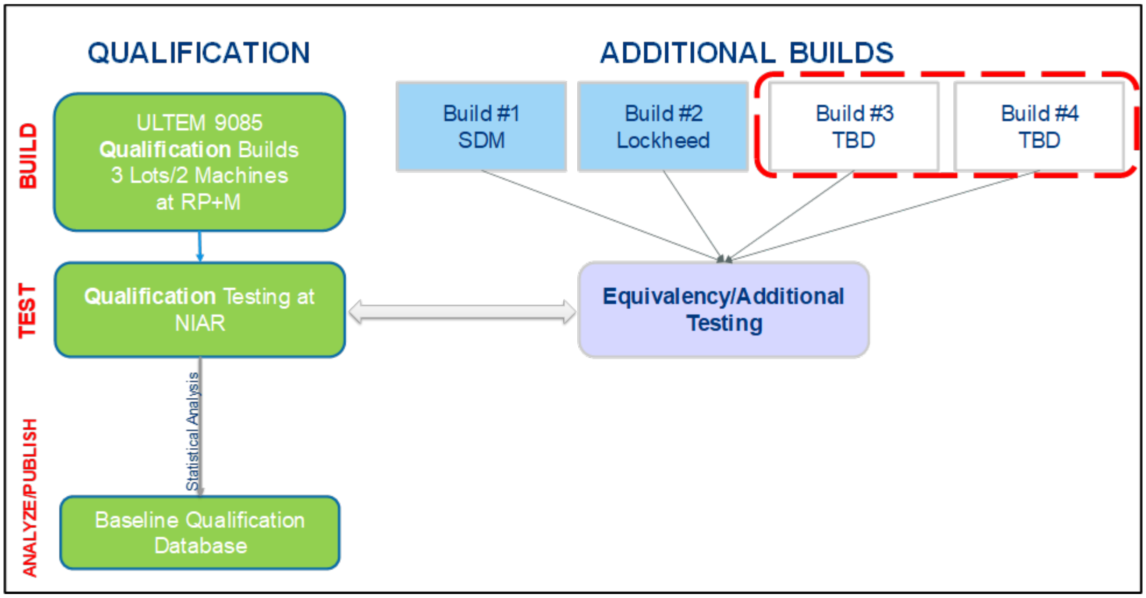

Figure 4. Qualification flow chart

Machines located at two different "super-users" of the technology conducted equivalency tests. The first was performed by Stratasys Direct Manufacturing (SDM), located in Belton, Texas, which is a service provider of parts to several commercial and defense aviation adopters. The second was conducted at Lockheed Martin (LMCO), based in Orlando, Florida. The equivalency test matrix followed a reduced set of test conditions, outlined in Table 1. Statistical comparison reports were created for each of the equivalency sites. The results of the two equivalency attempts came back with similar results, each passing around 73%, or 96 of the 132 test conditions. The ability to match open-holed compression data proved to be very difficult, with over one third of the failures coming from that test comparison alone; additional details for this test can be found in Section 4. As is standard practice, low performing specimens have been and will continue to be investigated to determine root cause sources. The latest analysis showed that for cold temperature dry (CTD) specimens, non-conformal breaks occurred in sections of the specimens that were driving either higher variation in the data or low performing specimens. These invalidating data points appeared to only occur on tension specimens but were consistent in both of the equivalency data sets. Other sources of low performance appeared to be moisture induced (elevated temperature wet; ETW). NIAR is working with SSYS to refine the drying procedures and methodology to better control this source of variation.

Further investigation between baseline, SDM, and LMCO confirmed that non-standard breaks were leading to lower performance in tension specimens for multiple geometries for CTD and ETW. Micrographs and surface analyses for all specimens produced in the baseline qualification and both equivalencies were captured, compiled, and correlated back to low and high performing specimens. Additional studies on sources and types of variability have resulted in findings that may alter how future qualification and equivalency programs should be structured, at least for material extrusion.

Fractography has been completed on 66 XY-oriented tension specimens. Figure 5 shows the typical cross-section of the +/-45º toolpath strategy. The surface looks clean with a consistent microstructure, and the failure is observed along the bead-to-bead interfaces.

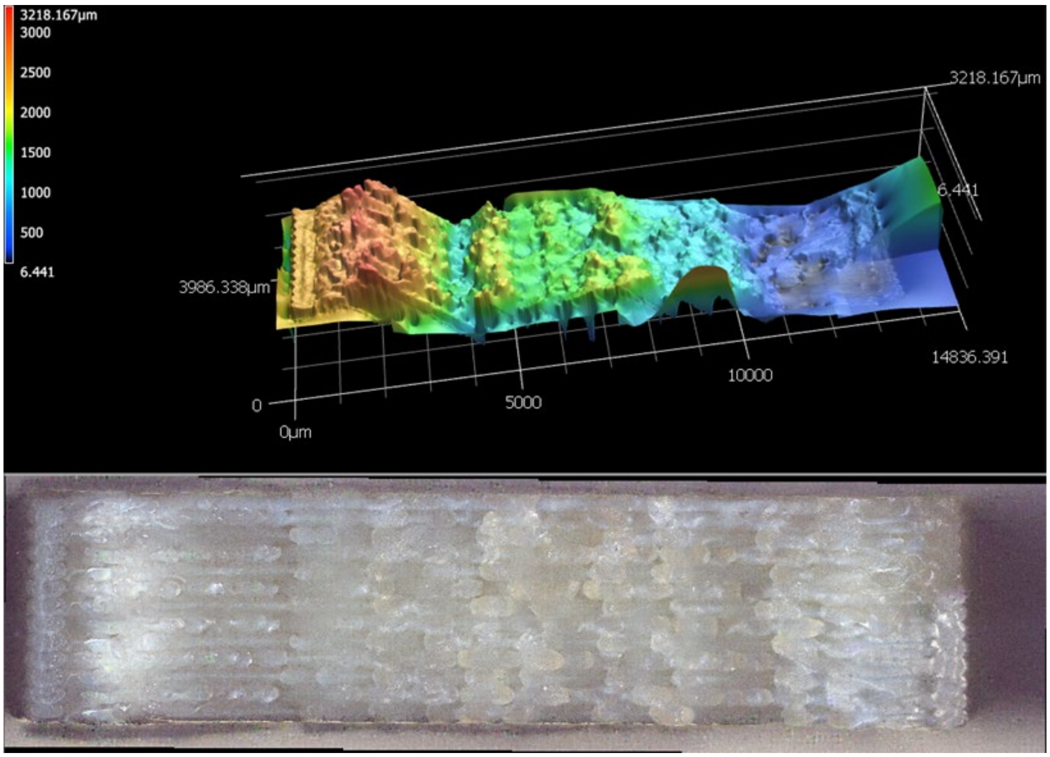

Figure 5. Fracture surface of an XY built specimen, tested for tension properties

In addition to tension specimen failures, the method of printing and introducing the hole in the open-hole and filled-hole specimens was evaluated. The material was produced as a solid block with different print strategies: aligned/stacked toolpaths at 0º and 90º rastering and offset toolpaths at 0º and 90º rastering. An example of 90º rastering can be seen in Figure 6. Specimens with these print strategies in all four orientations have been produced and are being prepped for testing to see if this is a valid alternative to testing, including open-hole compression (OHC) in future equivalencies and qualifications.

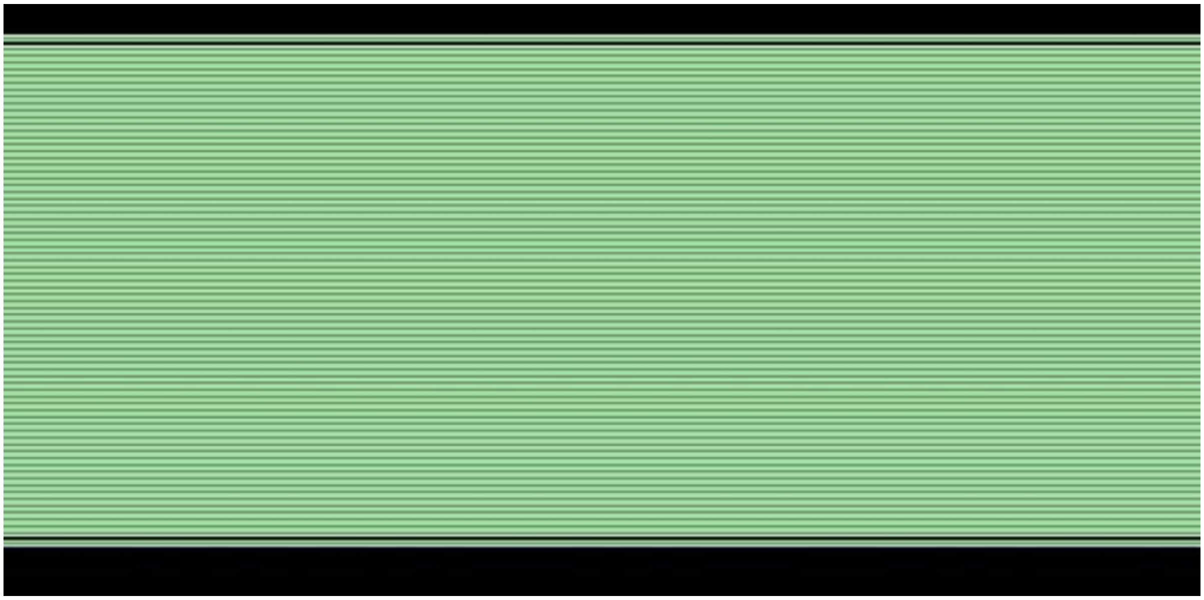

Figure 6. 90º rastering example

Open-hole and filled-hole compression (OHC, FHC) and tension (OHT, FHT) specimens were produced and tested for four orientations (XY, ZX, ZX, ZX45) with a machined hole introduced post-printing and following the four different “stack” or toolpath philosophies. Gaps in the toolpath planning led to a lack of adhesion between raster and contour beads (see Figure 7), suggesting that thickness needs to be adjusted to perfectly accommodate the theoretical thickness of the beads’ cross-sectional area per stacking strategy. Specimens produced with a 0º bead direction for the contours in the XY print orientation have produced failure results that are consistent with traditional composites. This provides high confidence that tailored build methods can produce repeatable results but without multiple orientations to account for different part configurations.

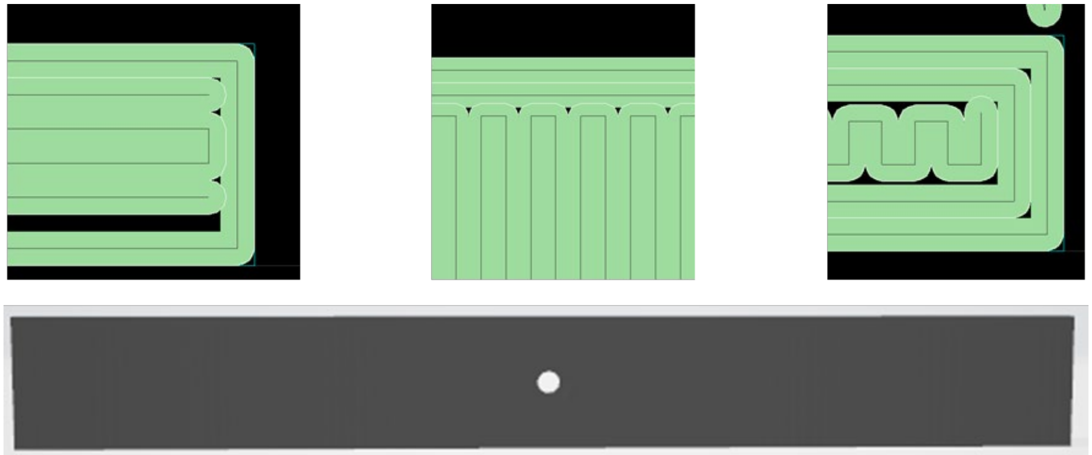

Figure 7. Gaps in toolpath plans lead to lack of adhesion between raster and contour beads

Complimentary work with NASA and ASTM provided input on this study that investigated converting this test into developing notch-like properties to help aid in the design and scalability studies. Since notched data has been proven to provide good representative design information as a means to account for unidentified/characterized defects, an alternative method is being explored by NIAR. Using a worse-case void or lack of fusion calculation, a controlled defect can be added to these specimens to best characterize knockdowns in performance to provide conservative design values for each orientation. Following this methodology, a toolpath strategy consistent with how the qualification and actual parts are produced will allow for more realistic data to be produced and provided to the design community.

Shear properties did not provide consistent results and required some preliminary experimentation. A V-notch iosipescu (VIPS) was tested, but appropriate failure mechanisms were only observed in the XY-orientation. As such, an alternative shear geometry (developed at NIAR) was investigated for shear failures for multiple print orientations. Figure 8 shows the new shear geometry that has been printed and analyzed. Like the VIPS specimen, the alternative shear geometry required some final machining to reach the dimensions required for repeatable characterization of this property.

Figure 8. Example of the alternative shear specimen geometry developed at NIAR

Testing of the alternative shear specimen showed that three of the four orientations provided good, consistent results with coefficient of variances (COVs) ranging from one to four percent. The on-edge (XZ-orientation) specimens had a much larger COV, but a limited sample size, and are being investigated further to understand why that orientation was problematic. Additionally, this alternative geometry provided challenges for obtaining strain data which may require another coupon configuration to obtain strain data or a custom gauge to extract strain data from the current specimen geometry. Both paths will be explored in future research.

3.4 Task 4: Development of statistical guidelines

Data generated from Task 2 were statistically evaluated utilizing CMH-17 STATS software (Composites Material Handbook-17, 2013). The researchers considered the process parameters including machine-to-machine, lot-to-lot, and build-to-build variability. The data generated and the resulting statistical analyses were compared to typical polymer matrix composite data trends as well as PBAM data from the Phase 1 America Makes program. A report summarizing the data, statistics, and comparisons has been completed and can be found on the NCAMP website (National Center for Advanced Materials Performance, n.d.). Equivalency analyses have been completed and reported in two separate equivalency reports as previously mentioned. Recommendations on statistical guidelines specific to FDM were included in the final statistical report. The recommendations include both qualification and equivalency guidance similar to those developed for polymer matrix composites (DOT/FAA/AR-03/19, 2003).

3.5 Task 5: Guidelines and recommendations

NIAR is working with several complementary SDOs and communities of excellence, such as CMH-17, SAE, and ASTM. Each organization is creating consistent documentation, specifications, or standards that were derived from the original qualification framework. This ensures that, regardless of the source, the same information will be provided, eliminating the risk of conflicting approaches or procedures.

3.5.1 Composite Materials Handbook-17 (CMH-17)

Data and guideline documents are currently being drafted for consideration in a chapter of the Composite Materials Handbook CMH-17 AM. The initial collaboration began at the August 2016 PBAM kickoff meeting. CMH-17 Working Group Chairs were informed of this effort. Formal collaboration, including content development, began during the fall 2018 PMC Coordination meeting as PBAM data became available. The data and guidelines developed have been refined and submitted to CMH-17 for publication consideration. NIAR has provided an assessment of the placement of the new data and guidelines sections within the handbook. CMH-17 has held several meetings where working groups created writing plans and content based on the results and learnings from the ULTEM qualification.

3.5.2 Society of Automobile Engineers

NIAR has assumed a leadership role as the cochair of the SAE AM non-metallic working group. The initial goal of the working group, formed in 2017, was to create four specifications. Of the specifications, two generic specifications were for AM material, and processes and two others detailed specifications (known as *slash sheets*) to encompass the work completed using ULTEM 9085 and the Fortus 900mc printer. New ballots for the SAE base specifications and data submission guidelines were issued to incorporate the comments from the committee.

3.5.3 American Society for Testing and Materials

In 2018, an AM center of excellence (COE) was created by ASTM to address gaps and issues related to current standards used to control and test AM technologies. NIAR became one of the first strategic partners of the COE and was made responsible for leading the Polymer AM working group and participating in the R&D and the Workforce Development and Education working groups. NIAR has continually supported ASTM Committee F42 on Additive Manufacturing Technologies and Committee D20 on Plastics in order to share lessons learned and address some of the shortcomings of test methods that are complicated by the additive process. NIAR has facilitated telecom meetings to complete specific work items that are expected to directly transition into ASTM standards. NIAR has investigated alternatives to ASTM D638 (2022) and D695 (2016) test methods looking at multiple materials that are commonly used in the MEX process with each test having several geometries: six for tension and four for compression.

The first ASTM work item executed in support of the ASTM COE investigated certain issues with tension and compression testing. Three criteria were applied to a round-robin study to determine alternative testing methods for tension and compression: printability, failure mechanisms, and variance (Figure 9). Printability assessed how difficult it was for a wide array of printers and materials to produce the specimen without failure. Failure mechanisms focused on the type of failures observed and the location of the failures. Traditional D638 specimen geometry often yields failures that break outside the gauge section and therefore invalidate the result. The third metric of interest explored the consistency of the data. Variability was measured as a coefficient of variance (COV) and remained the most difficult, since it is dependent on the printer itself to be repeatable and not skew the results of the study. In order to minimize the effect of printer variance, a control specimen, D638 Type 1, was added to the round-robin study. The test matrix diagram in Figure 10 shows the extent of the round-robin study for each geometry of interest.

	Geometry	Printability	Failure Mechanism	CoV
Polycarbonate	Double Flare, Sub Scale (DF)	●	●	◕
	Full size, thick grip (FT)	◕	TBD	◑
	Reduced Length (RL)	●	TBD	◔
	D638 Type 1 (T1)	●	TBD	◕
	Streamline Radius 2 (SR2)	◕	TBD	◔

	Geometry	Printability	Failure Mechanism	CoV
ULTEM 9085	Double Flare, Sub Scale (DF)	●	●	◑
	Full size, thick grip (FT)*	◑	◔	◕
	Reduced Length (RL)	●	●	◑
	D638 Type 1 (T1)*	◑	◕	●
	Streamline Radius 2 (SR2)*	◑	◑	◑

Figure 9. Round robin geometry types

With regard to printability, there were some issues with even the standard D638 Type 1 specimen's inability to print without failure or without much user modification/interaction. The printer used for this study showed a tendency to have the Z-45 specimens break or droop before completing the print. This showed that even the baseline testing standard had flaws in its configuration.

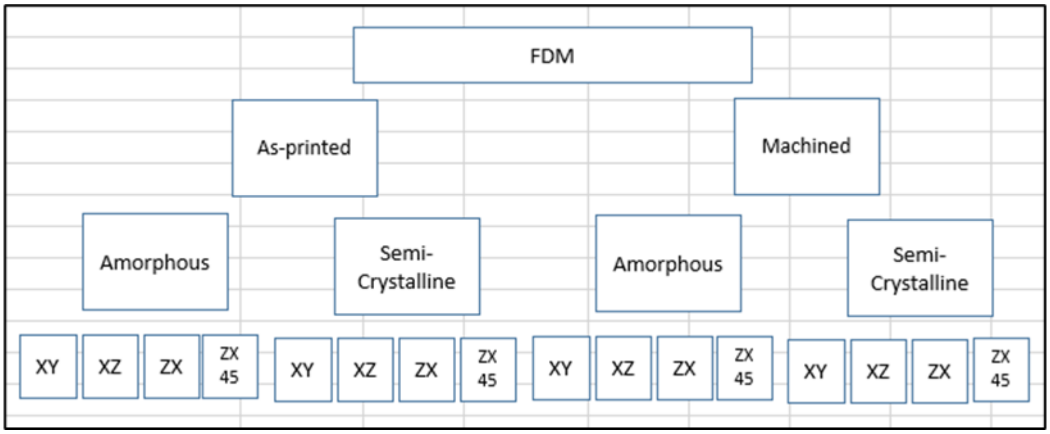

Figure 10. Test matrix diagram showing all variants to be considered for a new standard

The conclusions from the two tested materials and geometries are summarized in Figure 9. Overall, the results were fairly consistent, offering a high potential for a new geometry to be more reliably used for characterizing tensile strength for AM polymer materials.

Based on the current findings, the double flare specimen with an overall length of 50mm appears to be the likely candidate for further investigation and will be proposed as the geometry to evaluate other technologies and materials. Figure 11 shows a representative geometry of the double flare (DF) specimen. Experimentation will continue with modified grips and gripping techniques to finalize a guide for utilizing these alternative geometries for AM specific testing.

Figure 11. Double flare geometry

Data from the ULTEM study per orientation per geometry is shown in Figure 12. Absolute values per specimen were not of as much concern compared to the spread of the data. The spread (COV) from each of the specimens per geometry were fairly consistent with an average COV of 3% for both ultimate tensile strength (UTS) and 0.2% offset, with the highest COV being still below 8%.

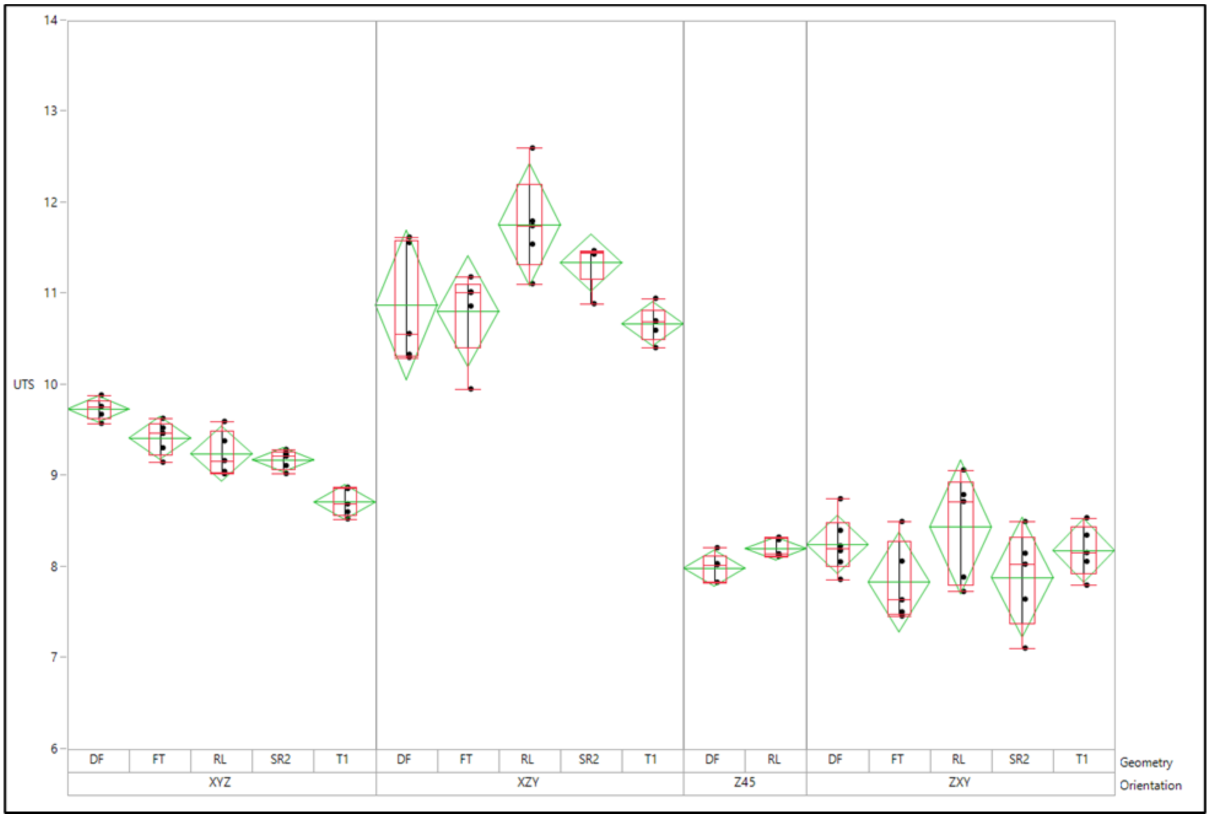

Figure 12. ULTEM study per orientation per alternative tension geometry

The last metric for evaluation of this study was the failure mechanism and break location. Figure 13 shows a Pareto of break location for each geometry. Unfortunately, every geometry had some breaks outside the gauge length or strain gauge section. However, two of the specimens had very promising results where minor modifications to the process should result in consistent breaks.

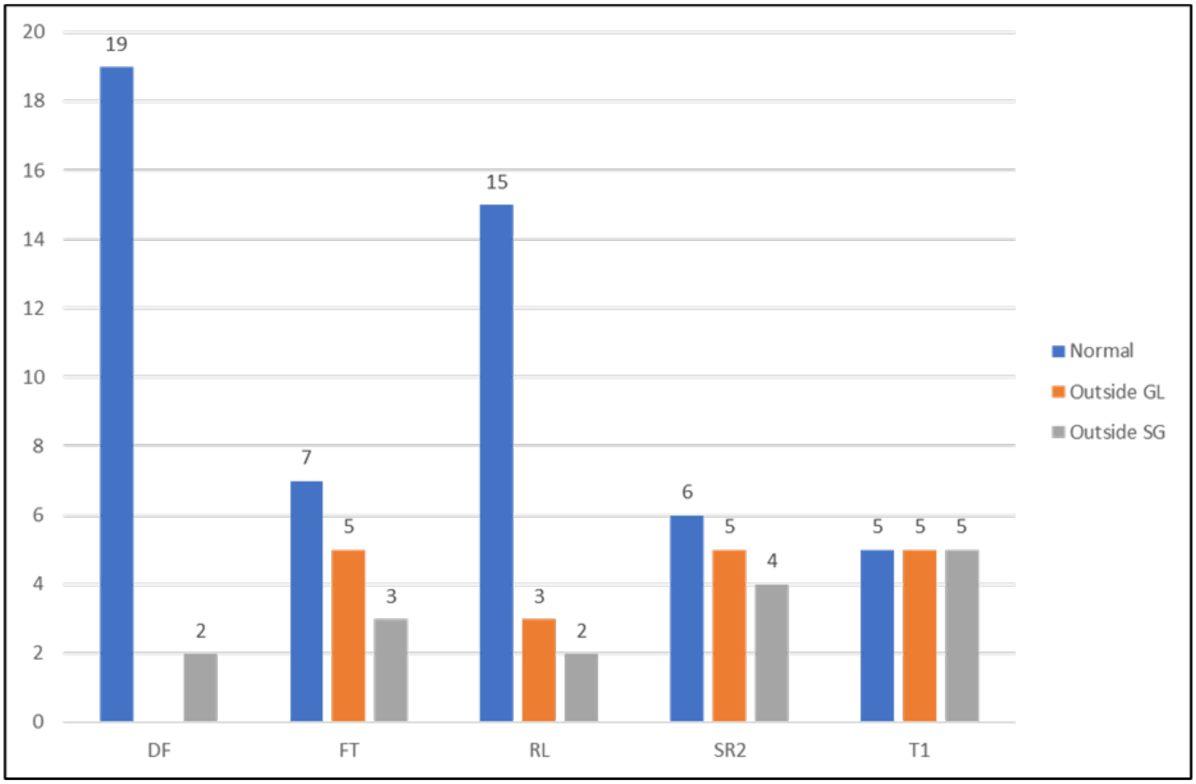

Figure 13. Failure mechanism and break location

4 Lessons learned

Throughout the execution of the qualification program tasks, the main focus was on creating a qualification framework for polymer AM with opportunities for expansion. There remain elements that require deeper attention and investigation, resulting in some key lessons learned. The first lesson discusses the methods of performing mechanical testing. The second outlines how the qualification was conducted and how to appropriately generate the data pedigree needed for post-qualification analysis. Lastly, there remain some challenges encountered during the first qualification and equivalencies that can serve as teaching opportunities to potentially eliminate issues in future AM material qualifications.

4.1 Characterization

The use of standards that have historically been used by either thermoplastic processes or composites remains the center of heated dialogue amongst industry leaders and experts. The ULTEM qualification effort highlighted some of the gaps that currently exist in testing standards when applied to AM. The intent of creating a framework was to document what the steering committee felt was the best combination of historical test methods, allowing only minor deviations and adjusting for future programs based on the results. Throughout the program, several decisions had to be made, most of which are outlined and detailed below.

4.1.1 Machining vs. as-printed

The first critical decision revolved around the final state in which the specimens would be tested. Based on other materials and processes, one line of thinking was that machining specimens would result in the best representative data. Individuals/experts in testing also agreed that machined specimens would result in more consistent data and eliminate testing setup variations when specimen geometries would meet specific geometric dimensioning and tolerances (GD&T) callouts from the standards governing the test methods. On the other side of the conversation, printing experts argued that most parts produced in a production environment go from chamber to vehicle without undergoing any machining steps. Therefore, testing a machined version does not accurately reflect reality. Additionally, the literature showed that the act of machining polymer AM parts more often than not introduced other defects on the surface resulting in arbitrarily low but tight data sets. Without a consistent method for machining specimens, it was determined that when specimens could remain in an as-printed state for testing, this would be the best course of action.

The as-printed approach held true for all specimens, with a few exceptions where an as-printed surface would result in the inability to conduct that test at all. Compression specimens were oversized and had their gripping ends machined to guarantee parallelism per the ASTM D695 standard (2016). Any specimens that required holes to be produced, such as open and closed hole tension and compression and single shear bearing, followed industry best practices. Namely, printing undersized holes that were then later reamed to ensure adherence to concentricity and location requirements that could have negatively influenced the testing of the specimen. Lastly, shear specimens that conformed to ASTM D5379 (2021) required the grinding of the notch to meet dimensional requirements of the notch radius as well as surface roughness requirements.

For future testing programs, especially those involving a MEX process, it is still recommended that nearly all specimens remain in the as-printed state. There are, however, exceptions to this. Any of the test specimens that will be used for notched data (OHT, FHT, OHC, FHC, VIPS, notched impact, etc.) should be printed as a solid component and have the notch introduced in its entirety after printing. For bearing studies, an undersized hole that is reamed to final dimensions is still a more accurate and realistic approach to these characterization methods.

4.1.2 Test methods

Of the ten mechanical test methods chosen for the qualification, 100% of them underwent some discussion and preliminary experimentation. This was done to ensure the test standard could be upheld and yield meaningful results that could be used to determine either process control or design values. Details for each chosen test method are provided below.

4.1.2.1 Tension

Before conducting the qualification, three different types and seven different geometries were compared to understand how different standards would dictate different behaviors and thus different data sets. The three types were categorized as: containing fillets, containing tabs, or flat specimens. ASTM D638 (2022) type 1, type 2, and type 4 were evaluated under the fillets category. ASTM D3039 (2017) printed with 0º and 90º rasters and as-printed tabs were evaluated under the tabbed category. Lastly, specimens printed flat with 0º and 90º raster orientations were evaluated for the flat category. Figure 14 shows the different types and break locations associated with stabilizer walls that are required for the fabrication of ZX-orientation specimens.

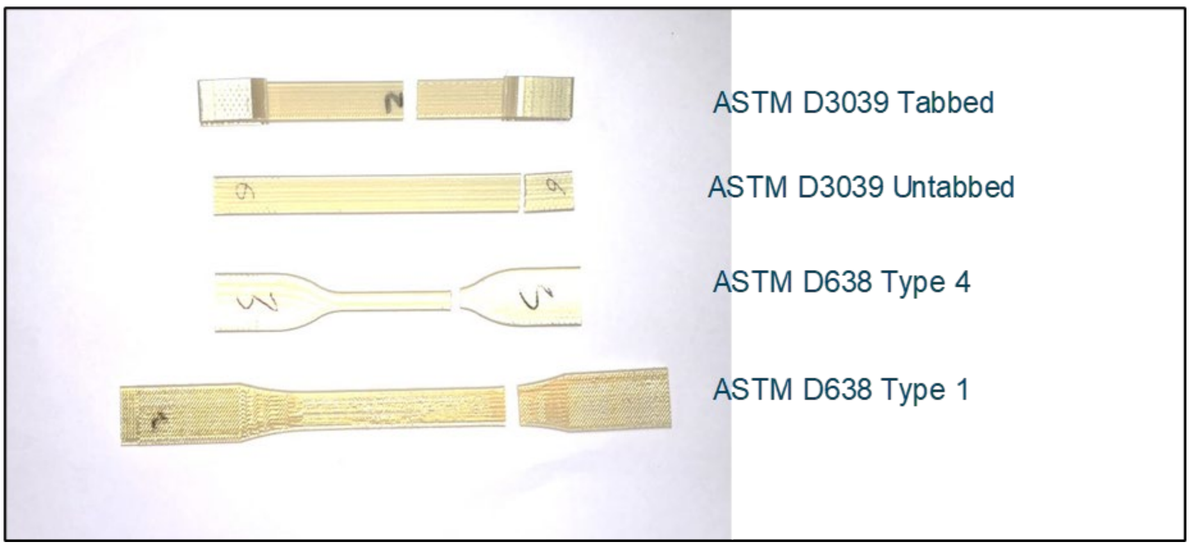

Figure 14. Different ASTM methods and geometry types evaluated

In addition to test types, a method for supporting certain specimens was also a critical decision point. Two different stabilizer wall methods were employed to ensure that printability could occur without introducing defects or flaws. Figure 15 shows the methods that were compared since certain geometries require either support or stabilizer walls to be printed.

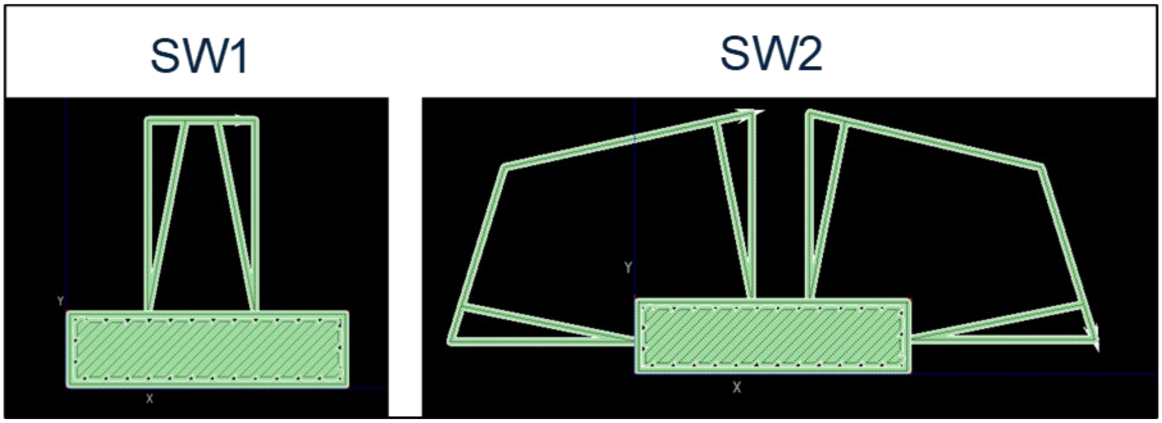

Figure 15. Two different approaches to stabilizer walls

In order to conduct an accurate comparison, it was determined that specimens from each type and category would be printed in a single pack in the center of the build chamber. Figure 16 shows the print layout of specimens in packs of four to eliminate all other sources of variability and determine which test method was most reliable.

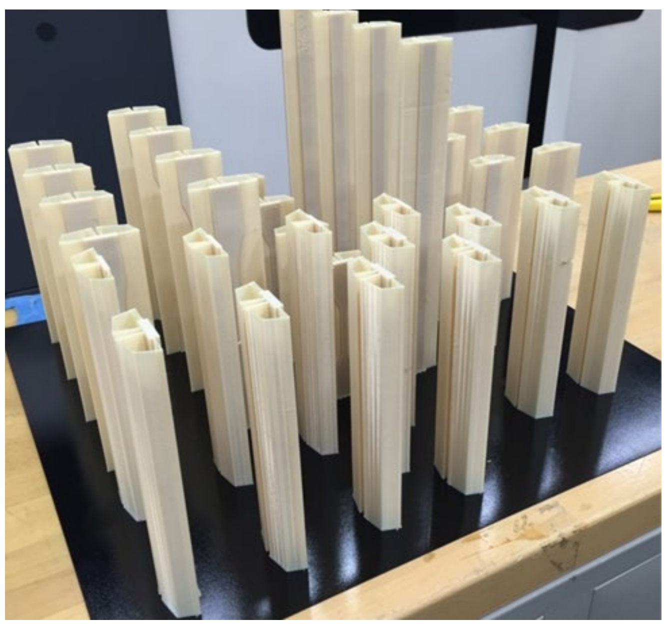

Figure 16. ZX specimens print layout for comparison

Figure 17 shows the results of the side-by-side specimen comparison.

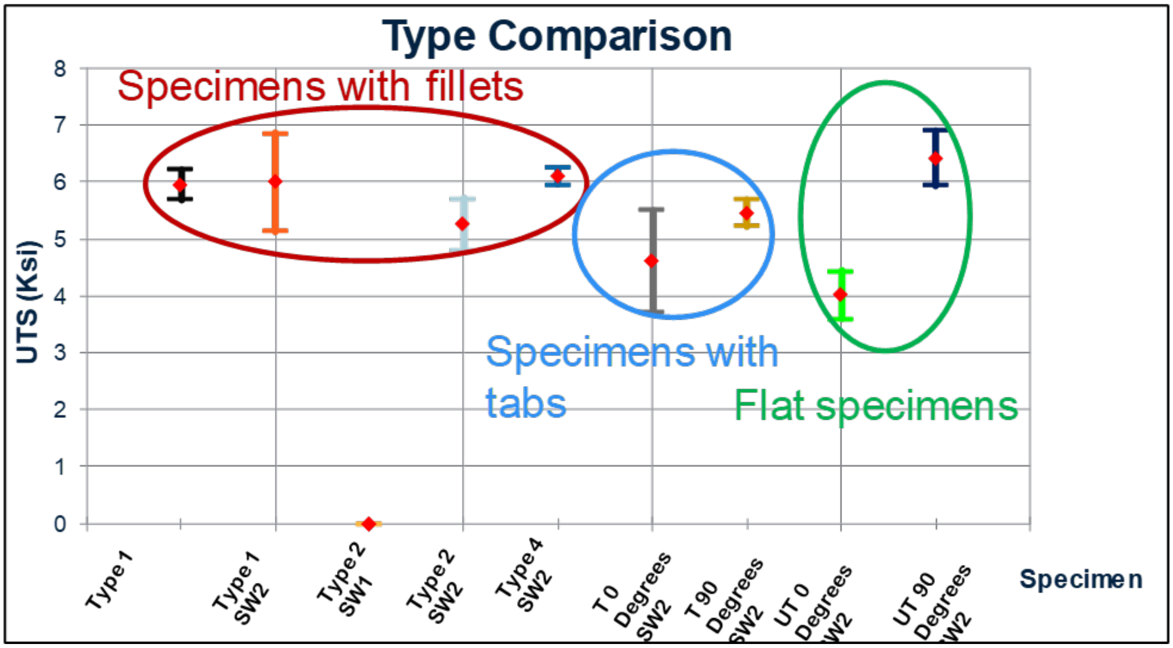

Figure 17. Comparison of geometries and methods used to capture tension strength

After several iterations of this study, it was concluded that ASTM D638, Type 1 would be leveraged based on printability, consistency of test results, and appropriate failure mechanisms realized. However, the 0º and 90º study for flat specimens should be revisited for other directional bead evaluation design values.

Reported values for tension consisted of ultimate tensile strength, 0.2% offset tensile strength, and tensile modulus. It is important to note that the offset tension strength values provided in the NIAR reports are calculated at the 0.2% offset strain value. At room temperature, this level of strain was indicative of the yield point of the tensile specimens. This strain-yield relationship was assumed to remain consistent across all other temperatures. It is also worth noting that due to the bulge created in the AM process, which is a result of the inherent circular shape of a bead, sharp corners are extremely difficult to produce in the MEX process without post-machining as shown in Figure 18.

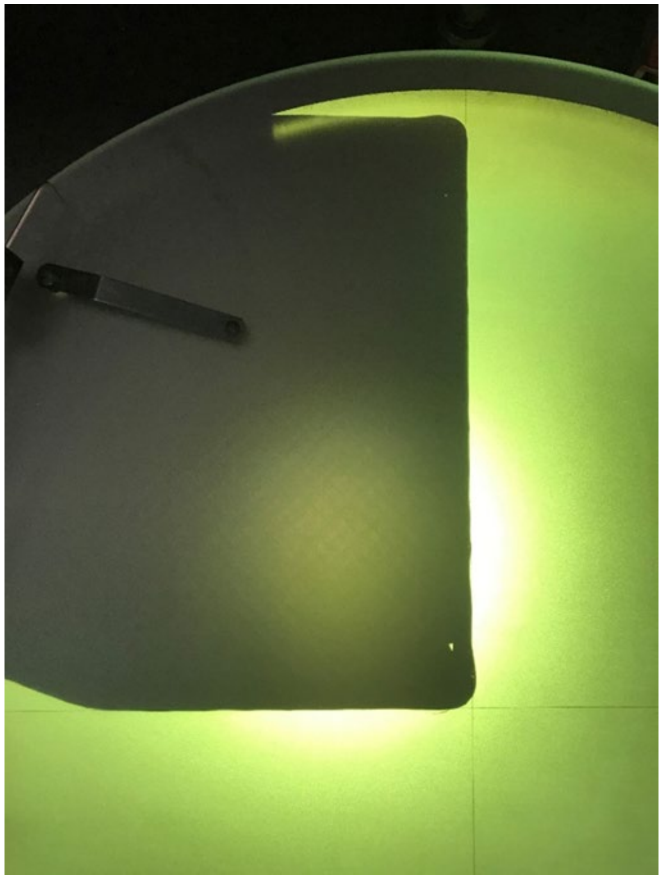

Figure 18. Example of the bulges witnessed at square corners

4.1.2.2 Compression

ASTM D695 modified (2016) was used to execute the compression configuration testing. Extracted data includes yield compression strength (strength offset values at 0.2% and 1.0% are reported with strain ranges of 2000με-6000με) and compression modulus. D695 contains two additional geometries that are commonly used to characterize AM materials known as cylinder and prism. Both cylinder and prism geometries utilize different aspect ratios that can be either 2:1 or 4:1; in most instances, they are used to determine different properties. It was determined and validated by the technical steering committee that using a modified dog-bone geometry would allow for both compression strength and modulus to be collected utilizing two-sided extensometers versus having to produce two different aspect ratios of a prism or cylinder to get the same properties. The steering committee expressed interest and placed value on getting modulus and compression strength from a single specimen in this proposed manner. Ultimate compression strength was not reported because specimens tested per ASTM D695 modified type 6.7.2 (2016) exhibited a buckling effect before reaching an appropriate failure mode.

In order to get consistent test results utilizing the modified dog-bone geometry, fixture modifications were performed as seen in Figure 19.

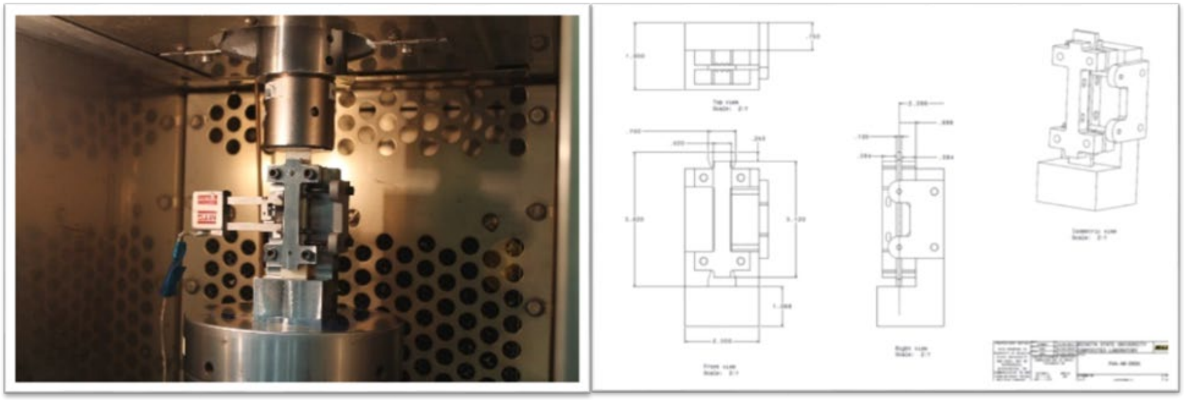

Figure 19. Modifications to the fixtures required to test compression specimens

4.1.2.3 Shear

Multiple shear specimens and methods were evaluated similarly to what was done with the tension testing. In a previous effort, different bead orientations were studied versus different build orientations to determine if appropriate shear failure modes could be observed. Based on that study, it was determined that only specimens printed in the XY-orientation would exhibit appropriate failure modes when produced with a +/-45^{o} toolpath strategy applied. Prior to the qualification effort, NIAR looked at three leading testing methods that were evaluated prior to performing a full test using ASTM D5379 (2021). ISO 15310 (International Organization for Standardization, 1999) did exhibit appropriate failure modes when a pseudo-isotropic ply approach was applied; however, strain gauges were not able to be attached in order to extract modulus results. Figure 20 shows a specimen being tested per ISO 15310:1999. ASTM D2344 (2022) SBS did not fail in a shear manner for any of the orientations evaluated and was determined to not be an appropriate test for this material and process.

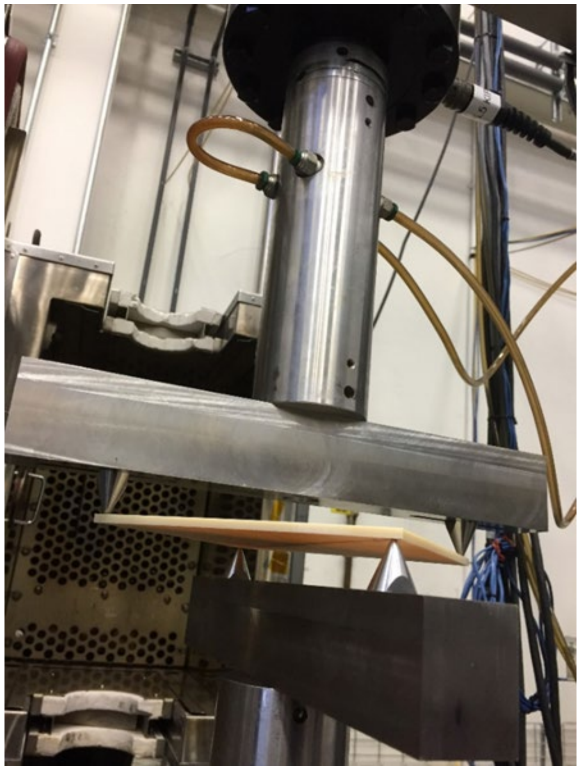

Figure 20. A new ISO standard to test for shear in composites and AM materials

Extracted data for the data report included shear strength and shear modulus (values are reported with strain ranges of 2000με-6000με). As described above, only the XY orientation exhibited applicable behavior to characterize the shear of this material and process. In order to meet GD&T requirements per ASTM, a flat panel was built, and the notches were added in a post-process step.

4.1.2.4 Flex

ASTM D790 (2017) was used to execute the flexure configuration testing. It was determined that all specimens tested observed invalid failures as per ASTM D790, note 18. Extracted data prepared in the data reports were 0.2% offset flexure strength and flexure modulus as a result of the load cell bottoming out. Even though ultimate flexural strength values were not reported, NIAR believes that flex is still a good test method for evaluating the repeatability of an AM system as well as offering valuable design data for part consideration.

4.1.2.5 Open-hole and filled-hole (notched)

Four different test methods were introduced to the test matrix to determine if there was a way to establish a set of design values related to notched knockdowns that could account for printing flaws that may not have been captured in standard tension and compression testing or that could account in any differences in scaling factors in parts that are printed versus specimens tested.

Following the PMC methodology, ASTM D5766 (2018), D6742 (2017), and D6484 (2020) standards were offered up to generate this valuable set of data. It was extensively debated how the hole should be introduced, which resulted in the fabrication of an undersized hole with multiple contours and a printed material plug that was then machined to the final dimensions. The material plug allowed for a center point to be established to improve the repeatability of the machining step, as seen in Figure 21 (left). When capable, multiple contours were utilized so that when machining would occur, the machine bit would not break through, exposing a raster and introducing defects.

In this case, the introduction of the hole, even as a pilot in the as-printed state, needs to be reassessed. Several holes after reaming resulted in defects as seen in Figure 21 (right). The specimens that contained these hole defects lead to easily predicted failures around the holes. Figure 22 shows an example of a failure along a toolpath that started at a defect.

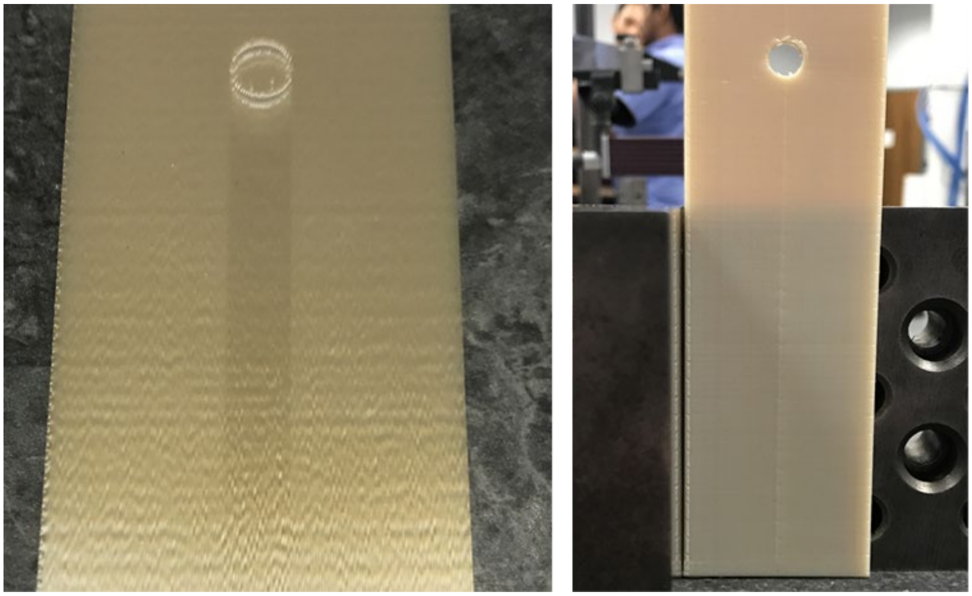

Figure 21. Example sacrificial plug (left), and defects created by the printing process (right)

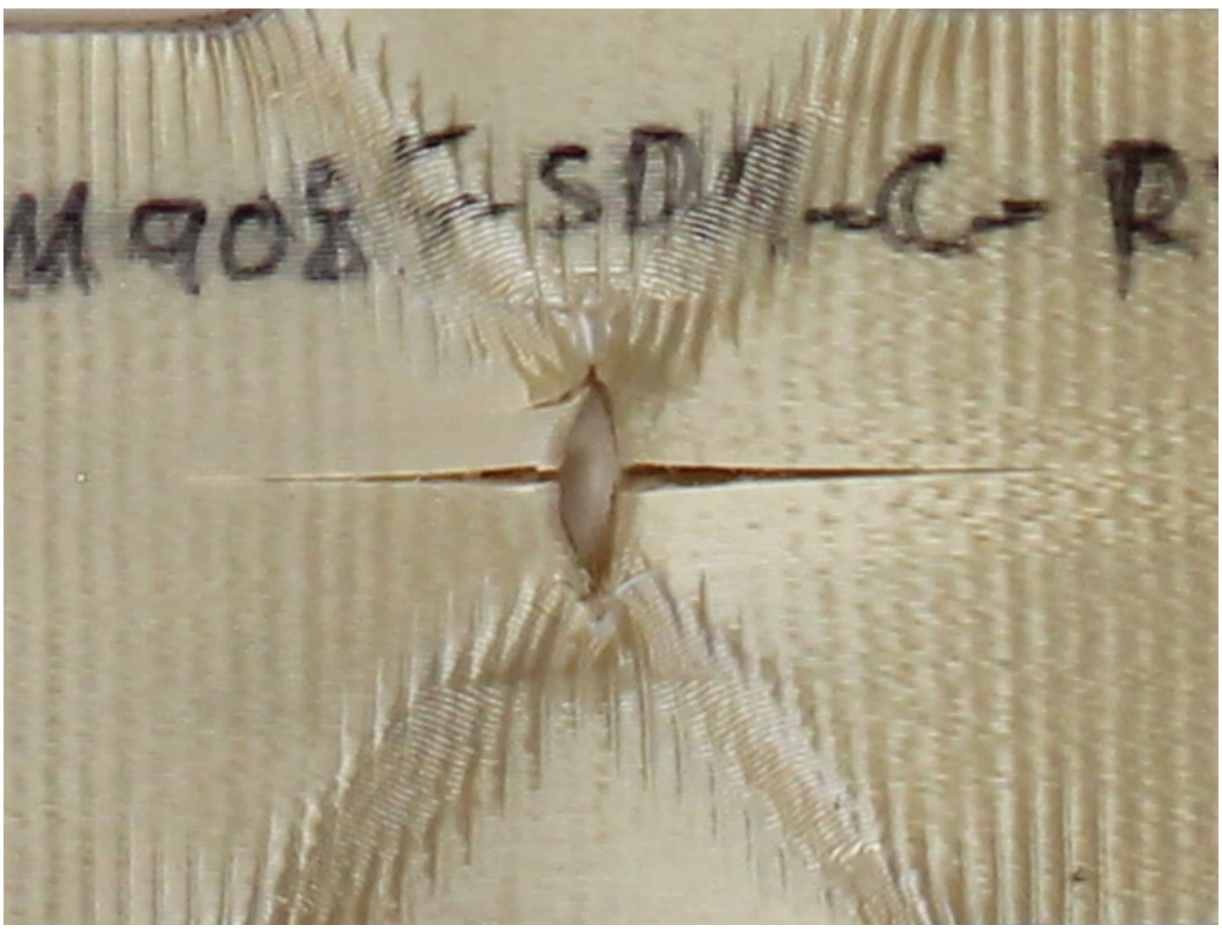

Figure 22. Post-test failure of an OHC specimen produced in the XZ-orientation

Material extrusion is known for not being able to accurately produce holes, especially ones that run orthogonal to the build orientation. Based on the observed behaviors from the qualification and the equivalencies, it is clear that both the test method and fabrication approach require additional investigation. A new methodology is necessary to make sure that the data being collected is useful and indicative of a design property used to appropriately size and select parts produced with this material and process. Currently, open-hole and filled-hole data are recommended to be collected, but until a better methodology is established, this data should not be used to evaluate equivalency.

4.1.2.6 Single shear bearing

The ASTM D5961 standard (2017) was used to execute the Single shear bearing (SSB) configuration testing. Extracted data for this test consists of single shear bearing strength and single shear bearing cord stiffness (values are reported with strain ranges of 1000με-3000με). The single shear bearing specimens were produced similarly to the open- and filled-holed specimens in which an undersized hole was first fabricated and then machined to final dimensions. Unlike the notched data, the SSB data is less sensitive to the difficulties of the technology to produce a hole feature; however, it did show some variations in the data based on the defects witnessed after machining. For production simulation purposes, it is worth creating a procedure to machine, heal, and introduce inserts to the part, similar to what is done on parts in use.

4.1.2.7 IZOD impact

Impact resistance is another property that is found on several datasheets for AM polymer materials. There are two common methods, IZOD (ASTM International, 2018) and Charpy (ASTM International, 2018), each with a notched and unnotched type of test method. The program tested a notched type through IZOD impact. Previous studies have shown that there is no established method for introducing the notch without creating a defect at a layer line or seam. The data showed high forms of variation for several of the orientations, confirming that this is not a recommended approach for testing impact resistance. Going forward, unnotched methods or falling weight methods will be explored and offered up as more appropriate replacements for this property.

4.2 Qualification approach components

The setup of an AM qualification requires a lot of preparation and planning to ensure that all sources of process variation are accounted for and build characteristics become part of the study. Documentation in file creation, specimen removal, and specimen labeling are all critical steps that must happen correctly at the right moment in the qualification, because it is impossible in most instances to go back and collect that information after the fact. Critical variables were identified and balanced against the total specimen count. The minimum number of specimens was produced and tested that still produced enough samples to understand correlations or make statistical comparisons. Those critical variables consisted of chamber location, build orientation, feedstock lots/batches, machines, runs, specimen build order, and job number.

4.2.1 Specimen identification

The following was taken from the ULTEM 9085 test plan to demonstrate the complexity that goes into each specimen produced for qualification. All specimens were uniquely identified by a 10-code reference system, cross referenced with descriptive identification information as follows:

Test Plan Document Number-AM Material Manufacturer ID-Material Code-Fabricator ID-Batch ID-Machine ID-Actual Test Type-Specimen Location-Test Condition-Specimen Number.

For example, [NTPAMP001-SSYS-UM9085-RPM-A-M1-XT-13-RTD-3] denotes AM Material Manufacturer: SSYS, AM Material Name: UM9085, Fabricator: RPM, AM Material Batch: A, Machine ID: M1, Test Direction: X Tension, Test Specimen Location: Bottom Right Corner (13), Test Condition: Room Temperature Dry, and Specimen Number: 3.

The above parameters can have the values as listed in Table 1.

Table 1. Specimen identification

Parameter	**Possible Values**
Test Plan Document Number:	NTPAMP001
AM Material Manufacturer ID:	SSYS (Stratasys)
Material Code:	UM9085 (ULTEM 9085)
Fabricator ID (Company that builds the coupons):	RPM (RP+M)
Batch ID (Material batch that builds the coupons, to be cross referenced with raw resin batches and filament lots):	A, B, C, D, E, F etc.
Machine ID (Matches machine number used):	M1, M2
Actual Test Types:	XT: X Tension, XFHT: X Filled Hole Tension, YT: Y Tension, YFHT: Y Filled Hole Tension, ZT: Z Tension, ZFHT: Z Filled Hole Tension, Z45T: Z (45) Tension , Z45FHT: Z (45) Filled Hole Tension, XC: X

Parameter	Possible Values
	Compression, XOHC: X Open Hole Compression, YC: Y Compression, YOHC: Y Open Hole Compression, ZC: Z Compression, ZOHC: Z Open Hole Compression, Z45C: Z (45) Compression, Z45OHC: Z (45) Open Hole Compression, XF: X Flex, TFS: Tension Fluid Sensitivity, YF: Y Flex, DMA: Dynamic Mechanical Analysis, ZF: Z Flex, TMA: Thermomechanical Analysis, Z45F: Z (45) Flex, XFLM: X Flammability Vertical Burn Test, XFHC: X Filled Hole Compression, YFLM: Y Flammability Vertical Burn Test, YFHC: Y Filled Hole Compression, ZFLM: Z Flammability Vertical Burn Test, ZFHC: Z Filled Hole Compression, Z45FLM: Z(45) Flammability Vertical Burn Test, Z45FHC: Z (45) Filled Hole Compression, XFLM: X Flammability Vertical Burn Test, XSSB: X Single Shear Bearing, YFLM: Y Flammability Vertical Burn Test, YSSB: Y Single Shear Bearing, ZFLM: Z Flammability Vertical Burn Test , ZSSB: Z Single Shear Bearing, Z45FLM: Z(45) Flammability Vertical Burn Test , Z45SSB: Z (45) Single Shear Bearing, XNBS: X NBS smoke and toxicity, XOHT: X Open Hole Tension, YNBS:Y NBS smoke and toxicity, YOHT: Y Open Hole Tension, ZNBS: Z NBS smoke and toxicity, ZOHT: Z Open Hole Tension, Z45NBS: Z(45) NBS smoke and toxicity, Z45OHT: Z (45) Open Hole Tension, XHRP: X Heat Release Peak, XVIPS: X Vnotch IPS, YHRP: Y Heat Release Peak, ZHRP: Z Heat Release Peak, Z45HRP: Z(45) Heat Release Peak
Specimen Location:	11, 12, 13, 14, 15
Test Condition:	CTD, RTD, ETD1, ETW1, …etc. TFS11RT, TFS12RT, TFS13RT, …etc. D, W
Specimen Number:	1, 2, 3, 4, 5, 6, 7, 8, 9, A, B, C…. etc.
Note that X, Y, Z, and Z45 correspond to build direction. X=XY, Y=XZ, Z=ZX, Z45=ZX-45, is the two-letter nomenclature for AM.	

4.2.2 Build packs

The original qualification program looked at utilizing multiple specimens per build in order to optimize machine and material utilization and to cut down on costs. A statistical analysis and simulation study were performed to determine what build factors contributed the most to variations resulting in failures per test and condition in the equivalency programs. The conclusion was that the sampling method used in the ULTEM 9085 program does not comply with the assumption of the statistical equivalency test, resulting in a false-positive rate higher than the intended 5%. Sampling multiple specimens from the same job affected the statistical equivalency test in two ways:

- Sampling the equivalency specimens from two jobs causes the mean estimator to have a higher variance. The statistical test does not take this greater variability into account, since it assumes the specimens are uncorrelated.
- Sampling the qualification specimens from six jobs lowers the estimate of the variability in the population.

Both of these factors have the effect of increasing the equivalency acceptance limits and inflating the false-positive rate. The simulation study estimated the true equivalency test false-positive rate for the ULTEM 9085 ETW XY Tension Strength property at 23%. This estimate applies only to that property, since other properties will have different variability. However, we can conclude more generally that, for any property where between group variability is present, the false-positive rate is significantly higher than 5%. This learned lesson is being documented within the Testing working group of CMH-17 and will influence the test plans for future qualification programs. Packing methods are being explored for future qualification programs that reduce the false-positive rate but still take into consideration machine utilization and printing times.

4.2.3 Build orientation comparison

Polymer AM processes require multiple orientations to be characterized due to the anisotropic behavior of the material and process. For this qualification study, four orientations were determined, as each one represents a different toolpath strategy and thus a unique microstructure. Most AM original equipment manufacturers will present data produced using either two orientations (the weakest and the strongest) or three orientations (XY, XZ, and ZX). A fourth orientation ZX-45 was introduced to see if a data curve between an upright specimen and a flat specimen could be created for better part property interpolation. Figure 23 shows the four fabrication orientations studied.

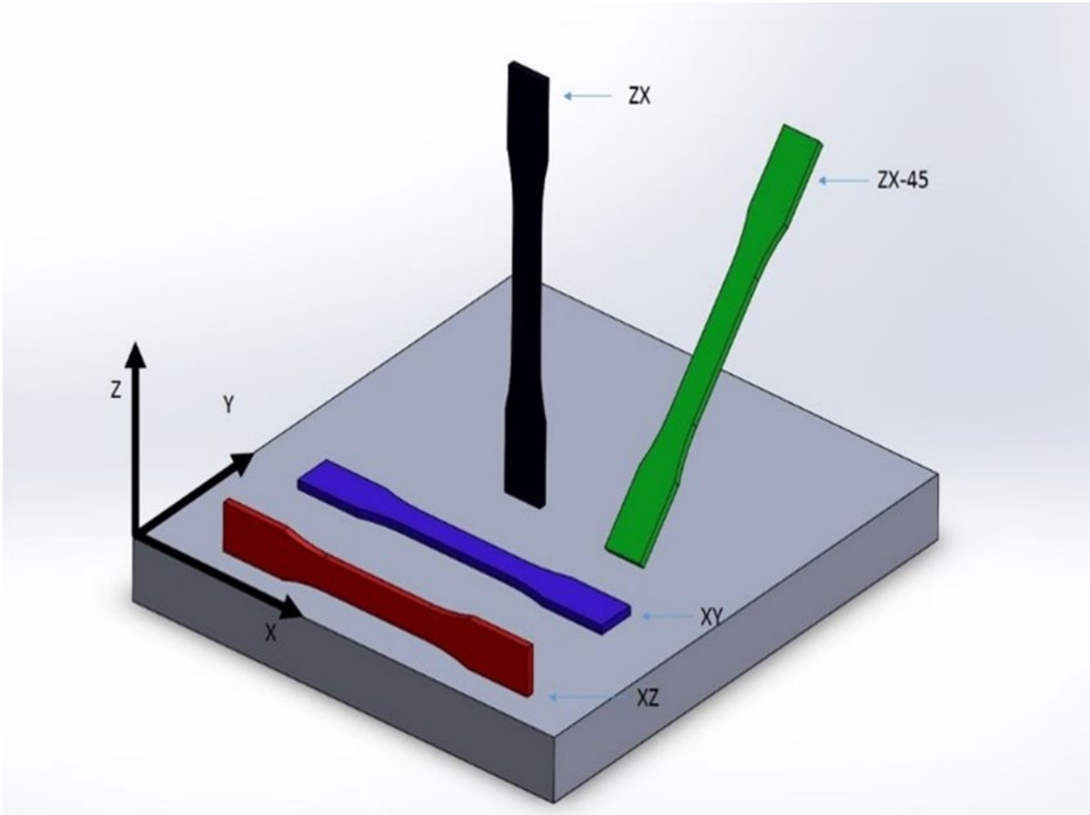

Figure 23. The four build orientations as demonstrated on a D638 dog bone

A statistical study was performed for every test condition and test type to see if there were opportunities for pooling data and finding commonalities. There were a few instances where ZX and ZX-45 data did not show statistically significant differences, but there was no consistency based on test type to warrant a blanket pooling of orientation properties. The graph in Figure 24 shows all the properties by orientation and where a few of the orientations can be pooled.

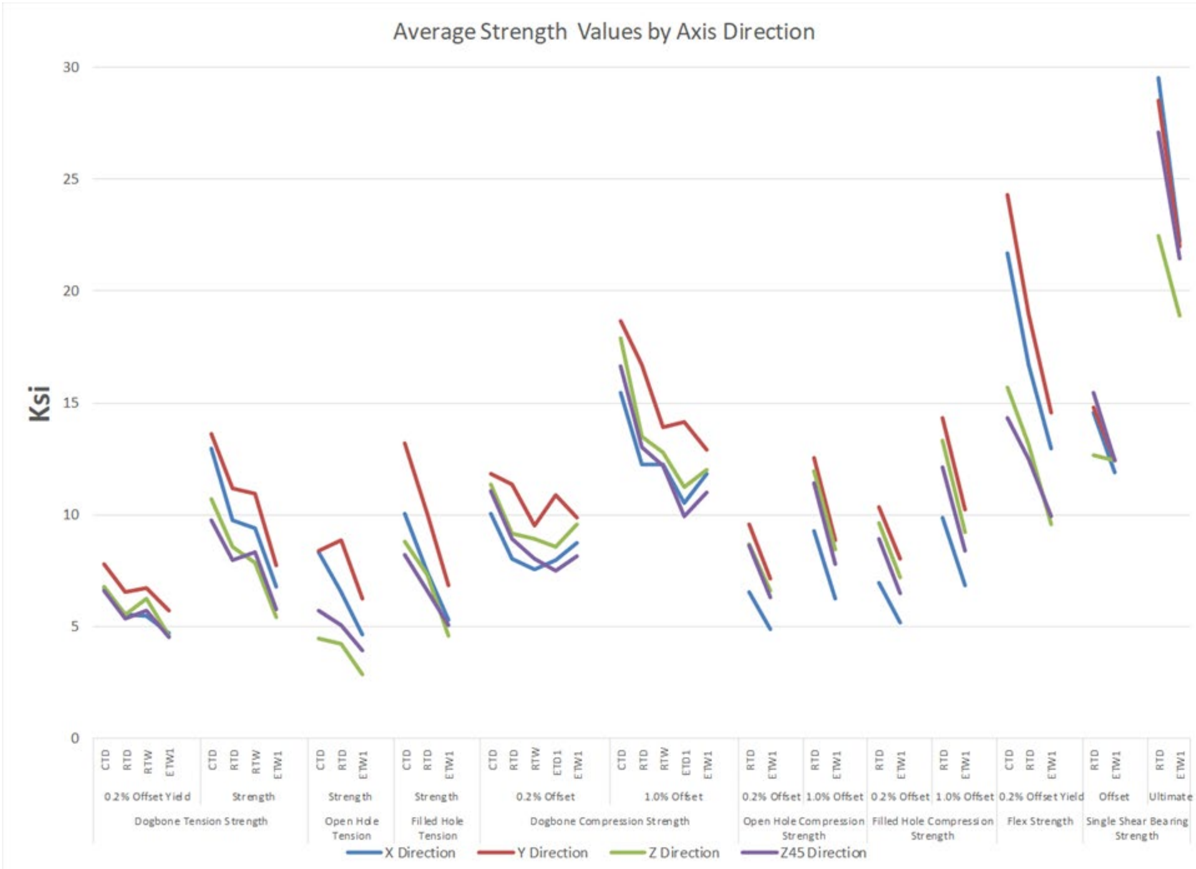

Figure 24. Results show consistency by orientation and suggests pooling some orientations

Based on the failure modes and data created with these orientations, it is our recommendation to minimize the orientations studied for a resulting cleaner dataset. In this case, the ZX-45 specimens did not offer unique data from the ZX orientation and did not offer insight into part properties that could be used to help with scaling studies.

4.2.4 Location study

Several differences can be observed within any given AM machine based on the technology and the system architecture. For SSYS's Fortus 900mc, there is a known temperature gradient within the oven that required specimens to be placed in one of five locations. Figure 25 shows the expected 15-degree variation from the heat source (back left) across the build platen. Based on the understanding of this temperature gradient, the qualification builds utilized four zones in which the back right and front left were alternated as the temperature characteristics were fairly consistent.

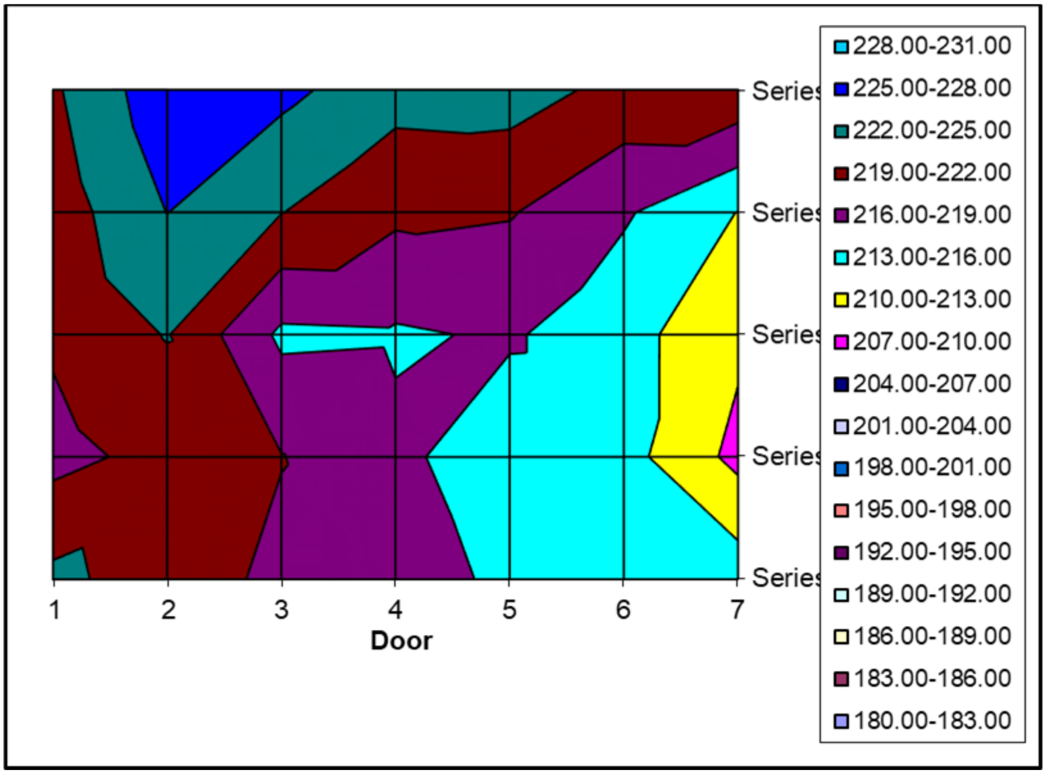

Figure 25. Temperature profile of a Fortus 900mc with marker 1 being the left of the build chamber

A statistical study was conducted to determine if the temperature gradient truly has any impact on mechanical performance. The conclusion was that build platform location has a detectable effect (an average of 2% mean difference was observed) only on specimens built in the XY orientation. For the XY orientation specimens, those built on the right side of the build platform were, on average, weaker than those built on the left. There was not a significant difference between specimens built on the top and bottom of the build platform on the same side. FDM experts commonly utilize hot and cold zones to help mitigate the thermal effects of different part geometries. Thin, flat parts like a dog bone have a much larger contact surface and either lose or retain heat at a greater rate than those built with a higher aspect ratio. As such, parts that are prone to curling are usually positioned closer to the back left corner. Larger parts do not show the same behavior, as larger thermal masses greatly influence this phenomenon.

4.3 Additional challenges

Fabrication of the specimens underwent some difficulties that required either intense maintenance on the systems performing the qualification/equivalency or forced editing to the process control documentation (PCD) used to calibrate and control the machines of interest. RP+M had selected two of their Fortus 900mcs that were known for producing good, consistent parts for their customers. However, there was a bit of an age difference between the two machines, which resulted in quite a bit of repair and part replacement work to bring the older machine up to a known good level as deemed by the PCD procedures.

While the older machine was under maintenance, the newer machine had produced one month's worth of specimens; however, this was on a refurbished head that was not properly calibrated coming off the production line. Therefore, the checks in the PCD to look for head calibration were followed. The volumetric output of the refurbished head was so low that the machine set-points could not appropriately correct for it. Figure 26 shows a comparison of the two machine's outputs as a function of microstructure and the average weight of the specimens produced on the different heads.

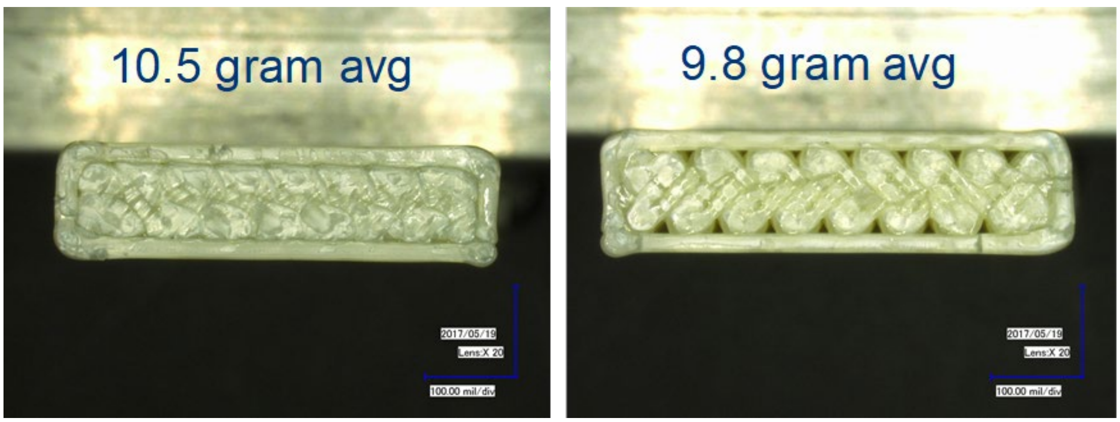

Figure 26. Under-extruded specimen microstructures differ by weight/densities

Based on these findings, an entire section was added to the PCD that provided a redundant check on volumetric output to ensure a miscalibrated head could not go on a machine. As an additional line of defense, the collection specimen weights were added to the AIR procedures and checklist.

5 Conclusion

In summary, a significant first step was made with the creation of a qualification framework that has been recognized by the AM industry (both commercial and Department of Defense) as a major impact to addressing a key barrier to the adoption of the technology. Based on the lessons learned from the program activities, tasks were created and executed in subsequent years to address the gaps that still exist to expand the framework created. Continuing to update the framework will provide additional opportunities for polymer AM to be implemented within the aviation industry. The results of this work are being added as new content to the CMH-17 AM Volume, ASTM standards, and SAE specifications with the intent to further proliferate the learnings from the work performed by CECAM.

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