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New Jersey 08405

Processing-Property Relationship of Oxide/Oxide Composites

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16. Abstract

A process-property relationship analysis has been undertaken on a set of Axiom Material AX-7800 data obtained under a program to develop the framework for qualification of oxide/oxide ceramic matrix composite materials. The data show high variability in physical and mechanical properties, resulting in unacceptably low B-basis allowables when using the statistical methods defined in the *Composite Materials Handbook* CMH-17 Volume 5, Chapter 17. An analysis of the effects of processing variables and physical properties on mechanical properties has been completed. Least squares regressions and multivariate linear regressions were executed to evaluate these relationships. Additionally, microstructural evaluations were performed to support the analysis and root cause determinations of variability. Based on correlations observed between the data, acceptance limits for the physical properties of density, porosity, and per ply thickness are proposed that reduce the effective variability in the data without effecting a major change to the process specification.

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Acronyms

Acronym	Definition
FAA	Federal Aviation Administration
2D	two dimensional
3D	three dimensional
ANOVA	analysis of variance
CMC	ceramic matrix composite
CMH-17	Composite Materials Handbook
CT	computer tomography
CV	coefficient of variation
ETD	elevated temperature dry
FAA	Federal Aviation Administration
IA	inside acceptance limits, above 80% of average value
IB	inside acceptance limits, below 80% of average value
ILT	interlaminar tension
JAMS	Joint Advanced Materials and Structures
mse	mean square error
NCAMP	National Center for Advanced Material Performance
NIAR	National Institute for Aviation Research
OA	outside acceptance limits, above 80% of average value
OB	outside acceptance limits, below 80% of average value
OHB	outside and higher than acceptance limits, below 80% of average value
OLB	outside and lower than acceptance limits, below 80% of average value
Ox/ox	oxide/oxide
RTD	room temperature dry
SBS	short beam shear
WSU	Wichita State University

Executive summary

A process-property relationship analysis has been undertaken on a set of Axiom Material AX-7800 data obtained under a program to develop the framework for qualification of oxide/oxide ceramic matrix composite materials. The data show high variability in physical and mechanical properties, resulting in unacceptably low B-basis allowables when using the statistical methods defined in the *Composite Materials Handbook CMH-17* Volume 5, Chapter 17. The process-property relationship analysis sought to understand the effects of processing variables and physical properties on the variability of mechanical properties.

Single variable least squares regressions were performed on all mechanical property strength and modulus data obtained from the qualification framework development program. Both room temperature and elevated temperature properties were analyzed. Least squares plots were generated for three physical properties and five processing parameters, and the R2 correlations were calculated. The strongest correlations observed were between room temperature strength properties and the physical properties of density and porosity. There were no significant correlations observed between mechanical properties and processing parameters.

To further examine the process-property relationships, multivariate linear regression was performed using a decision tree methodology and analysis of variance. In the first phase of this analysis, the relationship between processing conditions and physical properties was analyzed and two processing parameters were found to have a statistically significant effect. In the second phase, the relationship between physical properties and a subset of mechanical properties was analyzed, with similar results to the least squares regression. A final evaluation of porosity microstructure was performed to determine whether pore morphology was confounding the analysis, but results were inconclusive.

Based on the results of the regression analyses, physical property acceptance limits have been proposed. These limits have the effect of reducing the variability of mechanical property data, because test specimens with physical properties outside the limits are most likely to have highly variable mechanical properties. A total of 12.7% of the qualification framework data was rejected based on these limits. Additional data is needed to replace the rejected data, but when it is obtained, the final B-Basis allowables will be higher than would be calculated with the original, more variable, dataset. Minor modifications to the autoclave cure process for AX-7800 are additionally proposed to increase the likelihood of a given panel to fall within the physical property acceptance limits.

1 Introduction

The Federal Aviation Administration (FAA) is funding an effort through their Joint Center of Excellence for Advanced Materials (JAMS) program and Wichita State University (WSU) National Institute for Aviation Research (NIAR) to develop a framework for the qualification of ceramic matrix composites (CMCs) (Tomblin, Andrulonis, & Opliger, 2019) . This qualification framework includes guidelines and recommendations for their characterization, testing, design, and utilization.

Axiom Materials AX-7800-DF11-5HS3000D (AX-7800) satin weave oxide/oxide (ox/ox) prepreg was selected for the development of the framework. A total of four batches of prepreg have been manufactured over a three-year period, each containing a different lot of fabric. More than 100 panels have been fabricated, at two different facilities, from these four batches over the same period of time. More than 300 physical, 60 thermophysical, and 700 mechanical tests have been performed.

During the AX-7800 qualification activities, some test panels showed poor reproducibility, which led to significant variation in the data (Tomblin, Opliger, & Andrulonis, 2020; Tomblin, Opliger, Andrulonis, & Clarkson, 2020). Panel-to-panel variation was more significant than batch-to-batch variation. In some cases, the variation was so significant that the statistically derived B-basis material allowables obtained when using the statistical methods defined in the *Composite Materials Handbook CMH-17* Volume 5, Chapter 17 (CMH-17, 2017) were unreasonably low and impractical for design.

Figure 1 shows an example of variability observed in unnotched tension testing of AX-7800. In this figure, both room temperature dry (RTD) and elevated temperature dry (ETD) test data is shown for two panels from each of three batches of material. There is one outlier identified by a red circle in ETD Batch 2 Cycle 2, but otherwise intrapanel variability is low. However, interpanel variability is significant, particularly for the ETD test condition, where the coefficient of variation (CV) for ETD normalized data is 13.51%. This results in an ETD B-basis Grade A (CMH-17 unstructured by batch) material allowable that is 30% less than the mean. Alternative calculations for material allowables provided even worse results: ETD B-basis Grade B estimated allowable (CMH-17 structured by batch) that is 42% less than the mean and ETD B-basis Grade G (CMH-17 generic basis value) material allowable that is 50% lower than the mean. Note that ETD B-basis Grade C (CMH-17 modified CV) calculation was not applicable because of the already high CV. Even greater interpanel variability has been observed for other configurations and properties, resulting in very low material allowables.

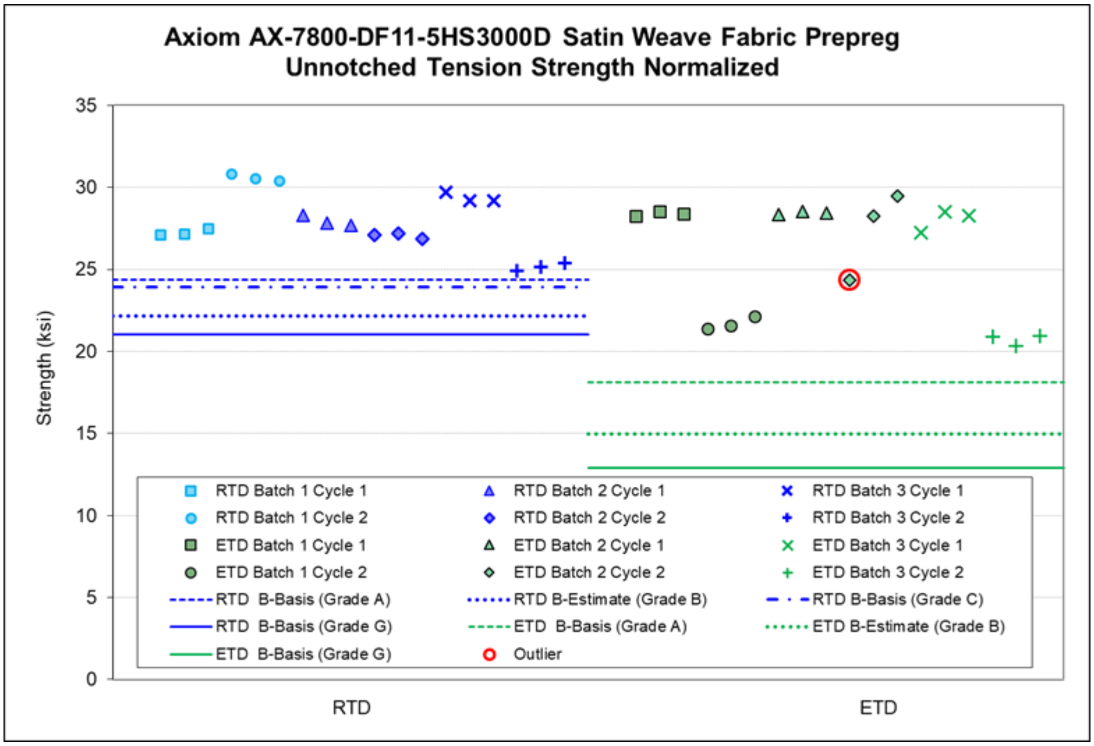

Figure 1: Unnotched tension results for AX-7800

2 Objectives

A process-property relationship study was devised to better understand the causes of panel-to-panel variation seen in the qualification data for AX-7800. This study was formulated based on the data obtained as part of the qualification framework development, rather than obtaining new data. The objectives for this study were as follows:

- Understand the effects of physical properties such as density, porosity, and cured ply thickness on variability of mechanical properties.
- Use physical and mechanical test data to establish physical property panel acceptance limits to discriminate good test panels from bad test panels and provide a basis for filtering the qualification data for the determination of material allowables.
- Use process-property relationships to guide process specification improvements.

Process specification improvements must be considered “minor” changes, in accordance with National Center for Advanced Material Performance (NCAMP) qualification guidelines, in order for future processing of the material to be equivalent to the original qualification process. Therefore, this study seeks only to improve test panel reproducibility, which is considered a "minor" change, rather than improve intrinsic material properties, which is considered a "major" change.

3 Approach

This process-property relationship study was assembled based on data already collected as part of the framework development activities with AX-7800. As part of the framework development, three batches of AX-7800 material were used to produce more than 100 panels. The panels were cured and sintered in numerous separate cure cycles, in accordance with the NCAMP process. The processing variables, physical properties, and mechanical properties extracted from the qualification study and used in this analysis are presented in Table 1.

Table 1: Process-property relationship variables

Variables		
Processing	**Physical Testing**	**Mechanical Testing**
Min Vacuum During AC Cure ["Hg]	Density [g/cm3]	All Test Types (e.g., WT, FT, WC, FC, ILS)
Sintering Temperature [°F]	Porosity [% Vol]	All Properties (i.e., Strength and Modulus)
Sintering Hold Time [minutes]	Fiber Volume [% Vol]	All Test Temperatures (i.e., RTD and ETD)
Time at First Dwell [minutes]	Matrix Volume [% Vol]	
Time at Initiation of Full Pressure to Final Time of First Dwell [minutes]	Per Ply Thickness [in]	

The first analysis evaluating process-property relationships was a linear least squares regression. Analyses were performed in order to determine whether any single processing parameter or physical property correlated with any mechanical test property. Subsequently, a multivariate regression analysis was performed to see if certain parameters in combination with one another show correlation with physical or mechanical properties. A regression decision tree was used to model the data. Separately, coupon microstructure was evaluated to identify if pore size, shape, or distribution may have acted as a confounding variable on the effect of processing parameters on mechanical properties.

Following these analyses, acceptance limits were defined for AX-7800 porosity, density, and cured ply thickness. Implementation of acceptance limits reduces variability in the qualification data such that useable B-basis allowables are obtainable. Importantly, this is accomplished as a "minor" change to the process specification and does not invalidate the qualification data.

4 Analysis

4.1 Least squares regression

Single parameter least squares regression was performed to evaluate process-property relationships using the variables defined in Table 1. All processing data were taken from the cure and sintering runs corresponding to specific panels. All physical test data except per ply thickness was determined on representative specimens from each panel. Per ply thickness was already measured for each specimen mechanically tested, so representative samples were not needed.

Figure 2 and Figure 3 present a subset of the many linear regressions performed on the AX-7800 qualification data. In Figure 2, each sub-figure plots a mechanical strength or modulus versus a processing parameter. In Figure 3, each sub-figure plots a mechanical strength or modulus versus a physical property. In both sets of sub-figures, least squares lines are fitted to the data, and an equation for the line as well as an R2 correlation value are shown.

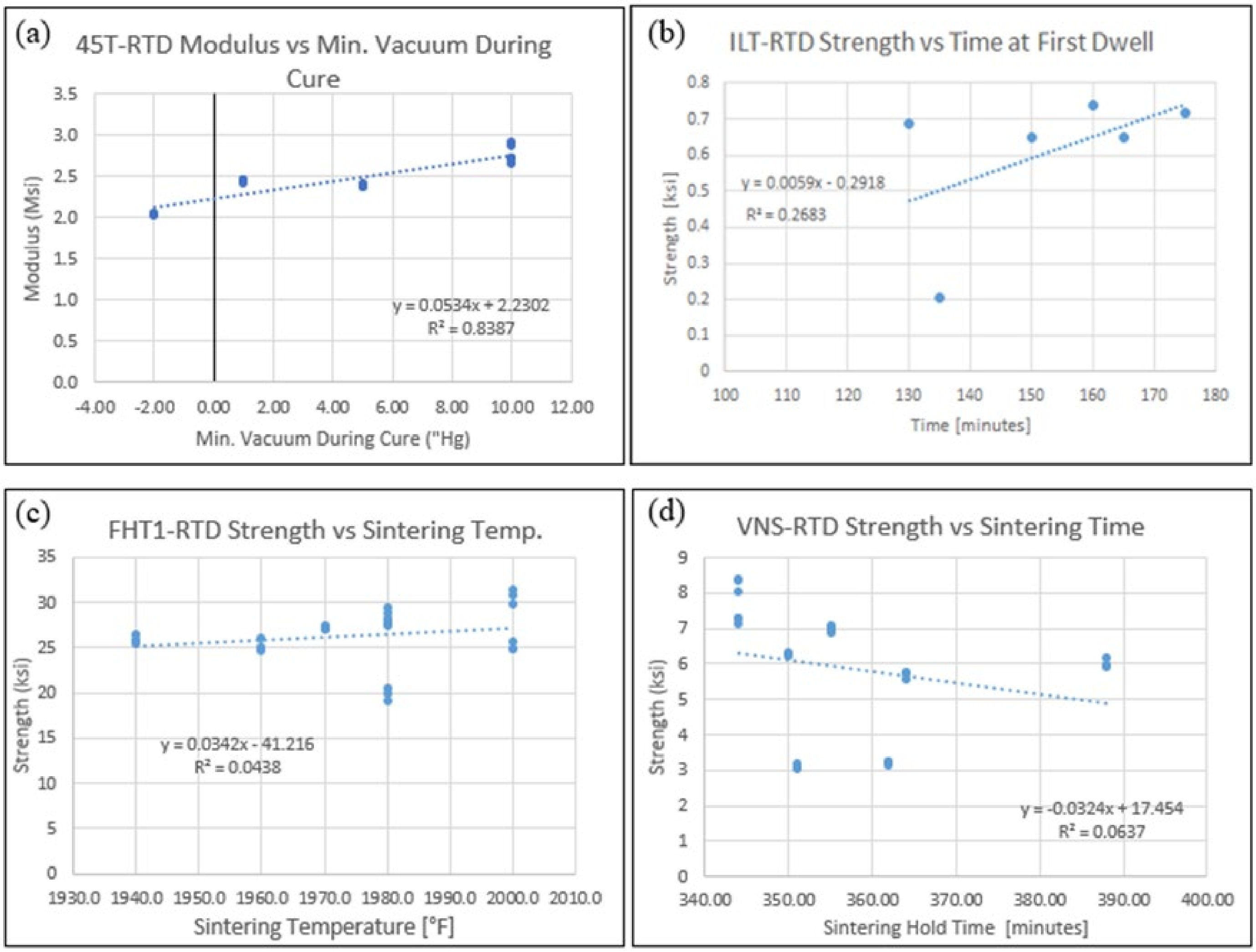

Figure 2: Example mechanical property linear regression with processing variables

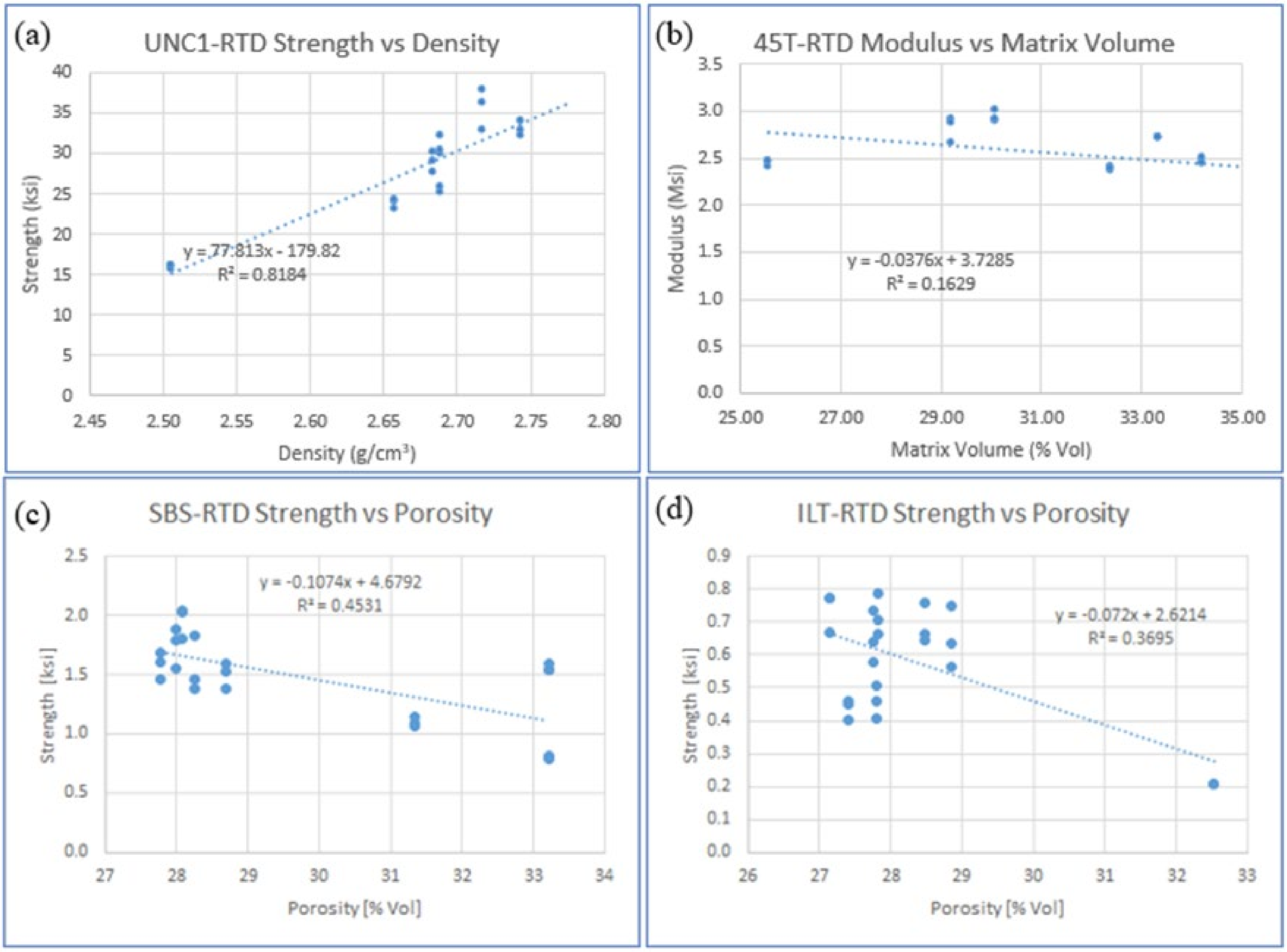

Figure 3: Example mechanical property linear regression with physical property variables

Table 2 presents the R2 correlations for physical properties and room temperature dry (RTD) strengths. Table 3 presents the same types of correlations but for RTD moduli. Correlations for strength and modulus in the ETD condition are presented in Table 4 and Table 5. The strongest correlations observed are between room temperature strength properties and both density and porosity. Fiber volume and per ply thickness have much higher correlation with most modulus properties than strength properties at room temperature, but the correlations are still comparatively low. Correlations were somewhat lower for ETD properties, but similar trends are present. However, no well-defined relationships are identified from this analysis, as few correlations surpass a traditional R2 > 0.8 threshold, and for those few correlations that do, there are not universal trends across similar properties.

It can be particularly challenging to correlate the processing conditions directly to mechanical properties using this process. One issue is that it is not currently possible to define causation, another is that the results are also too scattered to identify mechanisms in the current data set. For example, for in-plane shear (±45° tension), for both RTD and ETD conditions, density is correlated with strength. Examination of the minimum vacuum pressure versus density results for panels from the data set used to generate RTD in-plane shear strength reveals that minimum vacuum pressure is correlated with density, which relates to the correlation with strength. However, in the panels used for ETD in-plane shear (±45° tension) strength, minimum vacuum pressure and density are not correlated, and minimum vacuum pressure and ETD in-plane shear strength are not correlated. It is also not clear why ±45° tension in-plane shear strength is correlated with density, and sometimes minimum vacuum pressure, but v-notch in-plane shear is correlated with neither. The fundamental issue appears to be multivariate effects between processing conditions and physical properties. To further assess this, the next section looks at multivariate effects in process property relationships.

Table 2: Correlations between physical and processing properties and RTD strengths

Test - RTD Strength	R2 Value							
	Density [g/cm^3]	Porosity [% Vol]	Fiber Volume [% Vol]	Matrix Volume [% Vol]	Per Ply Thickness [in]	Min Vacuum During AC Cure ["Hg]	Sintering Temperature [°F]	Sintering Hold Time [min]
Warp Tension	0.41	0.39	0.07	0.00	0.23	0.01	0.00	0.09
Warp Compression	0.72	0.70	0.35	0.00	0.43	0.00	0.25	0.00
Fill Tension	0.06	0.12	0.16	0.01	0.17	0.28	0.11	0.12
Fill Compression	0.29	0.56	0.67	0.52	0.10	0.62	0.17	0.01
In-Plane Shear (+/-45 Tension)	0.76	0.60	0.04	0.03	0.32	0.87	0.23	0.17
Unnotched Tension	0.23	0.16	0.01	0.09	0.04	0.13	0.34	0.05
Unnotched Compression	0.82	0.75	0.60	0.01	0.43	0.26	0.00	0.01
Open-Hole Tension	0.08	0.04	0.12	0.10	0.24	0.20	0.23	0.00
Open-Hole Compression	0.17	0.38	0.04	0.34	0.01	0.02	0.17	0.03
Filled-Hole Tension	0.56	0.49	0.17	0.01	0.20	0.01	0.04	0.23
Interlaminar Shear (Double Notch)	0.01	0.02	0.00	0.05	0.00	0.14	0.19	0.15
Single-Shear Bearing	0.73	0.80	0.29	0.01	0.31	0.29	0.33	0.11
Double-Shear Bearing	0.00	0.03	0.13	0.43	0.09	0.18	0.22	0.00
In-Plane Shear (V-Notch)	0.06	0.07	0.10	0.54	0.07	0.02	0.08	0.06
Interlaminar Shear (Short-Beam)	0.46	0.45	0.39	0.10	0.37	0.00	0.12	0.00
Interlaminar Tension	0.43	0.37	0.33	0.01	0.21	0.08	0.38	0.13
Flexure	0.13	0.16	0.02	0.35	0.47	0.00	0.00	0.05

Correlation Color Scale								
0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9

Table 3: Correlations between physical and processing properties and RTD modulus

Test - RTD Modulus	R2 Value							
	Density [g/cm^3]	Porosity [% Vol]	Fiber Volume [% Vol]	Matrix Volume [% Vol]	Per Ply Thickness [in]	Min Vacuum During AC Cure ["Hg]	Sintering Temperature [°F]	Sintering Hold Time [min]
Warp Tension	0.15	0.09	0.76	0.52	0.78	0.30	0.00	0.33
Warp Compression	0.57	0.55	0.66	0.27	0.67	0.00	0.18	0.19
Fill Tension	0.08	0.16	0.09	0.00	0.12	0.05	0.28	0.06
Fill Compression	0.19	0.18	0.12	0.00	0.14	0.03	0.07	0.17
In-Plane Shear (+/-45 Tension)	0.37	0.39	0.47	0.13	0.80	0.84	0.17	0.01
Unnotched Tension	0.43	0.29	0.22	0.02	0.40	0.35	0.02	0.15
Unnotched Compression	0.55	0.46	0.27	0.01	0.15	0.20	0.05	0.05
In-Plane Shear (V-Notch)	0.18	0.22	0.00	0.23	0.00	0.00	0.14	0.08
Flexure	0.32	0.40	0.28	0.05	0.00	0.11	0.07	0.29

Correlation Color Scale

0.1 0.2 0.3 0.4 0.5 0.6 0.7 0.8 0.9

Table 4: Correlations between physical and processing properties and ETD strength

Test - ETD Strength	R2 Value							
	Density [g/cm^3]	Porosity [% Vol]	Fiber Volume [% Vol]	Matrix Volume [% Vol]	Per Ply Thickness [in]	Min Vacuum During AC Cure ["Hg]	Sintering Temperature [°F]	Sintering Hold Time [min]
Warp Tension	0.06	0.08	0.25	0.12	0.07	0.08	0.49	0.10
Warp Compression	0.11	0.02	0.04	0.04	0.12	0.07	0.04	0.03
Fill Tension	0.05	0.03	0.66	0.27	0.24	0.03	0.30	0.25
Fill Compression	0.11	0.18	0.02	0.08	0.40	0.00	0.01	0.20
In-Plane Shear (+/-45 Tension)	0.69	0.47	0.01	0.18	0.00	0.14	0.13	0.09
Unnotched Tension	0.40	0.37	0.39	0.07	0.26	0.03	0.12	0.22
Unnotched Compression	0.00	0.01	0.09	0.16	0.05	0.27	0.05	0.01
Open-Hole Tension	0.09	0.00	0.47	0.50	0.50	0.18	0.27	0.14
Open-Hole Compression	0.02	0.04	0.25	0.47	0.14	0.35	0.60	0.00
Filled-Hole Tension	0.51	0.50	0.77	0.19	0.72	0.01	0.11	0.26
Interlaminar Shear (Double Notch)	0.03	0.06	0.00	0.09	0.00	0.02	0.27	0.06
Double-Shear Bearing	0.20	0.20	0.01	0.06	0.03	0.66	0.45	0.58

Correlation Color Scale

0.1 0.2 0.3 0.4 0.5 0.6 0.7 0.8 0.9

Table 5: Correlations between physical and processing properties and ETD modulus

Test - ETD Modulus	R2 Value							
	Density [g/cm^3]	Porosity [% Vol]	Fiber Volume [% Vol]	Matrix Volume [% Vol]	Per Ply Thickness [in]	Min Vacuum During AC Cure ["Hg]	Sintering Temperature [°F]	Sintering Hold Time [min]
Warp Tension	0.18	0.16	0.31	0.11	0.24	0.73	0.01	0.00
Warp Compression	0.25	0.23	0.33	0.30	0.48	0.23	0.00	0.25
Fill Tension	0.15	0.23	0.46	0.09	0.63	0.00	0.03	0.18
Fill Compression	0.12	0.10	0.30	0.30	0.33	0.08	0.00	0.33
In-Plane Shear (+/-45 Tension)	0.18	0.19	0.40	0.16	0.37	0.01	0.07	0.02
Unnotched Tension	0.12	0.03	0.17	0.08	0.46	0.42	0.18	0.50
Unnotched Compression	0.11	0.05	0.01	0.00	0.01	0.20	0.01	0.11

Correlation Color Scale

0.1 0.2 0.3 0.4 0.5 0.6 0.7 0.8 0.9

4.2 Multivariate regression

As exemplified by the data in Figure 2(d), analysis of the processing data and mechanical properties appeared to be influenced by more than a single variable. Therefore, multivariate regression analyses were performed to identify the interaction between properties. In the first phase on the multivariate regression analysis, the relationship between processing variables and physical properties was analyzed. In the second phase, the relationship between physical properties and mechanical properties was analyzed.

A multivariate decision tree technique was used to analyze the data. The first step was the determination of appropriate variables to include in the decision tree analysis. To this end, correlations between the processing variables were calculated to determine whether any were dependent on any other and could therefore be omitted from the analysis. These correlations are shown in Table 6, where it can be seen that no variables show a particularly high absolute correlation. The threshold in this analysis was 0.8. Since no correlations were significant, all five variables were included in the decision tree and regression analysis.

Table 6: Correlations between processing variables

CORRELATION of Independent Variables	*Min Vacuum During AC Cure ["Hg]*	*Sintering Temperature [°F]*	*Sintering Hold Time [minutes]*	*Time at First Dwell [minutes]*	*Time at Initiation of Full Pressure to Final Time of First Dwell [minutes]*
Min Vacuum During AC Cure ["Hg]	1				
Sintering Temperature [°F]	0.167709769	1			
Sintering Hold Time [minutes]	0.200950822	-0.067176687	1		
Time at First Dwell [minutes]	0.371643086	-0.359145077	0.293659783	1	
Time at Initiation of Full Pressure to Final Time of First Dwell [minutes]	0.086823174	-0.703997857	0.397645937	0.594743319	1

An example regression decision tree developed as part of this analysis is shown in Figure 4. The decision tree in this figure determines the process parameters affecting the physical property of density. In each decision box, a value of each processing variable, or combination of variables, has been determined that minimizes the error between two halves of a data set. The mean square error (MSE) is the value of the minimized error. The number of samples and the mean value of density for those samples are given as well. This independent variable binning is useful in evaluating acceptable processing condition variability as well.

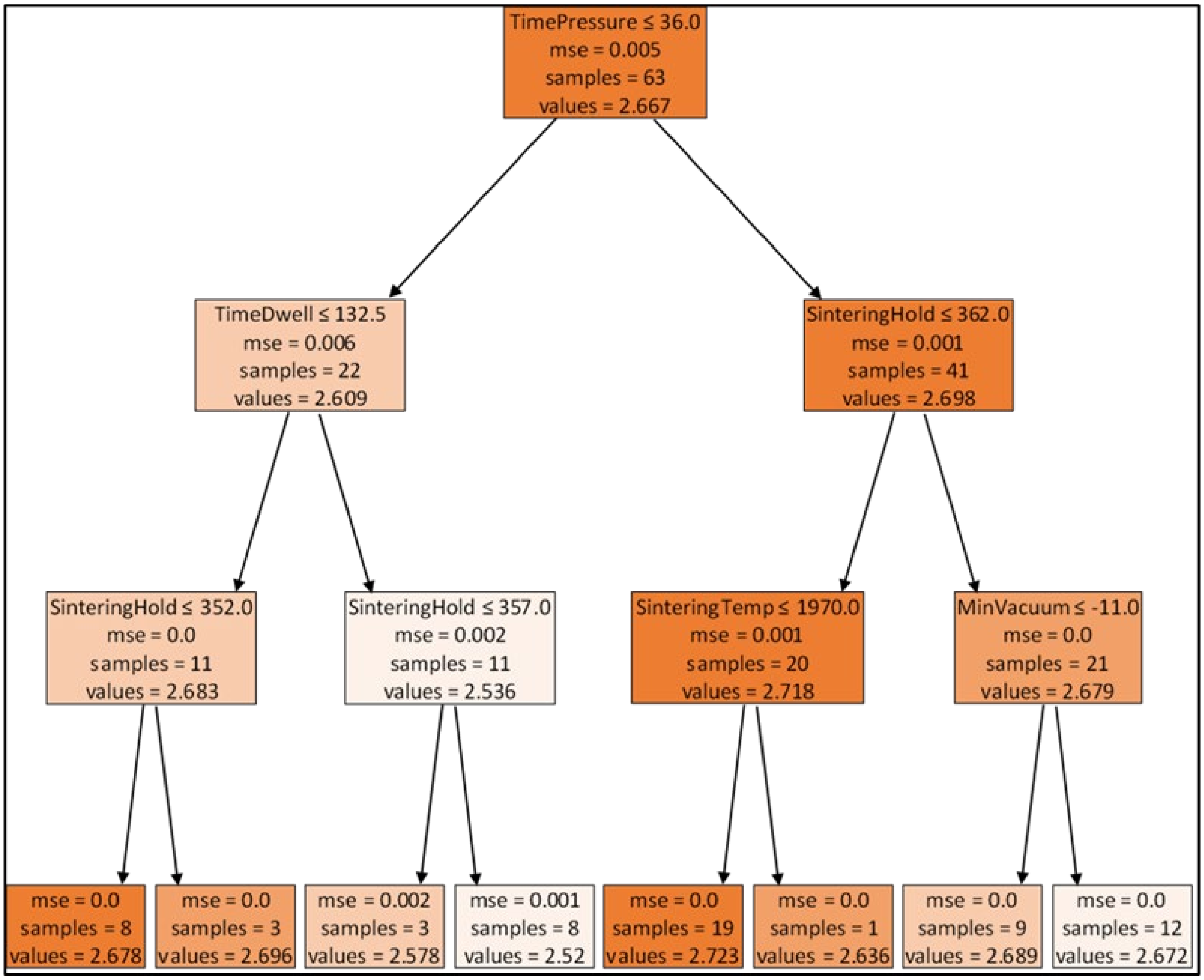

Figure 4: Regression decision tree for processing variable effects on density

An analysis of variance (ANOVA) was performed on the data using all processing variables as inputs and with physical properties as outputs. A summary of ANOVA numerical results for the physical property of density are given in Figure 5. For each input variable, a P-value is determined, quantifying the statistical significance of that variable on the output physical property. Interactions between several input variables and output properties have also be determined but are not shown in this figure. Similar statistics were calculated for the output properties of cured ply thickness and porosity. These analyses found the following processing parameters had a statistically significant effect (P-value ≤ 0.05) on physical properties:

- Sintering hold time
- Time at initiation of full pressure to final time of first dwell

Regression P1	Density (G/CM3)						
Summary Output							

Regression Statistics	
Multiple R	0.689480987
R Square	0.475384031
Adjusted R Square	0.429365087
Standard Error	0.051765437
Observations	63

ANOVA

	df	SS	MS	F	Significance F
Regression	5	0.138407	0.027681	10.33018	4.38255E-07
Residual	57	0.152741	0.00268		
Total	62	0.291148			

		Coefficien	Standard E	t Stat	P-value	Lower 95%	Upper 95%
	Invercept	2.883881	1.472581	1.958385	0.05508489	-0.06491	5.932675
VAR1	Min Vacuum During AC Cure	0.00017	0.000243	0.698541	0.487679096	-0.00032	0.000656
VAR2	Sintering Temperature	0.000167	0.000763	0.219104	0.827351977	-0.00136	0.001695
VAR3	Sintering Hold Time	-0.00184	0.000608	-3.02096	0.003768198	-0.00306	-0.00062
VAR4	Time at First Dwell	-0.00022	0.000356	-0.61433	0.541441501	-0.00093	0.000494
VAR5	Time at Initiation of Full Pressure to Final Time of First Dwell	0.003775	0.000804	4.694812	0.000017205	0.002165	0.005385

Figure 5: Regression statistics for processing conditions and density

In the second phase of the multivariate regression analysis, the effect of physical properties on mechanical properties was evaluated. The process again started with evaluating dependency between the different physical property variables. Table 7 presents correlations between the physical variables. It is unsurprising that there are some high correlations measured between the physical property variables. Per ply thickness is used in the calculation of fiber volume, and the sum of porosity, fiber volume, and matrix volume equals one. Based on the correlations obtained, this phase of analysis considered only density and matrix volume as independent variables.

Table 7: Correlations between physical property variables

CORRELATION of Physical Variables

	Density [g/cm3]	Porosity [% Vol]	Fiber Volume [% Vol]	Matrix Volume [% Vol]	Per Ply Thickness [in]
Density [g/cm3]	1				
Porosity [% Vol]	-0.987098247	1			
Fiber Volume [% Vol]	0.730506333	-0.743556284	1		
Matrix Volume [% Vol]	-0.042932047	0.04872413	-0.704108285	1	
Per Ply Thickness [in]	-0.758180766	0.763079022	-0.969316574	0.637543744	1

Only a subset of the mechanical test data were used in this phase of the analysis. The dependent variables selected were the RTD test conditions of v-notch shear strength and modulus, short beam shear (SBS) strength, and interlaminar tension strength (ILT). An example regression decision tree developed as part of this analysis is shown in Figure 6. Because the data used was sourced from the AX-7800 NCAMP qualification program, each mechanical property only has 8 data points available. Therefore, the results from this analysis are rough estimates only. More data would be required to draw any significant statistical conclusions.

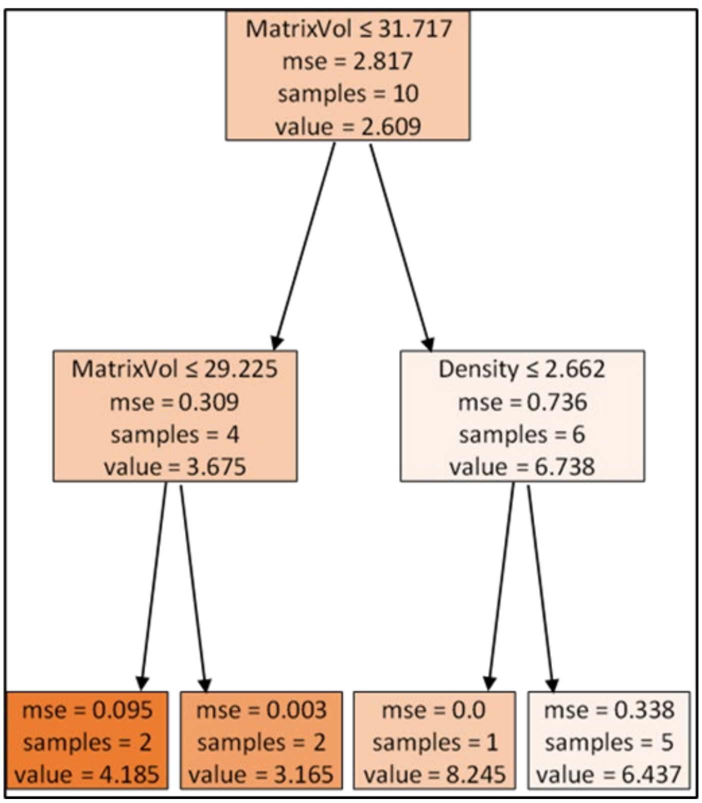

Figure 6: Regression decision tree - physical property variable of V-Notch Shear RTD strength

This regression analysis did not reveal any significant new findings. The same physical properties were found to have a statistically significant effect (P-value ≤ 0.05) on the evaluated mechanical properties as exhibited high R2 correlations in the linear least squares regression analysis. As in the previous analysis, matrix-dominant mechanical properties were more significantly affected by physical properties than fiber-dominant mechanical properties.

4.3 Evaluation of microstructure

While the least squares regression and multivariate regression analyses revealed some statistically significant relationships between processing parameters and properties, the analysis did not fully explain the high variability observed in the test data. Possibly confounding the analysis is the fact that there were some specimens with similar measured bulk porosity which resulted in significantly different mechanical properties, possibly indicating an effect of pore morphology. The ILT data presented in Figure 3(d) is an example of variability that was not explained by the regression analyses. Because the failure location for an ILT test is a single interlaminar plane, the specimen will fail at the weakest ply interface. A specimen containing even one interlaminar interface with long, interconnected pores in a single plane would exhibit a low ILT strength, even if the bulk porosity was the same as specimens with smaller, more distributed pores.

An X-ray computed tomography (CT) was utilized to determine pore size, location, and distribution in a sub-set of laminates. At least one laminate was selected from each batch and cure/sinter cycle. The X-ray CT scans were reviewed to look for differences between panels, and analysis was performed to determine if the features in the microstructure correlate with panel quality.

Figure 7 presents an analysis of pore size for three different panels from which select ILT specimens were cut. In each sub-plot of Figure 7, the upper left and right corners of the plot respectively show the average ILT strength measured for that panel and the bulk porosity for the panel. A section of each panel was then imaged using x-ray CT and between 17 and 25 virtual slices were created to separate the three-dimensional (3D) representation into a series of two-dimensional (2D) images. Image analysis software was then used to measure the thickness and area of every pore above a threshold in each slice. These morphological data are then plotted to determine qualitatively whether the morphology is in any way related to ILT strength.

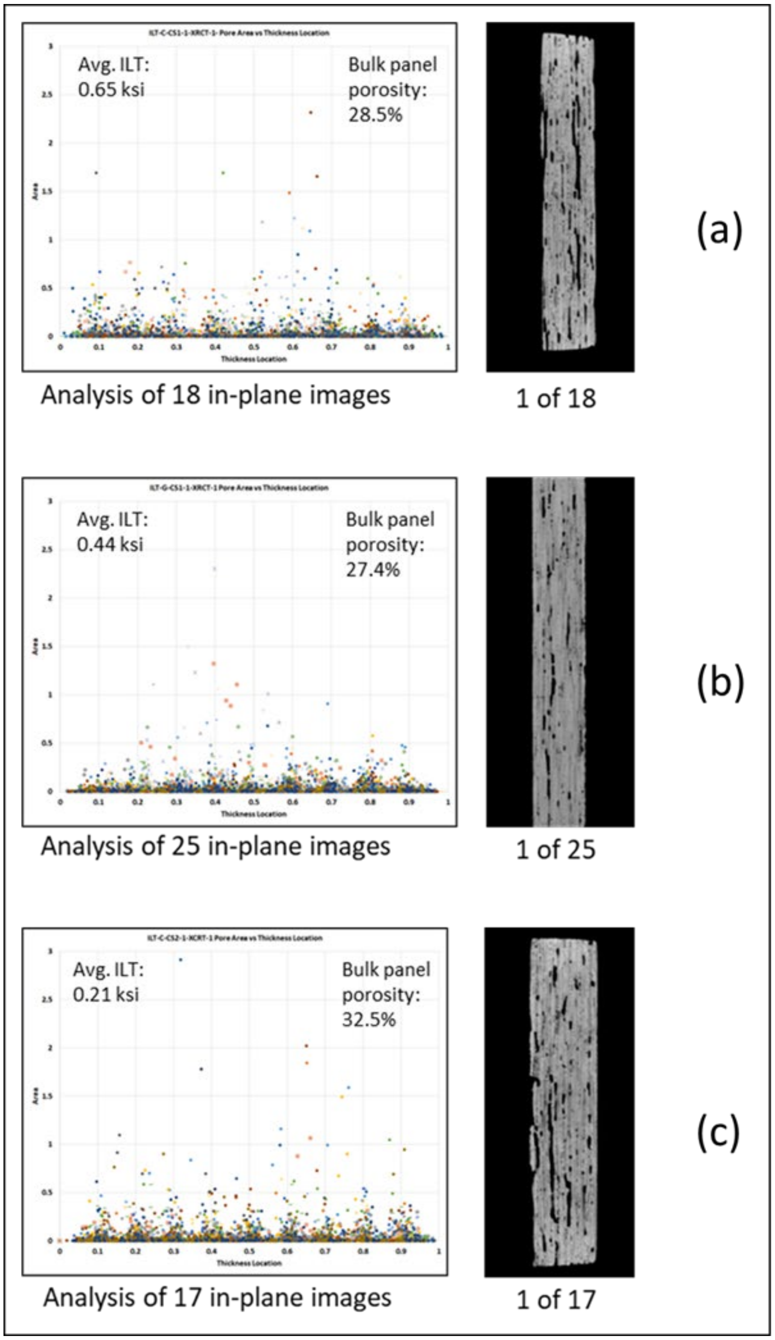

Figure 7: Porosity analysis for ILT data

The evaluation of microstructure did not reveal any clear relationships between the morphology of pores and measured ILT strength or other mechanical properties. However, it is not clear whether that is because no relationship is present, because the analysis of pore thickness and area was not sufficient to characterize pore morphology, or because porosity varied enough across each panel that the samples evaluated were not representative of the coupons mechanically tested.

4.4 Acceptance Limits

At the outset of the AX-7800 qualification activities, the material manufacturer was able to provide typical values for many physical properties. However, acceptance limits for physical properties developed from a robust dataset of varying material and panel fabrications processes have thus far not been available. The analysis presented in this report is useful for the development of such acceptance limits. To determine acceptance limits, data from all four material batches tested as part of the AX-7800 qualification were aggregated and normalized to the mean for each property. The normalized data was then plotted against three relevant and easily obtainable material properties: porosity, density, and average (per) ply thickness. The data was analyzed with the goal of optimizing acceptance limits such that the amount of "good data" within the acceptance limits was maximized while the amount of "bad data" within the acceptance limits was minimized. In terms of this optimization, "good data" and "bad data" refer to mechanical properties generally similar to the global population versus data outside of, and in particular lower than, the general population. This was accomplished initially with a script that determined the ratio of accepted-to-rejected strength data over a range of limits to guide initial acceptance limits. Limits were further optimized by evaluating the data graphically.

In the following plots developed to understand the acceptance limits, a series of acronyms is used to identify regions where the data falls. "A" refers to data above 80% of the average normalized strength, while "B" refers to data below 80% of the average normalized strength. "I" refers to data inside the acceptance limits, while "O" refers to data outside the acceptance limits. For two-sided acceptance limits, "OL" and "OH" refer to data outside and either lower or higher than the acceptance limits. Based on these definitions, the optimization of acceptance limits is accomplished by maximizing IA and OB data while minimizing IB and OA data.

Figure 8 presents porosity acceptance limits and the data used to determine them.

- 88.4% of all data are inside of limits (IA + IB), including 81.8% that are above (IA) and 6.6% that are below (IB) 80% of the mean.
- 11.5% of all data are outside of limits (OA + OB), including 2.7% that are above (OA) and 8.8% that are below (OB) 80% of the mean.
- The acceptable range for porosity is less than 30.5%.
- No lower limit has been established based on this data, but a lower limit likely does exist.

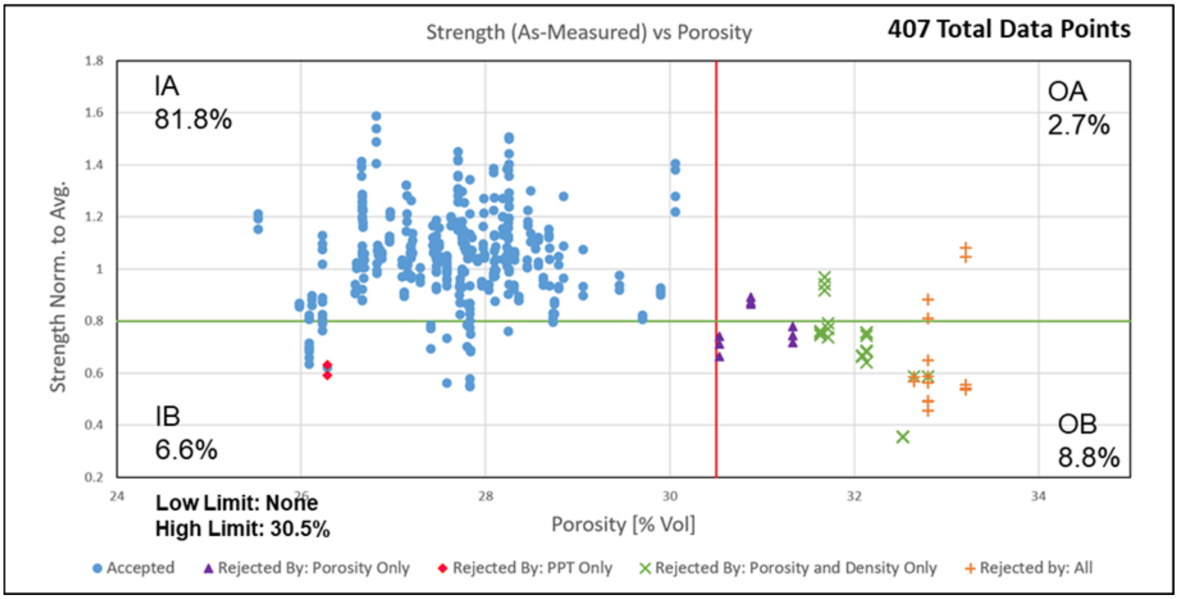

Figure 8: Determination of porosity acceptance limits

Figure 9 presents density acceptance limits and the data used to determine them.

- 90.7% of all data are inside of limits (IA + IB), including 82.6% that are above (IA) and 8.1% that are below (IB) 80% of the mean.
- 9.3% of all data are outside of limits (OA + OB), including 2.0% that are above (OA) and 7.3% that are below (OB) 80% of the mean.
- The acceptable range for density is between 2.57 g/cm^3 and 2.81 g/cm^3.

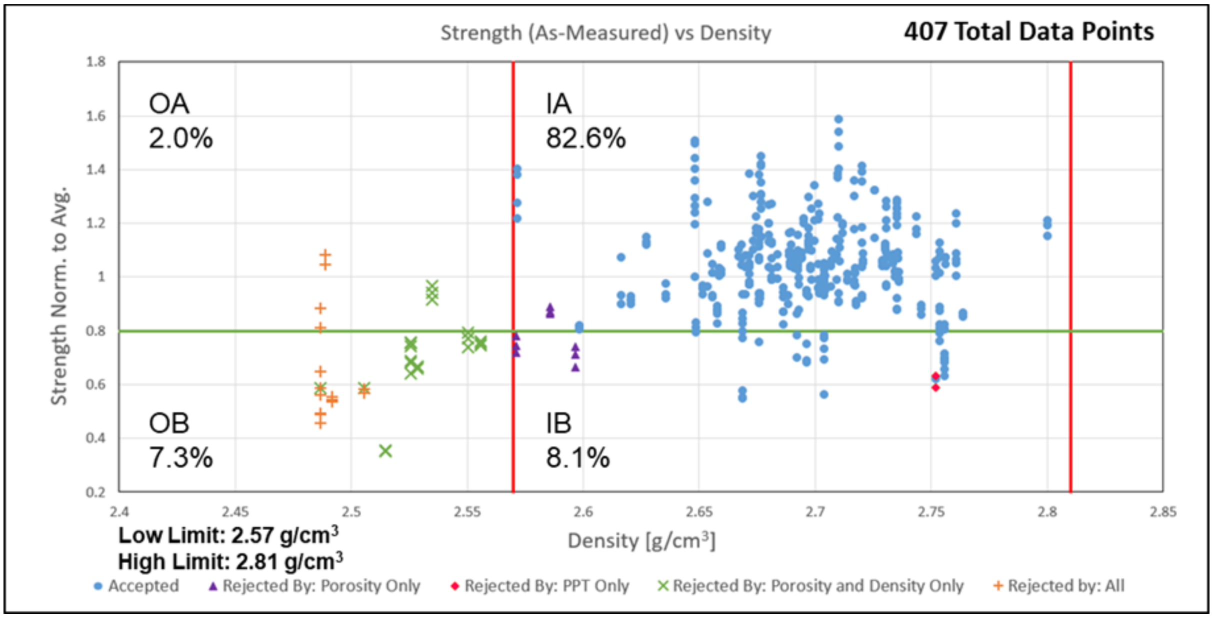

Figure 9: Determination of density acceptance limits

Figure 10 presents per ply thickness acceptance limits and the data used to determine them.

- 95.6% of all data are inside of limits (IA + IB), including 83.3% that are above (IA) and 12.3% that are below (IB) 80% of the mean.
- 4.4% of all data are outside of limits (OA + OLB + OHB), including 1.2% that are above (OA) and 3.2% that are below (OLB + OHB) 80% of the mean.
- The acceptable range for average ply thickness is between 0.0082 in/ply and 0.0102 in/ply.

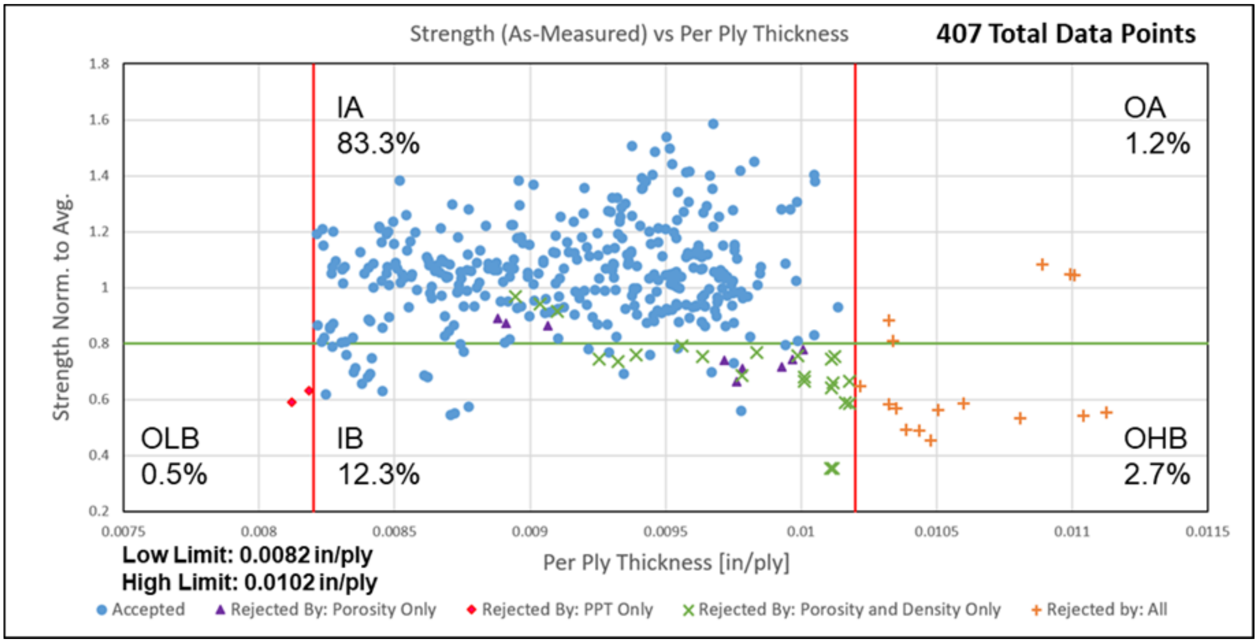

Figure 10: Determination of per ply thickness acceptance limits

Data outside any of the acceptance limits for the evaluated physical property is rejected. Many of the data were outside the acceptance limits for one physical property were also outside the acceptance limits for multiple physical properties. After filtering for porosity, density, and per ply thickness acceptance limits, 87.3% of all data are inside of limits and 14.5% are outside of limits.

5 Conclusions and future work

An analysis of the effects of processing variables and physical properties on mechanical properties has been conducted. This analysis uses a dataset obtained as part of CMC qualification framework development and seeks to assess the causes of the observed mechanical property variability. Least squares regressions and multivariate linear regressions were executed to evaluate these relationships. Additionally, microstructural evaluations were performed to support the analysis and root cause determinations of variability.

There were too few data points available in this study for the statistical analysis results to be more than estimates. However, it is clear that mechanical test variables like strength and modulus are predominantly influenced by two physical variables: density and matrix volume. In turn, these physical variables depend on the five processing variables evaluated. Based on these analyses, acceptance limits for physical properties have been proposed that have the desired effect of reducing variability without requiring a major change to the process specification.

The current study was limited to evaluating the relationship between process variables and physical properties for mechanical strength and modulus. Follow-on work is planned to also assess the relationship between process variables and physical properties with mechanical test failure modes in the present data set. It is hypothesized that changes in failure mode may provide explanation for why some properties showed different process-property relationships, RTD compared to ETD properties for example, which may support a mechanistic understanding in process-property relationships.

The autoclave cure cycle for AX-7800 is available in process specification NPS 87800 (NCAMP, 2023). Changes to the layup and autoclave cure cycle processes are proposed that may improve process reliability as well as increase the likelihood of a given panel meeting the acceptance limits identified in this study. Table 8 presents four process parameters that will be adjusted from the baseline NPS 87800 process in a follow-on study. The debulk and bleeder ply process changes come from updated guidance from Axiom Materials on their best practices. The initial dwell temperature change is based on new rheology data Axiom Materials collected after publication of NPS 87800.

Table 8: Manufacturing process parameters under evaluation

Parameter	**NPS 87800 Process**	**New Investigation**
Debulk	one 15-20 minute debulk after layup is complete	debulk at least every 6 plies during layup
Bleeder Plies	three plies	one bleeder ply per every two prepreg plies
Initial Dwell Temperature	hold at 250°F ±10°F	hold at 225°F ±10°F
Pressure	apply full pressure after 60 minutes into the initial dwell	apply full pressure at the beginning of the initial dwell

6 References

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