CHAPTER 2 GUIDELINES FOR PROPERTY TESTING OF COMPOSITES

2.1 INTRODUCTION

This chapter provides guidelines for the experimental characterization of polymer matrix composites and documents the requirements for publishing material property data in MIL-HDBK-17. Recommended test matrices for a number of uses are presented and discussed. Potential problem areas in testing and test matrix planning are highlighted and helpful options are provided. The chapter sections cover the following:

- Section 2.1 introduces the chapter and presents an approach to categorizing testing needs.
- Section 2.2 discusses a wide variety of factors that affect test results and basis values, focusing on issues of particular importance during test planning, whether for a single test or for a large testing program requiring the evaluation of hundreds or thousands of test specimens.
- Section 2.3 presents a number of preplanned test matrices organized by the key categories introduced in Section 2.1, covering the characterization of specific sets of properties at recommended test environments, and including requirements for batch and specimen quantities.
- Section 2.4 describes procedures for normalizing, reducing, and reporting test data.
- Section 2.5 describes detailed test population sampling requirements, and specific test data normalization and documentation requirements for inclusion of data into MIL-HDBK-17 Volume 2.

2.1.1 Building-block approach to substantiation of composite structures

Analysis alone is generally not considered adequate for substantiation of composite structural designs. Instead, the "building-block approach" to design development testing is used in concert with analysis. This approach is often considered essential to the qualification/certification[1] of composite structures due to the sensitivity of composites to out-of-plane loads, the multiplicity of composite failure modes and the lack of standard analytical methods.

The building-block approach is also used to establish environmental compensation values applied to full-scale tests at room-temperature ambient environment, as it is often impractical to conduct these tests under the actual moisture and temperature environment. Lower-level tests justify these environmental compensation factors. Similarly, other building-block tests determine truncation approaches for fatigue spectra and compensation for fatigue scatter at the full-scale level.

The building-block approach is shown schematically in Figure 2.1.1 and discussed in detail in References 2.1.1(b) and (c). The approach can be summarized in the following steps:

1. Generate material basis values and preliminary design allowables.
2. Based on the design/analysis of the structure, select critical areas for subsequent test verification.
3. Determine the most strength-critical failure mode for each design feature.
4. Select the test environment that will produce the strength-critical failure mode. Special attention should be given to matrix-sensitive failure modes (such as compression, out-of-plane shear, and bondlines) and potential "hot-spots" caused by out-of-plane loads or stiffness tailored designs.
5. Design and test a series of test specimens, each one of which simulates a single selected failure mode and loading condition, compare to analytical predictions, and adjust analysis models or design allowables as necessary.
6. Design and conduct increasingly more complicated tests that evaluate more complicated loading situations with the possibility of failure from several potential failure modes. Compare to analytical predictions and adjust analysis models as necessary.

[1]Design substantiation is often called "qualification" in U.S. DOD applications and "certification" in civilian applications involving the U.S. FAA. All three terms describe a similar process, but "substantiation" can be considered the more generic term, with "qualification" and "certification" often limited to the foregoing more restricted senses.

7. Design (including compensation factors) and conduct, as required, full-scale component static and fatigue testing for final validation of internal loads and structural integrity. Compare to analysis.

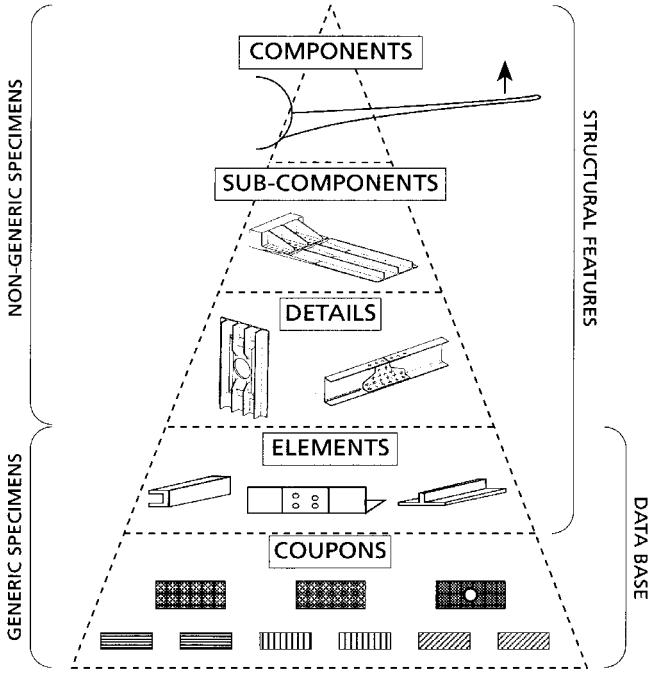


FIGURE 2.1.1 *The pyramid of tests (Reference 2.1.1(a)).*

2.1.2 Test levels and data uses

Testing activities can be defined in two basic ways, Structural Complexity Level and Data Application Category. The classes within each are discussed in more detail in the sections that follow, and can be used to map large-scale testing programs as an aid to test planning, as illustrated in Section 2.1.2.3.

2.1.2.1 Structural complexity levels

The five Structural Complexity Levels[1] are each geometry or form-based: constituent, lamina, laminate, structural element, and structural subcomponent. The material form(s) to be tested, and the relative emphasis placed on each level, should be determined early in the material data development planning process, and will likely depend upon many factors, including: manufacturing process, structural application, corporate/organizational practices, and/or the procurement or certification agency. While a single level may suffice in rare instances, most applications will require at least two levels, and it is common to use all five in a complete implementation of the building-block approach. Regardless of the Structural Complexity Level selected, physical and chemical property characterization of the prepreg (or the matrix,

[1]Due to the popularity of lamina-level testing and analysis, discussions in this handbook often emphasize development of a lamina-level database; however, this is not intended to inhibit use of any of the other Structural Complexity Levels, either singly or in combination. Also, this handbook does not emphasize the structural subcomponent category since it is so strongly application dependent; however, many of the test planning and data documentation concepts for coupon testing contained herein can be extended to structural subcomponent (or higher) testing.

if it is added as part of the process, as with resin transfer molding) is necessary to support physical and mechanical property test results. Each procurement or certification agency has specific minimum requirements and guidelines for use of data. Users of MIL-HDBK-17 are advised to coordinate with the procuring or certifying agency before planning and conducting any testing that supports structural qualification or certification.

The five Structural Complexity Levels cover the following areas:

Constituent Testing:
This evaluates the individual properties of fibers, fiber forms, matrix materials, and fiber-matrix preforms. Key properties, for example, include fiber and matrix density, and fiber tensile strength and tensile modulus.

Lamina Testing:
This evaluates the properties of the fiber and matrix together in the composite material form. For the purpose of this discussion prepreg properties are included in this level, although they are sometimes broken-out into a separate level. Key properties include fiber areal weight, matrix content, void content, cured ply thickness, lamina tensile strengths and moduli, lamina compressive strengths and moduli, and lamina shear strengths and moduli.

Laminate Testing:
Laminate testing characterizes the response of the composite material in a given laminate design. Key properties include tensile strengths and moduli, compressive strengths and moduli, shear strengths and moduli, interlaminar fracture toughness, and fatigue resistance.

Structural Element Testing:
This evaluates the ability of the material to tolerate common laminate discontinuities. Key properties include open and filled hole tensile strengths, open and filled hole compressive strengths, compression after impact strength, and joint bearing and bearing bypass strengths.

Structural Subcomponent (or higher) Testing:
This testing evaluates the behavior and failure mode of increasingly more complex structural assemblies. These are application specific and not specifically covered by MIL-HDBK-17.

2.1.2.2 Data application categories

Material property testing can also be grouped by data application into one or more of the following five categories: screening,[1] qualification, acceptance, equivalence, and structural substantiation. The starting point for testing most material systems is usually material screening. Material systems intended for use in engineering hardware are subjected to further testing to obtain additional data. While structural substantiation requirements, the last category, are not specifically addressed by MIL-HDBK-17 data generated in accordance with MIL-HDBK-17 guidelines may form part of these requirements. The five Data Application Categories cover the following areas:

Screening Testing:
This is the assessment of material candidates for a given application, often with a given application in mind. The purpose of screening testing is initial evaluation of new material systems under worst-case environmental and loading test conditions. This handbook provides guidelines for screening new material systems based on key properties for aerospace structural applications. The MIL-HDBK-17 screening test matrix provides average values for various strength, moduli, and physical properties, includes both lamina

[1]A more limited form of screening testing for the characteristic response of a limited number of specific properties (often only one property) is not explicitly named as a testing category, but is commonly performed. Such limited testing usually consists of small test populations of three to six, usually from a single material batch, and often focuses on a specific environmental condition. As each instance of testing of this type has a specific but widely varying purpose MIL-HDBK-17 does not provide explicit test matrix recommendations; however, the guidance provided for the remaining testing categories remains a useful reference for test planning.

and laminate level testing, and is designed both to eliminate deficient material systems from the material selection process and to reveal promising new material systems before planning subsequent, more in-depth, evaluations.

Material Qualification Testing:

This step proves the ability of a given material/process to meet the requirements of a material specification; it is also the process of establishing the original specification requirement values. Rigorous material qualification testing considers the statistics of the data and is ideally a subset of, or directly related to, the design allowables testing performed to satisfy structural substantiation requirements. (However, while a material may be qualified to a given specification, it still must be approved for use in each specific application.) The objective is quantitative assessment of the variability of key material properties, leading to various statistics that are used to establish material acceptance, equivalence, quality control, and design basis values. Since there are various sampling and statistical approaches used within the industry, the approach used must be explicitly defined. While a generic basis value can be obtained many ways, a MIL-HDBK-17 basis value carries with it well-defined sampling requirements and a specific statistical determination process, and emphasizes additional considerations like test methodology, failure mode, and data documentation.

Acceptance Testing:

This is the task of verifying material consistency through periodic sampling of material product and evaluation of key material properties. Test results from small sample sizes are statistically compared with control values established from prior testing to determine whether or not the material production process has changed significantly.

Equivalence Testing:

This task assesses the equivalence of an alternate material to a previously characterized material, often for the purpose of utilizing an existing material property database. The objective is evaluation of key properties for test populations large enough to provide a definitive conclusion, but small enough to provide significant cost savings as compared to generating an entirely new database. A significant use includes evaluation of possible second-sources of supply for a previously qualified material. However, the most common uses for this process are: 1) evaluation of minor constituent, constituent processing, or fabrication processing changes for a qualified material system, and 2) substantiation of previously established MIL-HDBK-17 basis values.

Structural Substantiation Testing:

This is the process of assessing the ability of a given structure to meet the requirements of a specific application. The development of design allowables, ideally derived or related to material basis values obtained during a material qualification task, is considered a part of this effort. When performed for the U.S. DOD this task is called structural qualification, and when the U.S. FAA is the certifying agency it is called structural certification.

2.1.2.3 Test program definition

A matrix is shown in Table 2.1.2.3 that can be used in test planning for large-scale testing programs. The material property tests from the Structural Complexity Levels and Data Application Categories are listed on the axes of an array, with each intersecting cell describing a distinct testing activity (though certain combinations will rarely be used). Groups of cells can be used to summarize the scope of entire building-block testing programs. The array shown in Table 2.1.2.3 illustrates a common (but by no means universal) testing sequence in the substantiation of a composite-based aerospace structural application. The sequence begins with the hatched cells at the upper left of the array and proceeds, with time, toward the cells at the lower right, with the numbered notes indicating the approximate order in the sequence. (The structural substantiation category and structural subcomponent level are shaded to indicated that they are not specifically addressed by MIL-HDBK-17).

TABLE 2.1.2.3 *Test program definition.*

STRUCTURAL COMPLEXITY LEVEL	DATA APPLICATION CATEGORIES				
	Material Screening	Material Qualification	Material Acceptance	Material Equivalence	Structural Substantiation
Constituent	1	-	-	-	-
Lamina	2	4		-	-
Laminate	-	5		-	7
Structural Element	3	6		-	8
Structural Subcomponent	-	-	-	-	9

This handbook defines a number of recommended test matrices in Section 2.3, organized by Data Application Category.

2.2 TEST PROGRAM PLANNING

2.2.1 Overview

Section 2.2 discusses a number of testing objectives that affect the execution of testing programs. The next section, 2.3 on Recommended Test Matrices, completes these items by providing recommended test matrices (types of tests and test quantities at various environments) for a number of composite material forms and objectives. These pre-defined test matrices may hove to be customized for use with a specific application.

Characterization of composite material properties is distinctly different than for either metals or unreinforced plastics. Section 2.2 provides information on many of the critical differences that affect testing and test planning, including:

- testing matrices,
- material sampling and pooling issues,
- statistical calculations,
- test method selection,
- material and processing variation,
- conditioning and non-ambient testing issues,
- alternative coupon configurations,
- data normalization and documentation, and
- application-specific testing.

All significant testing programs should begin with preparation of a detailed test plan document. A test plan specifies material properties to be evaluated, selects tests methods, eliminates options offered by

standard test methods by selecting specific specimen and test configurations, and defines success criteria. It is prepared by the contractor, approved by the certifying agency, and is the focal point for understanding between the contractor and certifying agency. A clearly written, well-prepared test plan is also a primary management tool to define the scope of the work, degree of success, and progress toward completion.

2.2.2 Baseline and alternate approaches for statistically-based properties

Much of MIL-HDBK-17 focuses on guidelines for establishing basis values for strength and strain-to-failure properties[1]. A specific statistical methodology for calculating basis values from test results, illustrated in Figure 8.3.1, has been developed by this handbook, is recommended for general use in reducing data, and is required for evaluation of data published in Volume 2.

Additional requirements imposed on data published within this handbook include: specific population sampling methods and reporting of supporting data. For the purposes of obtaining a reasonable evaluation of material variation, basis values published in this handbook are based on a minimum of thirty specimens from at least five batches of a material per environment and direction as discussed in Sections 2.2.5 and 2.5.3. These data are normalized (where appropriate) as discussed in Sections 2.2.11 and 2.4.3, statistically evaluated in accordance with the process described by Figure 8.3.1 and discussed in Section 8.3, and reported in accordance with Volume 2, Section 1.4.2.

This same statistical procedure can be used on populations of fewer batches and/or replicates, but, if data from such populations are submitted to the handbook for publication, the published data summary will not include a basis value.

Depending on both the application and the procuring or certifying agency, modifications to the baseline MIL-HDBK-17 approach may be justified when developing new material data. In such cases the handbook guidelines remain useful for support and reference. Alternate sampling and statistical approaches to development of basis values may be justified in certain instances, though they are less commonly used. These alternate approaches directly affect test matrix development and generally require a relatively sophisticated knowledge of both statistics and of the material behavior of the specific material system. An introduction to one type of alternate approach is provided in Section 2.3.6.1, with the related statistical background summarized in Section 8.3.5.3. When using such alternate approaches, advance approval of the procurement or certification agency is strongly recommended.

2.2.3 Issues of data equivalence

Evaluation for data pooling (whether data from two possibly different subpopulations are enough alike to be combined) and material equivalence (whether a material with common characteristics to another is sufficiently alike to use its data for design) are similar issues of data equivalence. Both require statistical procedures to assess the similarities and differences between two subpopulations of data[2]. These, and other related issues, are covered in more detail in Sections 2.3.4.1, 2.3.7, and 2.5.3.4. Assessment of the equivalence of data begins by examining key properties for various within-batch and between-batch statistics (see Section 8.3.2).

The ability to pool different subpopulations of test data is highly desirable, if for no other reason than to obtain larger populations that are more representative of the universe (see Section 2.2.5 for a summary discussion of sample size effects). Equally desirable is the ability to show one material without basis values equivalent to another that already has established basis values (see Sections 2.3.4.1, 2.3.7, and

[1]A B-basis value, as defined in Section 1.7, is the value above which at least 90 percent of the population of values is expected to fall, with a confidence of 95 percent. Statistical estimates of basis values for material properties are considered by the handbook to be material properties unto themselves.

[2]If some properties are found similar and others not, engineering judgment must assess the criticality for the given application of the dissimilar properties before the alternate material can be deemed equivalent. The equivalence then only applies to that application and must be reassessed for a different application.

2.5.3.4). Requirements for the use of pooled data or equivalent materials are normally established for each application during discussions with the certifying agency or, for data being considered for publication in MIL-HDBK-17, by the MIL-HDBK-17 Data Review Working Group.

Before determining statistical degree of equivalence, basic engineering considerations should be satisfied; the two materials should be of the same chemical, microstructural, and material form families. To some extent the criteria for this may be application dependent. For example, property data from two composite systems with the same matrix and similar fibers may not warrant pooling if the fiber/matrix interface is distinctly different, even if the fibers have similar modulus and tensile strength. Data equivalence is typically evaluated for data sets that differ due only to relatively minor changes in precursor manufacturing or material processing, such as:

- minor changes in constituents or constituent manufacturing processes,
- identical materials processed by different component manufacturers,
- identical materials processed at different locations of the same manufacturer,
- slight changes in processing parameters, or
- some combination of the above.

Statistical data equivalence methods currently assume that between- and within-laboratory test method variation is negligible. When this assumption is violated this test method-induced artificial variation severely weakens the ability of the statistical methods to meaningfully compare two different detests. This is discussed further in Sections 2.2.4 and 2.2.5.

2.2.4 Test method selection

Test results in an empirical determination of either an intrinsic material property (like material compressive modulus or tensile strength) or a generic structural response (like quasi-isotropic laminate open hole tensile strength) from a small and relatively simple specimen are often used as input to a simulation of the response of a larger and more complicated specific structure. Test methods historically developed for metals or plastics, in most cases, cannot be directly applied to advanced composite materials. While the basic physics of test methods for composites may be similar to their unreinforced counterparts, the heterogeneity, orthotropy, moisture sensitivity, and low ductility of typical composites often lead to significant differences in testing requirements, particularly with the mechanical tests, including:

- the strong influence of constituent content on material response, creating a need to measure the material response of every specimen,
- a need to evaluate properties in multiple directions,
- a need to condition specimens to quantify and control moisture absorption and desorption,
- increased importance of specimen alignment and load introduction method, and
- a need to assume consistency of failure modes.

Other distinguishing characteristics of many composite materials also contribute to testing differences, including:

- compressive strength often lower than tensile strength (though specific material systems like boron/epoxy may behave counter to this),
- operating temperatures relatively closer to material property transition temperatures (compared to metals),
- shear stress response uncoupled from normal stress response, and
- heightened sensitivity to specimen preparation practices.

One measure of a test method is the theoretical ability of a perfect test to produce a desired result, such as a uniform uniaxial stress state throughout the conduct of the test. However, the above factors tend to increase the sensitivity of composites to a wider variety of testing parameters than is seen with conventional materials. Therefore test method robustness, or relative insensitivity to minor variations in

specimen and test procedure, is just as important as theoretical perfection. Robustness, or lack thereof, is assessed by interlaboratory testing, and is measured by *precision* (variation in the sample population) and *bias* (variation of the sample mean from the true average).[1] The precision and bias of test methods are evaluated by comparison testing (often called "round-robin" testing) both within-laboratory and between laboratories. The obvious ideal is high precision (low variation) and low bias (sample mean close to true average) both within-laboratory and between laboratories. Such a test method would repeatedly produce reproducible results without regard to material, operator, or test laboratory. However, quantification of bias requires a material standard for each test; none of which are currently available for composites. As a result, bias of composite test methods can currently only be qualitatively assessed.

Somewhat separate from the precision and bias of a test method (for a given specimen) is the effect on precision and bias of variation in test specimen size and geometry. For heterogeneous materials, physically larger specimens can be expected to contain within the coupon a more representative sample of the material microstructure. While desirable, a larger specimen is more apt to contain a greater number of micro- or macro-structural defects than a smaller specimen, and thus can be expected to produce somewhat lower strengths (though possibly also with lower variation). Variations in specimen geometry can also create differing results. *Size* and *geometry* effects can produce statistical differences in results independent of the "degree of perfection" of the remaining aspects of a test method or its conduct; such effects should be expected. Therefore, even though the specimen response may not (and probably won't) be identical to that of the structure, the "ideal" test method will incorporate a specimen geometry that can be consistently *correlated* with structural response.

As the criticality of various test parameters are still being researched and understood (even for relatively common tests) and as "standard laboratory practices," upon close examination, are actually found to vary from laboratory to laboratory, it is critical to control or document as many of these practices and parameters as possible. ASTM Committee D-30, responsible for standardization of advanced composite material test methods, tries to consider all of these factors when improving existing and developing new standard test methods (see Reference 2.2.4). Due to both their completeness and their status as full-consensus standards, ASTM D-30 test methods, where applicable, are emphasized by this handbook.

Failure to minimize test method sensitivities, whatever the cause, can cause the statistical methods contained within MIL-HDBK-17 to break-down, as all variation in data is implicitly assumed by the statistical methods to be due to material or process variation. Any additional variation due to specimen preparation or testing procedure is added to the material/process variation, which can result in extraordinarily conservative, or even meaningless, basis value results.

Test methods, with emphasis on ASTM standards for advanced composites, are discussed in Chapters 3 through 7. The advantages and disadvantages of the various test methods for composites are discussed, including, for completeness, non-standard but often referenced methods that have appeared in the literature. Chapters 3 and 4 cover constituent testing. Chapter 5 covers prepreg test methods. Chapter 6 covers lamina and laminate testing. Chapter 7 covers structural element test methods. Data produced by the following test methods (Table 2.2.4) are currently being accepted by MIL-HDBK-17 for consideration for inclusion in Volume 2.

[1]The term "accuracy" is often used as a generic combination of aspects of both precision and bias. The terms "precision" and "bias", being more specific, are preferred for use where appropriate.

TABLE 2.2.4 *Summary of test methods for MIL-HDBK-17 data submittal (continued on next page).*

Test Category	Source of Test Method	
	ASTM	**SACMA**
Prepreg Tests		
Resin Content	D 3529, C 613, D 5300	RM 23, RM 24
Volatiles Content	D 3530	---
Resin Flow	D 3531	RM 22
Resin Gel Time	D 3532	RM 19
Fiber Areal Weight	D 3776	RM 23, RM 24
Moisture Content	D 4019	---
Tack	---	---
HPLC	---	RM 20
IR	E 1252, E 168	---
DMA (RDS)	D 4065, D 4473	RM 19
DSC	E 1356	RM 25
Lamina Physical Tests		
Moisture Conditioning	D 5229	RM 11
Fiber Volume	D 3171, D 2734	RM 10
Resin Content	D 3171, D 2734	RM 10
Void Content	D 2584	---
Density	D 792, D 1505	---
Cured Ply Thickness (CPT)	---	RM 10
Glass Transition Temperature, dry	D 4065	RM 18
Glass Transition Temperature, wet	---	RM 18
CTE, out-of-plane	E 831	---
CTE, in-plane	D 696, E 228	---
Equilibrium Moisture Content	D 5229	RM 11
Moisture Diffusivity	D 5229	---
Thermal Diffusivity	E 1461	---
Specific Heat	E 1269	---

TABLE 2.2.4 *Summary of test methods for MIL-HDBK-17 data submittal, concluded.*

Test Category	Source of Test Method	
	ASTM	SACMA
Lamina/Laminate Mechanical Tests		
0°/Warp Tension	D 3039	RM 4, RM 9
90°/Fill Tension	D 3039, D 5450	RM 4, RM 9
0°/Warp Compression	D 3410, D 5467	RM 1, RM 6
90°/Fill Compression	D 3410, D 5449	RM 1, RM 6
In-Plane Shear (1)	D 3518, D 5448, D 5379	RM 7
Interlaminar Shear	D 5379	---
Short Beam Strength	D 2344	RM 8
Flexure (7)	---	---
Open-Hole Compression	(draft)	RM 3
Open-Hole Tension	D 5766	RM 5
Single-Shear Bearing (2)	(draft)	---
Double-Shear Bearing (2)	(draft)	---
Compression after Impact	(draft)	RM 2
Mode I Fracture Toughness	D 5528	---
Mode II Fracture Toughness	(draft)	---
Tension/Tension Fatigue	D 3479	---
Tension/Compression Fatigue	---	---

Notes:
1) ASTM D 4255 will also be accepted for in-plane shear modulus of flat panels.
2) Bearing test procedures are presented in Chapter 7 until the draft ASTM test method that is based on them are released. These Chapter 7 test methods will also be accepted.
3) Certain material forms or processes (like filament winding) may, for a specific material property, be restricted to a single test method. See the detailed test method descriptions in Chapters 3 through 7, or the test methods themselves, for a more complete explanation.
4) SACMA test methods, in many cases, are subsets or supersets of the referenced ASTM test methods, and in other cases have either a different scope or use a different testing methodology. For cases where a SACMA test method exists, and either there is no ASTM test method covering the same property or the existing ASTM test method uses a different methodology, ASTM is considering adopting a form of the SACMA test method. Where ASTM and SACMA test methods overlap, ASTM and SACMA are working to consolidate the test methods into the next release of the ASTM standard.
5) For properties where there are more than one test method listed for either ASTM or SACMA, the different test methods either apply to different material forms or use different testing methodologies.
6) Data from other test methods not listed may be considered by the Testing and Data Review Working Groups, following the guidelines described in Section 2.5.5.
7) See Section 6.7.7.

2.2.5 Population sampling and sizing

Unlike MIL-HDBK-5 for metals, MIL-HDBK-17 for composites does not require simultaneous determination of B-basis values and A-basis values from the same population. This is not because of any fundamental difference in material behavior, but due to a relative lack of need for A-basis properties, to date, for composites. As a result, the composite material B-basis sample population (30+) is much smaller than the MIL-HDBK-5 A/B-basis sample population (100-300) for metals. Unfortunately, since there are usually more composite properties and directions under test, and since testing matrices for composites are often fully populated not only at room temperature but also at the environmental extremes, the total number of specimens in a B-basis composite testing program often exceeds the total number of coupons in an A/B-basis metals testing program.[1] However, included in and allowed by MIL-HDBK-17 are advanced statistical regression techniques that offer the possibility, in specific instances and when combined with different sampling distributions, of being able to reliably determine A-basis values from a total number of composite material specimens similar in quantity to those previously needed for B-basis values (see Section 2.3.6.1).

The sampling approach required for MIL-HDBK-17 B-basis nonregression data, and described in detail in Section 2.5.3, includes at least five batches of production material, using a minimum of 30 specimens distributed among the batches, and fully tests each property at each environment under consideration. The first five prepreg batches are each made using distinct fiber and matrix constituent lots (not required of batch numbers greater than five). For each condition and property, batch replicates are sampled from at least two different test panels covering at least two separate processing cycles. Test panels are non-destructively evaluated using ultrasonic inspection or another suitable non-destructive inspection technique. Test coupons are not extracted from panel areas having indications of questionable quality. A test plan (or report) documents laminate design, specimen sampling details, fabrication procedures (including material traceability information), inspection methods, specimen extraction methods, labeling schemes, and test methods.

For general data development, sampling techniques and sample sizes may be application or qualification/certification agency dependent. A desirable goal of any sampling scheme making use of MIL-HDBK-17 statistical methods is to have multiple batches composed of uniformly-sized subpopulations. The five-batch minimum requirement only applies to material properties that are to be incorporated in MIL-HDBK-17. An alternate number of replicates and batches may be employed upon approval of the procuring or certifying agency. However, mechanical strength data should be evaluated by the statistical methods recommended by this handbook to ensure statistically acceptable basis values.

2.2.5.1 Sample size selection

Regardless of the sampling scheme, for small sample populations, the result of any basis value calculation is strongly dependent on the sample size. Smaller sample populations are obviously less costly to test, but there is a price of a different kind to pay since, as the population size decreases, so does the calculated basis value. Figure 2.2.5.1 shows, for a hypothetical example, the effect of sample size on the calculated B-basis value[2] for samples of various sizes drawn from a given infinite population normally distributed. In the limit, for very large sample sizes, the B-basis (ten percentile) value for this example would be 87.2. The dotted line in the figure is the mean of all possible B-basis values for each sample size; this line can also be interpreted as the estimated B-basis value as a function of population size for a fixed sample coefficient of variation (CV) of 10%. The dashed lines represent the one-sigma limits for any given sample size (a two-sigma limit would approximately bound the 95% confidence interval).

[1]MIL-HDBK-5, the metals handbook, focuses on A-basis values and requires a minimum of 100 tensile specimens, but uses small populations of compressive shear, bearing, and non-ambient tests ratioed to the room temperature tensile properties to estimate compressive, shear, bearing and non-ambient basis values. MIL-HDBK-17 requires at least 30 specimens for each direction, for each property, and for each environment to determine B-basis values. The MIL-HDBK-17 requirement increases to 90 coupons for A-basis values. However, when using MIL-HDBK-17 advanced statistical regression techniques, the specimen populations can sometimes be spread over all of the environments under test, thus reducing the total number of test specimens needed.

[2]Any statistical calculation based on a subpopulation is only an estimate of the real value for the entire population, although the larger and more representative the sample, the better the estimate.

Not only does the estimated B-basis value increase with larger sample sizes, but, as the one-sigma limits illustrate, the expected variation in estimated B-basis value significantly decreases. The lower one-sigma limit is farther from the mean B-basis value than the upper one-sigma limit, illustrating a skew in calculated B-basis value that is particularly strong for small sample sizes. As a result of this skew, for small populations the calculated B-basis value is substantially more likely to be overly conservative than under-conservative, increasing the significant penalty in B-basis value paid by use of small populations. While similar examples for non-normal distributions would have different quantitative results the trends with sample size can be expected to be similar. Additional discussions on effects of sample size are located in Section 8.2.5.

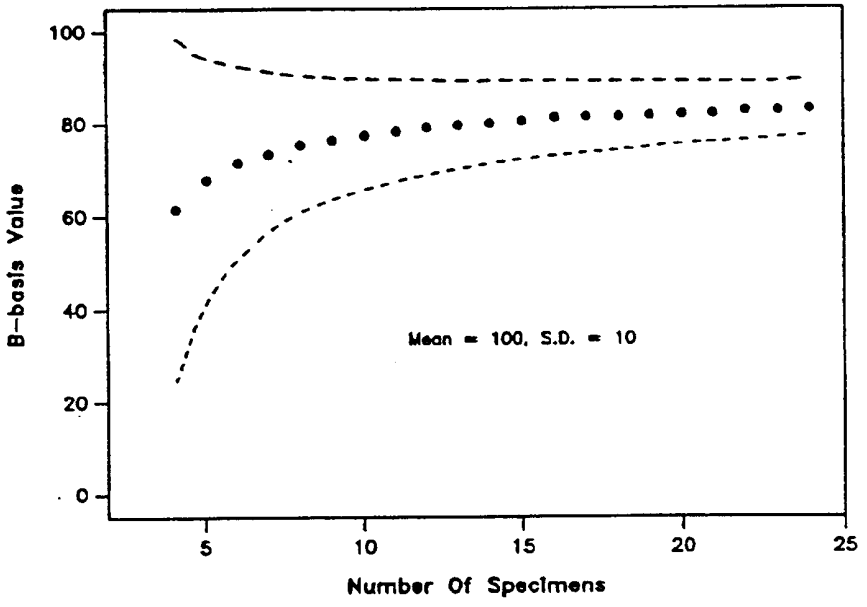


FIGURE 2.2.5.1 *Normal B-basis values with one-sigma limits.*

2.2.5.2 Batch quantity effects on ANOVA

The MIL-HDBK-17 statistical methodology (Figure 8.3.1) includes a statistical test to assess the degree of batch-to-batch variation. If the resulting statistic indicates excessive batch-to-batch variation, the data are not conventionally pooled but are instead evaluated using an Analysis of Variance (ANOVA) approach. However, the statistical methods are only as good as the quality and quantity of data that they evaluate.

Small numbers of batches can cause the ANOVA approach to produce extremely conservative basis values, since it essentially treats the average of each batch as a single data point for input to a conventional normal distribution technique for basis value determination (Section 2.2.5.1 describes the effect of small samples on basis values). As the MIL-HDBK-17 statistical methods assume that testing variation is negligible, variation caused by testing (see related discussion in Section 2.2.4), either within or between batch, is treated as real material/process variation and can result in unrealistically low basis values.

Also, the between-batch variation test becomes progressively weaker as the number of batches decreases, or as the variation between batches decreases, or both. For example, when only a small number of batches are sampled, a batch variation test result indicating no significant batch variation may be deceptive. Additional batch samples may indicate that batch variation really exists, but was masked by the small original number of batches.

The above should be understood when batch variation exists and ANOVA basis values are calculated on fewer than five batches.

2.2.6 Material and processing variation, specimen preparation and NDE

In the sections of Volume 1 that follow in the handbook, the reader will find an extensive compilation of test methods for a variety of fibers, resins and composite material forms and structural elements. In most cases these materials or structural elements are the products of complex multi-step materials processes. Figures 2.2.6(a) and 2.2.6(b) illustrate the nature of the processing pipeline from raw materials to composite end item. (Each rectangle in Figure 2.2.6(b) represents a process during which additional variability may be introduced into the material.) These processes may require elevated temperature, stress or pressure. They often involve evolution of volatiles, resin flow and consolidation, and readjustment of reinforcing fibers. If the measured properties of composite materials are to be interpreted correctly and used appropriately, the variability of the properties of the materials must be understood. This variability arises during routine processing and may be increased by any of the legion of anomalies which may occur during processing.

2.2.6.1 Materials and material processing

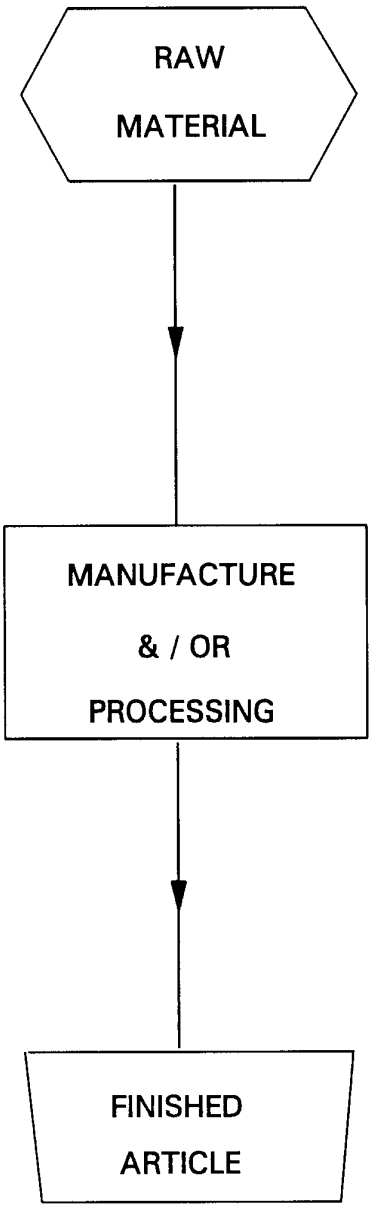
The constituents of the composite materials covered in this handbook are organic matrices (either thermosetting or thermoplastic) and organic or inorganic reinforcing fibers. Variation in the mechanical properties of the reinforcing fibers can arise from many sources, such as flaws in fiber microstructure, or variations in degree of orientation of the polymer chains in an organic fiber.

Thermoplastic matrices can exhibit variations in molecular weight and molecular weight distribution as a result of processing. The melt viscosity and subsequent processability of the thermoplastic matrix may be strongly affected by such variability. Thermosetting resins are often applied to fibers in a prepregging operation and some forms partially cured to what is referred to as a B-stage. Other methods for stabilizing thermoset resin systems may also be employed prior to the prepregging operation. Stability of these materials is important because there are many potential sources of variability during packaging, shipping and storage of improperly, or even properly, stabilized intermediate forms such as prepreg tape, fabrics and roving.

The placement of reinforcing fibers may be accomplished through many manual or automated processes. Lack of precision in fiber placement or subsequent shifting of reinforcing fibers during matrix flow and consolidation can introduce variability. Depending on the process (e.g., pultrusion compared to RTM), cure and/or consolidation can occur simultaneously with fiber placement, or after fiber placement has occurred. This step in the process is especially vulnerable to the introduction of variability.

As an example, consider the cure of a composite part from B-staged prepreg tape in an autoclave, a press or an integrally heated tool. When the resin is heated and has begun to flow, the material consists of a gas phase (volatiles or trapped air), a liquid phase (resin), and a solid (reinforcement) phase. To avoid variability in material properties due to excessive void volume, void producing gas phase material must be either removed or absorbed by the liquid phase. In order to avoid variability due to variations in fiber volume fraction, the resin must be uniformly distributed throughout the part. The fiber must maintain its selected orientation in order to avoid variability or loss of properties due to fiber misalignment.

Pertinent process parameters and material effects should always be documented to aid in process control and troubleshooting. If potential processing and manufacturing pitfalls are not identified and avoided in this way, resources may be wasted in testing materials which are not representative of those which will occur in an actual part or application. In addition, heavy weight penalties may be paid to allow for avoidable material variability. A better understanding of these processing parameters and their potential effect on material properties will also allow a composites manufacturer to avoid the considerable expenses involved in the production of materials, parts or end items with unacceptable properties.



ALL PRODUCTS, REGARDLESS TO STAGE OF WORK, ARE CONSIDERED AS RAW MATERIALS. THESE MAY BE CHEMICALS FOR RESINS, OR SAND TO PROCESS INTO GLASS PRODUCTS, OR PRECURSORS FOR FILAMENTS, OR WOVEN GOODS, OR FLAT PROCESSED SHEETS AND/OR MANY OTHER ARTICLES WHICH HAVE YET TO BE PROCESSED TO AN END ITEM.

EACH RAW PRODUCT THEN IS PROCESSED, OR MIXED WITH OTHER RAW PRODUCTS, OR ALTERED TO BECOME STILL ANOTHER RAW ARTICLE TO EXPERIENCE YET ADDITIONAL PROCESS STEPS THROUGH THE PIPELINE. EACH AS RECEIVED RAW ARTICLE MUST BE PROCESSED IN SUCH A MANNER DURING ITS PROCESSING STEP(S) THAT VARIABILITY IS MINIMIZED AT THE NEXT PIPELINE FUNCTION. PROCESSING FUNCTIONS MAY BE COMPLEX; SUCH AS MATRIX IMPREGNATION, OR THEY MAY BE RELATIVELY SIMPLE; SUCH AS SHIPPING. REGARDLESS, EACH STEP MUST BE EFFECTIVE IN THAT IT DOES NOT INTRODUCE UNCONTROLLED CHANGES THAT ALTER THE PRODUCT FOR SUBSEQUENT USE OR END ITEM PERFORMANCE.

FINISHED ARTICLES LEAVING ONE PROCESSING FUNCTION IS USUALLY STILL CONSIDERED A RAW PRODUCT WHEN DELIVERED TO THE NEXT. THE ONUS FOR CONSISTENCY OF THIS PRODUCT MUST BE RECOGNIZED AND ATTENDED TO DURING THE JUST COMPLETED PROCESSING STEP(S). THE MATERIALS PIPELINE IS NOT COMPLETE UNTIL THE END ARTICLE IS FULLY FUNCTIONAL AS IS.

FIGURE 2.2.6(a) *Composite materials and processing, basic pipeline common to all materials and processes.*

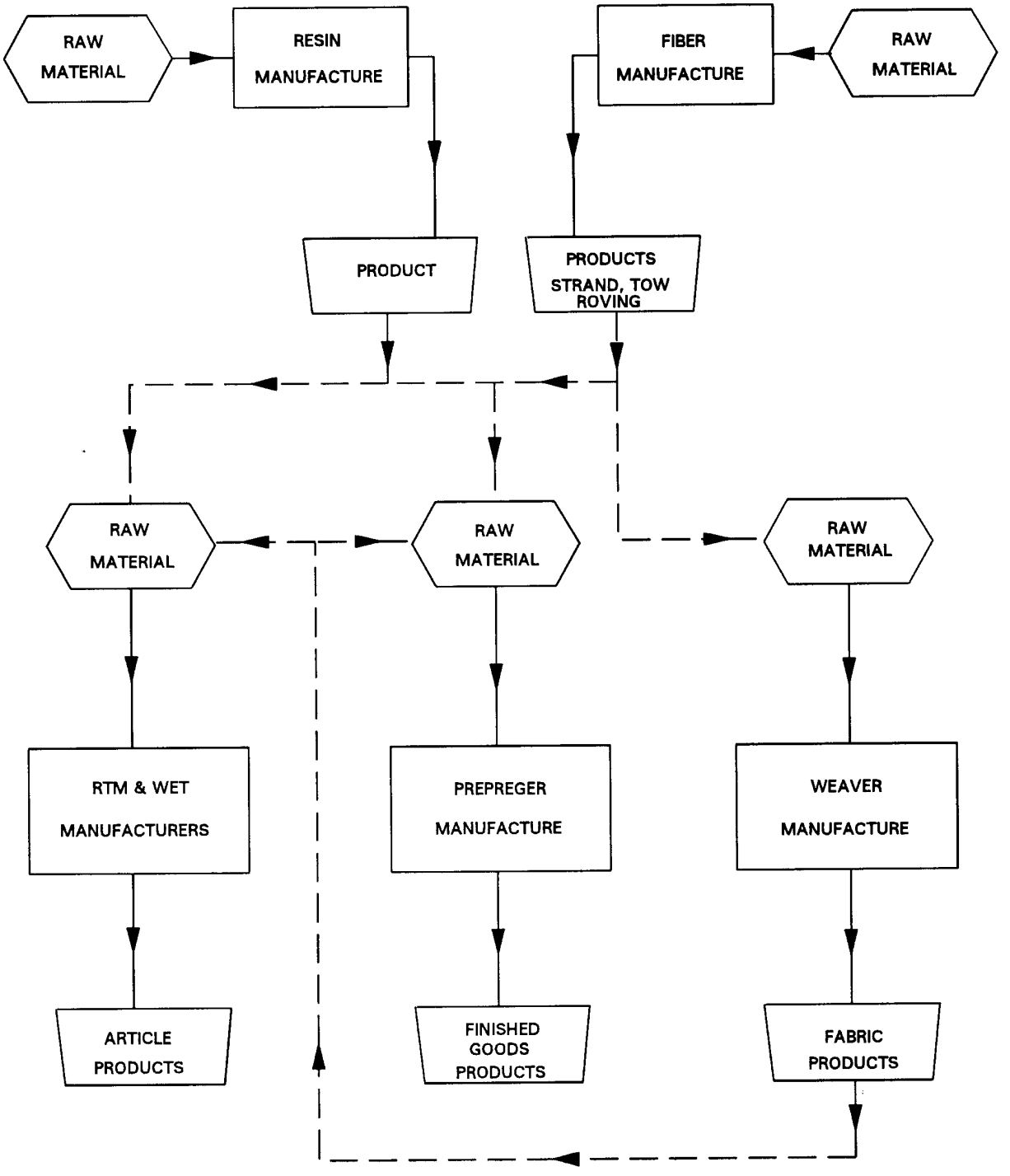


FIGURE 2.2.6(b) *Raw materials pipeline (example).*

This section is meant to be a brief discussion of variability in composite properties arising from the various processes which are encountered in the materials and processing pipeline. For a more extensive and detailed treatment of this subject, the reader is referred to the broader discussion of these issues which may be found in Volume 3, Chapter 2 entitled Materials and Processes - The Effect of Variability on Composite Properties. Volume 3, Chapter 2 also includes a discussion of preparation of materials and processing specifications. The composite end item manufacturer has no direct control over the processing of incoming materials, and the use of such specifications is essential in minimizing materials variability.

2.2.6.2 Specimen preparation and NDE

This section is reserved for future use.

2.2.7 Moisture absorption and conditioning factors.

Most polymeric materials, whether in the form of a composite matrix or a polymeric fiber, are capable of absorbing relatively small but potentially significant amounts of moisture from the surrounding environment.[1] The physical mechanism for moisture gain, assuming there are no cracks or other wicking paths, is generally assumed to be mass diffusion following Fick's Law (the moisture analog to thermal diffusion). While material surfaces in direct contact with the environment absorb or desorb moisture almost immediately, moisture flow into or out of the interior occurs relatively slowly. The moisture diffusion rate is many orders of magnitude slower than heat flow in thermal diffusion. Nevertheless, after a few weeks or months of exposure to a humid environment, a significant amount of water will eventually be absorbed by the material. This absorbed water may produce dimensional changes (swelling), lower the glass transition temperature of the polymer, and reduce the matrix and matrix/fiber interface dependent mechanical properties of the composite (effectively lowering the maximum use temperature of the material---see Section 2.2.8). Because absorbed moisture is a potential design concern for many applications, material testing should include evaluation of properties after representative moisture exposure. Since the amount of moisture absorbed by a material is thickness and exposure time-dependent, fixed-time conditioning methods should not be followed.[2] Instead, a conditioning procedure such as ASTM D 5229/D 5229M (Reference 2.2.7(c)) should be followed that accounts for the diffusion process and terminates with the moisture content nearly uniform through the thickness.[3]

There are two moisture properties of a Fickian material: moisture diffusivity and moisture equilibrium content (weight percent moisture). These properties are commonly determined by a gravimetric test method (such as ASTM D 5229/D 5229M Procedure A) that exposes an initially dry specimen to a humid environment and documents moisture mass gain versus the square-root of time. During early weighings this mass-time relation will be linear, the slope of which is related to the rate of absorption (the moisture diffusivity). As the moisture content in a substantial volume of the exterior of the material begins to approach equilibrium the mass gain versus square-root time slope becomes increasingly smaller. Eventually, as the interior of the material approaches equilibrium, the difference between subsequent weighings will approach zero and the slope will be nearly parallel to the time axis. The weight percent mass gain at this point is the moisture equilibrium content. This process is illustrated in Figures 2.2.7(a) and (b). Figure 2.2.7(a) shows the total mass gain versus root-time during specimen moisture exposure, also showing the difference in response due to different temperatures. For the 150°F condition (the diamonds in Figure 2.2.7(a)), Figure 2.2.7(b) shows the moisture profile through the thickness of the specimen for

[1]While certain polymers, like polybutadiene, resist moisture absorption to the point that moisture conditioning may not be required, these materials are still considered rare exceptions. On the other hand, a great many reinforcements, including those in the carbon, glass, metallic, and ceramic fiber families, are not hygroscopic. As a result, except for polymeric fibers like aramid, it is usually assumed that any moisture absorption is limited to the polymer matrix.
[2]Examples of fixed-time conditioning methods include ASTM D 618 (Reference 2.2.7(a)) and D 570 (Reference 2.2.7(b)) for plastics.
[3]The discussion focuses on through the thickness moisture absorption; however, in-plane moisture absorption will locally dominate near edges, and may even dominate the overall absorption process in those cases where edge area is a substantial portion of the total exposed area. As the in-plane moisture absorption response may be substantially different than the through the thickness response, due to non-Fickian moisture wicking provided by the presence of the fibers, one should not assume that edge effects will be negligible except for very small ratios of edge area to surface area.

several early time periods, illustrating the rapid moisture uptake near the surface together with the relatively slow uptake of moisture in the middle of the specimen.

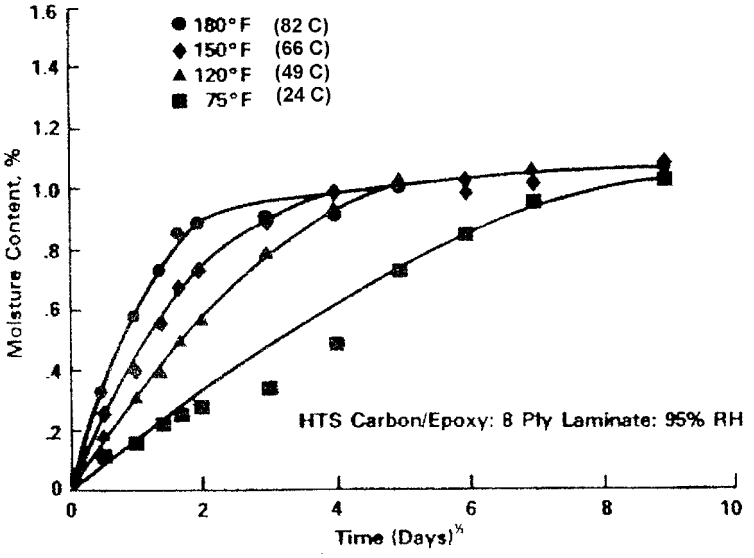


FIGURE 2.2.7(a) *Typical moisture absorption response (Reference 2.2.7(d)).*

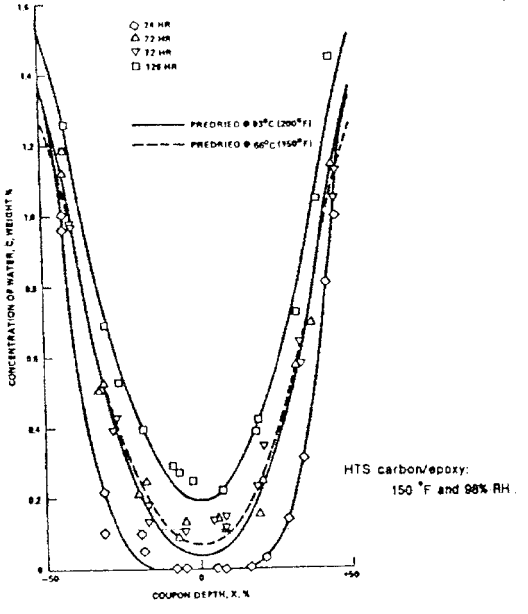


FIGURE 2.2.7(b) *Through the thickness moisture profile versus time (Reference 2.2.7(d)).*

2.2.7.1 Moisture diffusivity

The rate of moisture absorption is controlled by the material property called moisture diffusivity. Moisture diffusivity is usually only weakly related to relative humidity and is often assumed to be a function only of temperature, usually following an Arrhenius-type exponential relation with inverse absolute temperature. This strong temperature dependence is illustrated in Figure 2.2.7.1(a), which shows moisture diffusivity versus temperature for a particular type of carbon/toughened epoxy. Figure 2.2.7.1(b) illustrates, for a different material system, a family of moisture mass gain curves obtained at several temperatures. For this material system, a decrease in conditioning temperature of 60°F (33°C) increased the time required to absorb 1% moisture by a factor of five.

2.2.7.2 Moisture equilibrium content

Moisture equilibrium content is only weakly related to temperature and is usually assumed to be a function only of relative humidity. The largest value of moisture equilibrium content for a given material under humid conditions occurs at 100% relative humidity and is also often called the saturation content. The moisture equilibrium content at a given relative humidity has been found to be approximately equal to relative humidity times the material saturation content; however, as illustrated by Figure 2.2.7.2, this linear approximation does not necessarily hold well for every material system. Regardless, if a material does not reach the moisture equilibrium content for the given relative humidity, then the local moisture content is not uniform through-the-thickness. Another point to be emphasized is that moisture absorption properties under atmospheric humid conditions are generally not equivalent to exposure either to liquid immersion or to pressurized steam. These latter environments alter the material diffusion characteristics, producing a higher moisture equilibrium content, and should not be used unless they simulate the application environment in question.

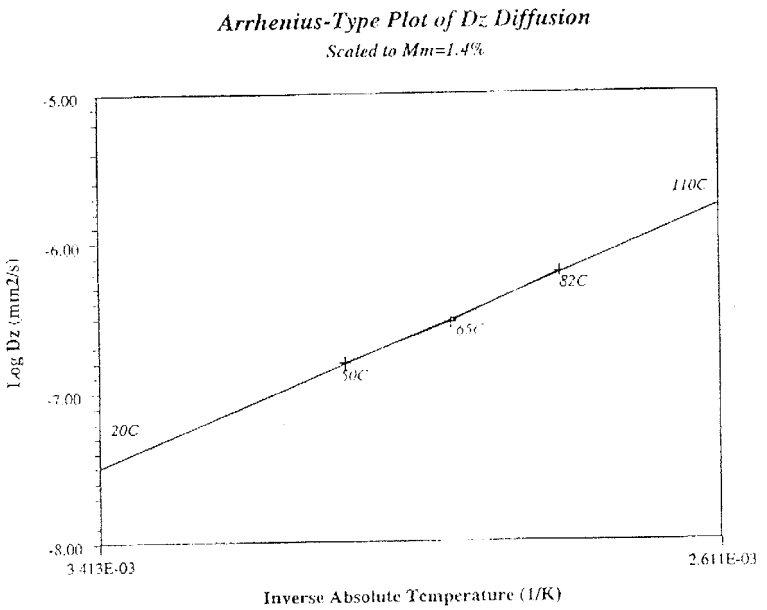


FIGURE 2.2.7.1(a) *Moisture diffusivity as a function of temperature.*

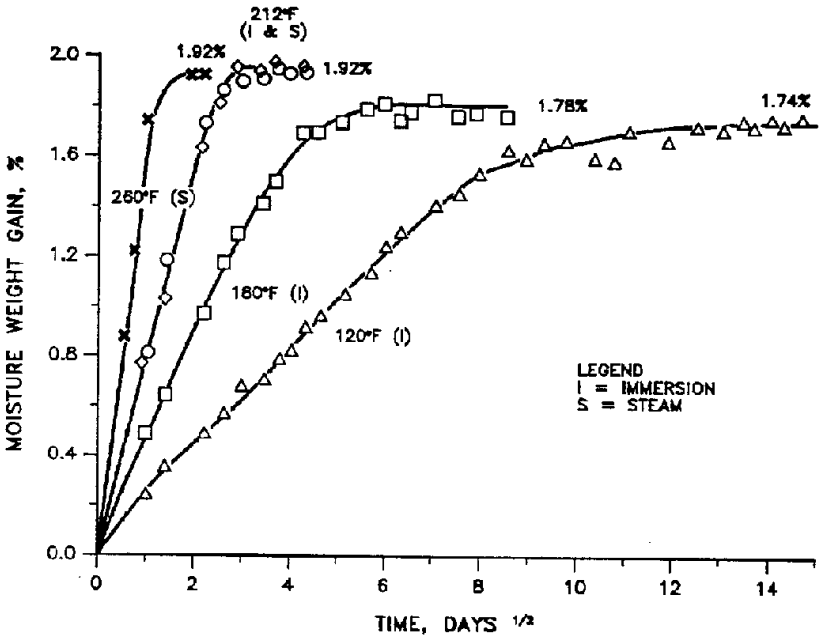


FIGURE 2.2.7.1(b) *Effect of temperature on moisture absorption rate in hybrid boron-graphite/epoxy (5505-AS/3501) laminate (3.0 x 0.5 x 12 in., 76 x 13 x 3.0 mm) (Reference 2.2.7.1).*

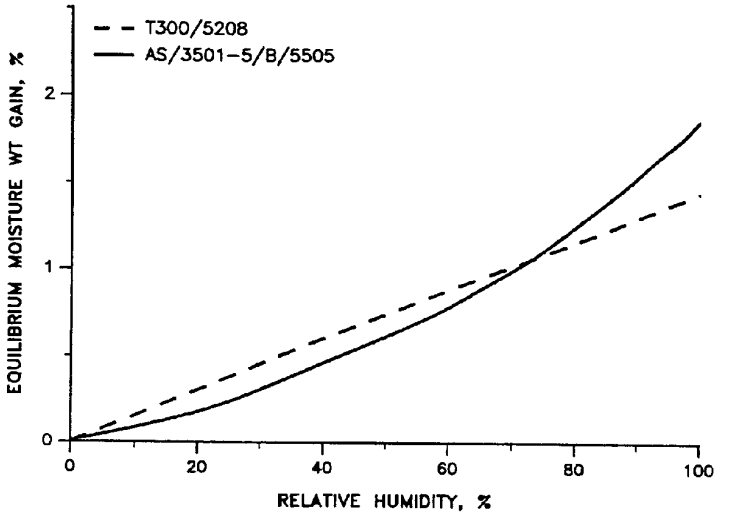


FIGURE 2.2.7.2 *Equilibrium moisture content versus relative humidity.*

2.2.7.3 Conditioning and test environment

To evaluate worst-case effects of moisture content on material properties, tests are performed with specimens preconditioned to the design service (end-of-life) moisture content (assumed equivalent to equilibrium at the design service relative humidity). The preferred conditioning methodology uses ASTM D 5229/D 5229M, the process of which is summarized in Section 6.3.

The design service moisture content is determined (if it is not specified by the procuring or certifying agency) from semi-empirical calculations that consider secondary effects on a particular type of structure, or more conservatively established by simpler assumptions. An example of the first case is documented in Reference 2.2.7.3(a), where worldwide climatic data and USAF aircraft-basing data were combined to define runway storage environmental spectra for each of the three classes of USAF air vehicles: fighters, bombers, and cargo/tankers. The study applied a ranking procedure to select baseline and worst-case locations with respect to the absorption of moisture by typical carbon/epoxy composite structures. Such data can be used to establish design service moisture content for a particular application; a typical specific design service relative humidity might be 81% RH for a tropically-based supersonic aircraft. Another, more conservative, approach is to use the average relative humidity for a selected diurnal cycle taken from a reference such as MIL-STD-210 (Reference 2.2.7.3(b)), the U.S. military guide to worldwide environmental exposure conditions. This usually leads to a higher design service relative humidity (88% RH being typical), since dry-out due to solar radiation, flight excursions (supersonic in particular), and seasonal climatic changes are not considered.

Given these and other historical considerations, the MIL-HDBK-17 Coordination Group has agreed that a reasonable upper-bound value for aircraft design service relative humidity is 85%, and that this value may be used when a specific determination of design service moisture content has not been established for a specific aircraft application. Use of a design service moisture content of 85% RH will obviate extrapolation of data when test specimens are conditioned to equilibrium at this moisture level. Accepted design service moisture levels for other applications have not yet been established.

Hot-wet test data being submitted to MIL-HDBK-17 should have specimens conditioned to an equilibrium moisture content and tested at the material operational limit (MOL) temperature or below (see Figures 2.2.8(a)-(c)). As can be seen in Figure 2.2.8(a), the effect of environment is generally small for matrix-dependent properties at temperatures below room temperature. However, the fiber-dependent properties of many material systems experience a steady degradation with increasingly colder temperatures, though without a cold MOL. A comparison of tensile (fiber-dominated) and compressive (matrix-influenced) response to varying temperature is shown in Figure 2.2.7.3. Due to these factors, qualification/certification testing programs typically do not require moisture conditioning below room temperature, and since there is generally no need to determine a cold MOL, are simply tested at the coldest design service temperature (often -55°C (-67°F)).

2.2.8 Material operational limit (MOL)

As noted earlier, properties of polymer matrix composites are influenced markedly by temperature and moisture. Generally, matrix-dominated mechanical property values decrease with increases in moisture content and increases in temperature above room temperature. For properties that are highly dominated by reinforcement (fiber) properties (unidirectional tension, for example), this reduction may be reversed, not occur, or be minimal over reasonable temperature ranges. For properties influenced by the organic matrix (shear and compression, for example), the degradation of properties can be significant. Furthermore, the degradation is not linear. At a given moisture content, it becomes more severe with increasing temperature until a temperature is reached where dramatic property reductions begin to occur, and beyond which these reductions may become irreversible. It is desirable to specify this onset of dramatic reduction as a "characteristic temperature", which is also defined to be the material operational limit (MOL), or the maximum operating temperature.

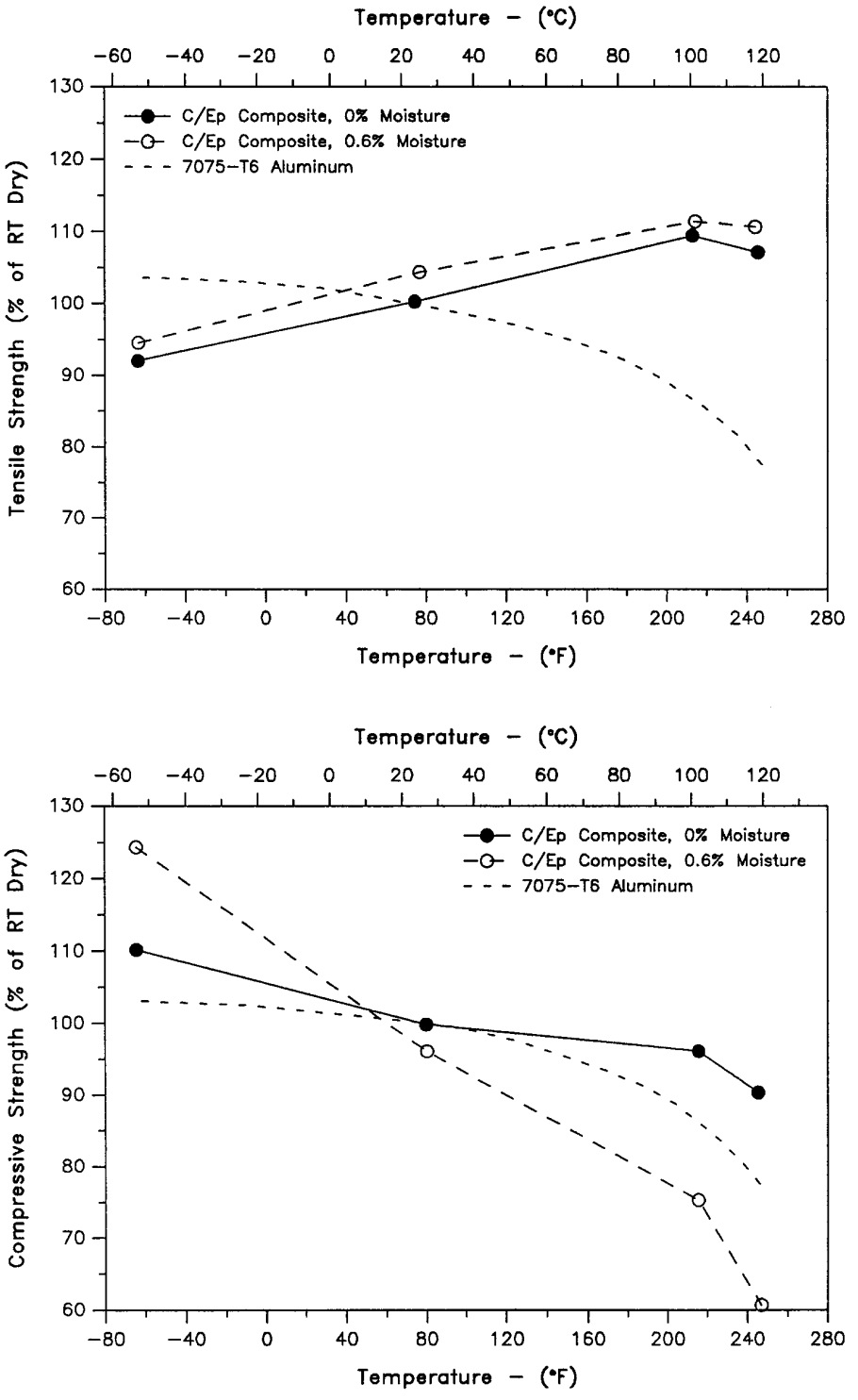


FIGURE 2.2.7.3 *Effect of temperature and moisture on strength (Reference 2.2.7.3(c)).*

The amount of absorbed moisture in the composite has a significant effect on property reduction with increasing temperature. As shown in Figure 2.2.8(a), property degradation at a given temperature is generally more severe with increasing moisture content. Thus, the MOL becomes lower as moisture content increases. Although different MOLs could be determined at a number of moisture levels, the general practice is to establish a single wet MOL at a "worst-case" moisture content. For some applications, a dry MOL may also be established.

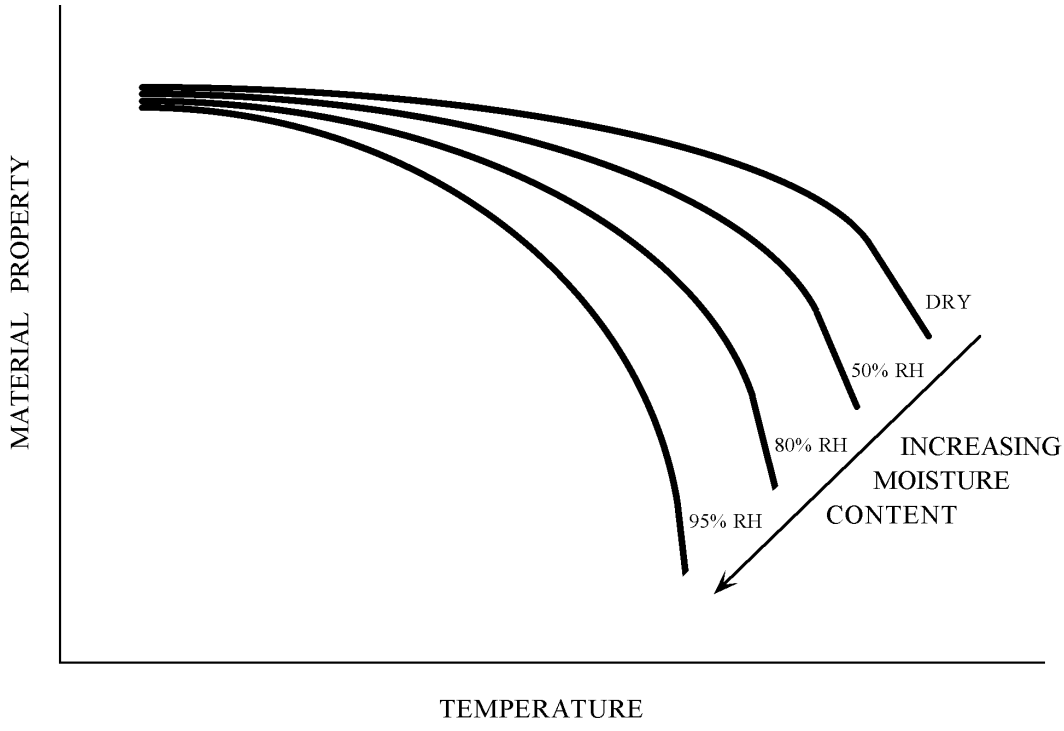


FIGURE 2.2.8(a) *Influence of temperature and moisture on matrix-dependent failure strain.*

The purpose of establishing the MOL is to assure that materials are not operated in service under conditions where a *slight* increase in temperature might cause a significant loss in strength or stiffness, and to absolutely avoid irreversible property changes.

It should be noted that fiber-dependent properties may degrade as temperature decreases below room temperature. However, since these properties do not typically show a sharp falloff as temperature decreases, testing at the lowest anticipated service temperature is sufficient, and there is no need to establish a generic minimum operational temperature, as discussed in Section 2.2.7.3, and illustrated by Figure 2.2.7.3.

Although the upper limits of specific application environments might be below the established MOL temperature(s) for the material(s) used, each material should be characterized at its MOL temperature for a moisture level corresponding to equilibrium at the highest practical relative humidity. For aircraft, 85% is typically considered to be a worst-case relative humidity. Testing at the MOL (in addition to room temperature and cold temperature) will ensure that materials will be used in appropriate applications, and that maximum advantage will be taken of each material's capabilities. Properties at specific application environments may be conservatively estimated using linear interpolation. Limited testing at specific applica-

tion conditions may be added at a program level for verification and reduction of conservatism if required. Figure 2.2.8(b) depicts this process.

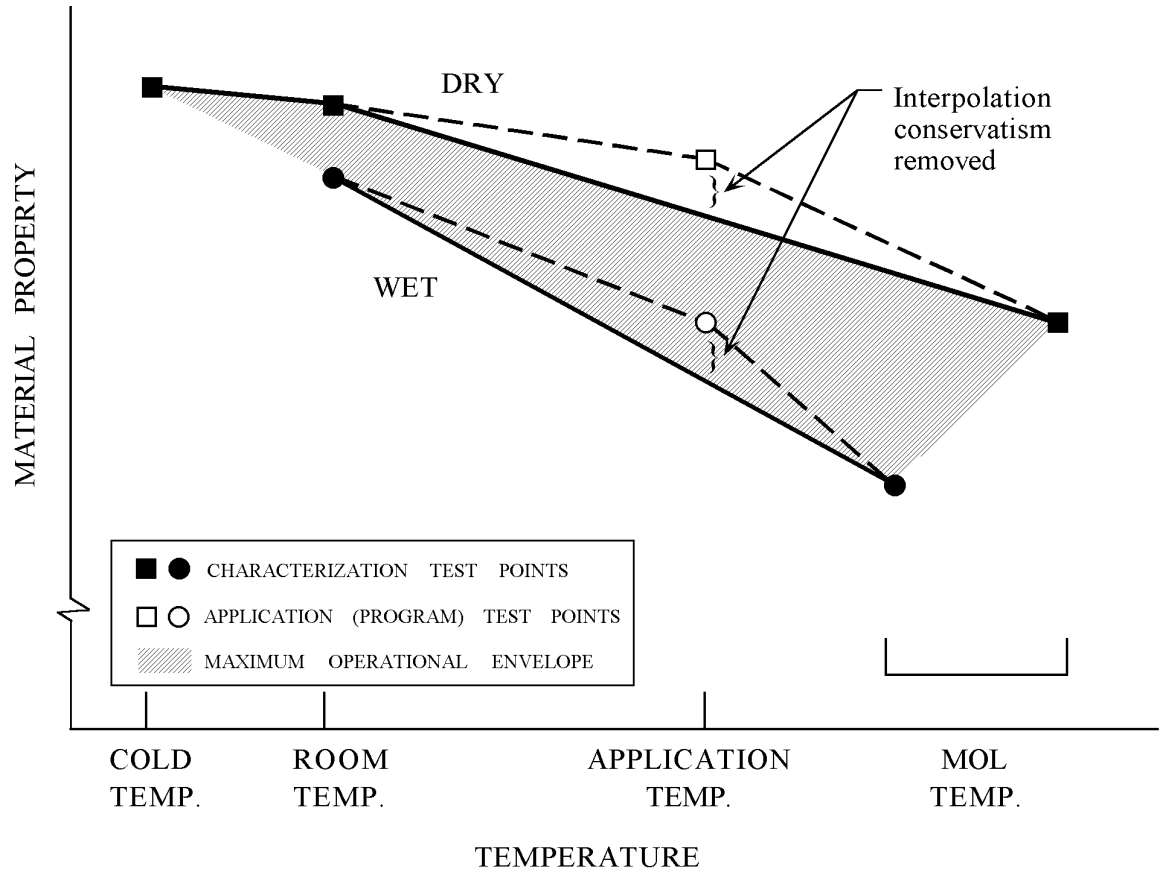


FIGURE 2.2.8(b) *Characterization at limits of material capability.*

There are not yet any fixed criteria for establishment of a MOL. One method (References 2.2.8(a) - 2.2.8(c)) utilizes the glass transition temperature (T_g) as determined from DMA or similar data, reduced by some temperature margin ΔT. For epoxy matrix composites, 50 F° (28 C°) is commonly used for the value of the temperature margin, but it can be argued that smaller margins may be acceptable for particular applications when supported by other data. While glass transition temperature (T_g) is a useful tool, it should not be the sole basis for establishing MOL. Glass transition frequently occurs over a range of temperatures, and it is well known that measurement of T_g is test method dependent (see Section 6.4.3 on Glass Transition Temperature). Other data which are useful in establishing MOL include field experience (for established materials) and mechanical testing conducted over a temperature range which includes the $\pm\Delta T$ range around the measured T_g.

Evaluating the behavior of a matrix-dependent mechanical property (in the appropriate wet condition) as a function of temperature is considered a reliable method for verifying a MOL which has tentatively been determined from T_g data. Various investigators have used short beam strength, in-plane shear strength, in-plane shear modulus, and quasi-isotropic open hole compressive strength for this purpose, with the latter two being most successful as MOL indicators. Four or five temperatures are typically chosen to provide trend lines for the selected property. Figure 2.2.8(c) shows three possible scenarios when

mechanical testing is used to verify the MOL determined from T_g data. In the first instance, mechanical data corroborate the chosen T_g. In the second case, mechanical data suggest that the MOL predicted by T_g is conservative. In the third example, mechanical data do not support the MOL determined from T_g data, and indicate that a lower MOL should be chosen. One approach to determining the MOL from mechanical property data is to use the temperature at which the property versus temperature plot deviates from linearity by a given percentage. An example of this can be found in Reference 2.2.8(d). However, a specific criterion for determining MOL that includes results from both T_g and mechanical testing has not been standardized and is still being discussed. Nevertheless, the MOL value predicted from T_g measurements verified or modified by mechanical property data provides a practical approach for defining the MOL of a material.

The foregoing described a generic approach to MOL, based on T_g and mechanical property reduction. In addition, there are other factors which should be considered, and which might further reduce the effective MOL for specific applications and/or material types. Two such factors are of particular importance: steam pressure delamination and use of "high temperature" composite systems. These are discussed in the following sections.

2.2.8.1 Steam pressure delamination

A moisture/temperature failure mode (no mechanical loads) that must be considered in establishing the maximum operational temperature for a polymer matrix composite laminate is the steam pressure delamination failure (References 2.2.8.1(a) - 2.2.8.1(c)). As previously noted, polymer matrix composites (thermosets and thermoplastics) contain some degree of porosity and absorb moisture. As the matrix absorbs moisture from the environment by the process of diffusion, the voided areas will partially fill with water. If the laminate is exposed to temperatures above the boiling point of water, the water converts to steam. When the temperature and associated steam pressure reaches the level where it exceeds the laminate wet interlaminar (i.e., flatwise) tensile strength of the material, delamination occurs.

The steam pressure delamination mode can occur over a range of temperatures depending on the amount of absorbed moisture as indicated in Figure 2.2.8.1(a). Failure can be predicted when the wet flatwise tensile strength curve (which is a function of the design relative humidity and moisture equilibrium level) intersects the steam pressure curve. To determine the maximum operational temperature for a new material system for a range of design relative humidity, an experimental program similar to Figure 2.2.8.1(b) is recommended.

Panels should be preconditioned to equilibrium at three relative humidity levels plus a dry condition. These panels are then exposed to the mission time-temperature profiles. One issue in conducting the panel thermal exposure test is that the time-temperature exposure should simulate the actual in-service heating conditions so that laminate moisture drying is representative of the design application. Panels that see a slower heating rate than the design condition may have more dryout and attain a fictitious higher temperature before delamination occurs. For high heating rates such as those seen in missile applications, quartz lamps are recommended. For slower heating rates, a computer-controlled oven exposure may be acceptable. The allowable design temperature curve selected should include a safety tolerance (50°F in this example) below the temperature at which delaminations do occur.

2.2.8.2 MOL considerations for high temperature composite systems

The MOL for high temperature composite systems is dependent on other service environment conditions besides moisture. MOL is dependent on the mission life requirements of the actual applications. The life of the part is a function of time, temperature, pressure, and mechanical loading.

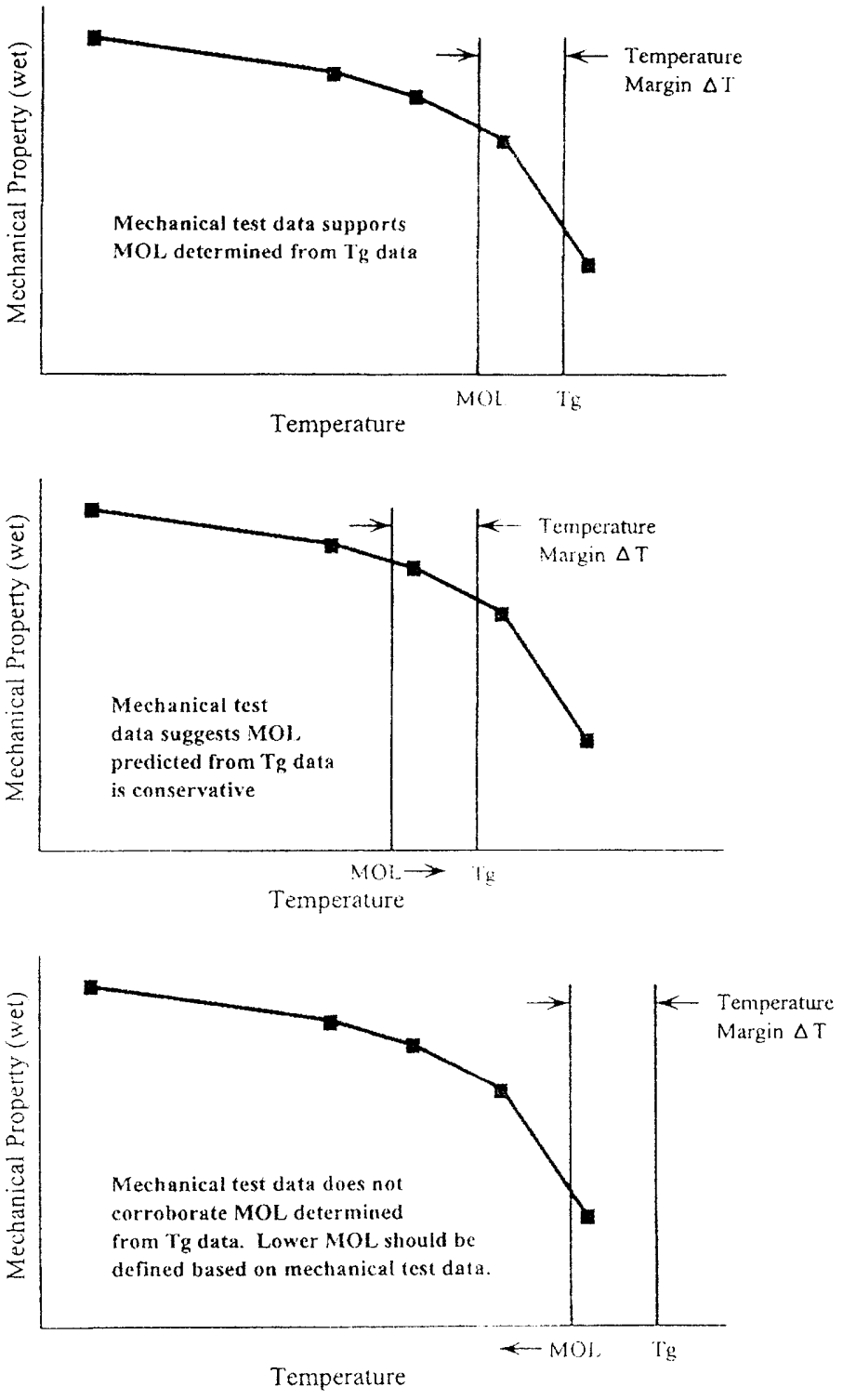


FIGURE 2.2.8(c) *Use of mechanical and T_g data to determine MOL.*

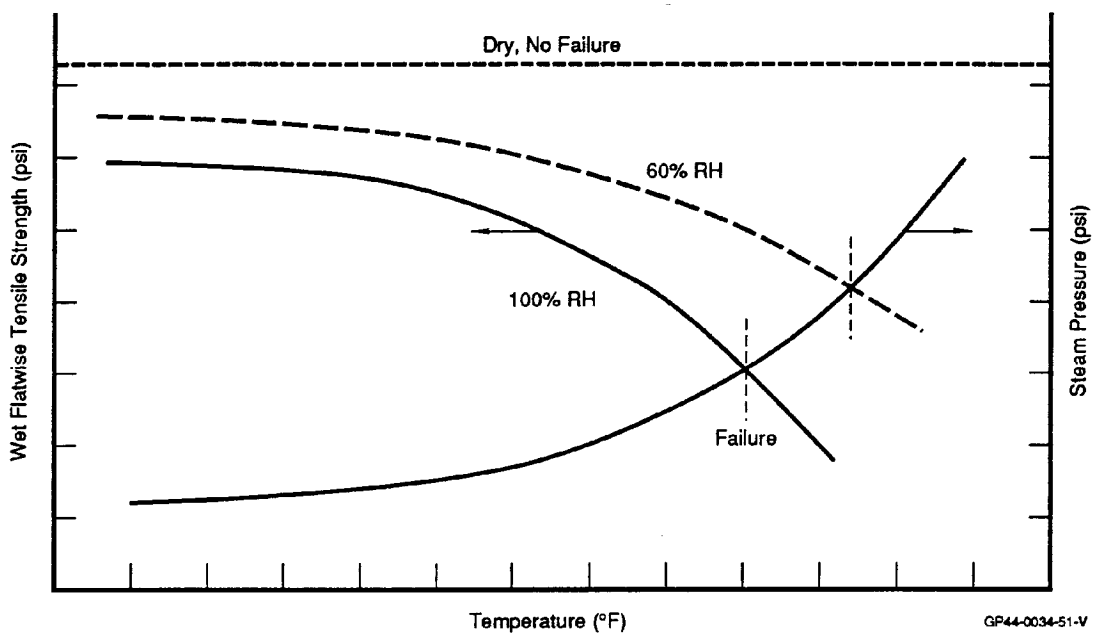


FIGURE 2.2.8.1(a) *Failure occurs when internal steam pressure exceeds flatwise tensile strength.*

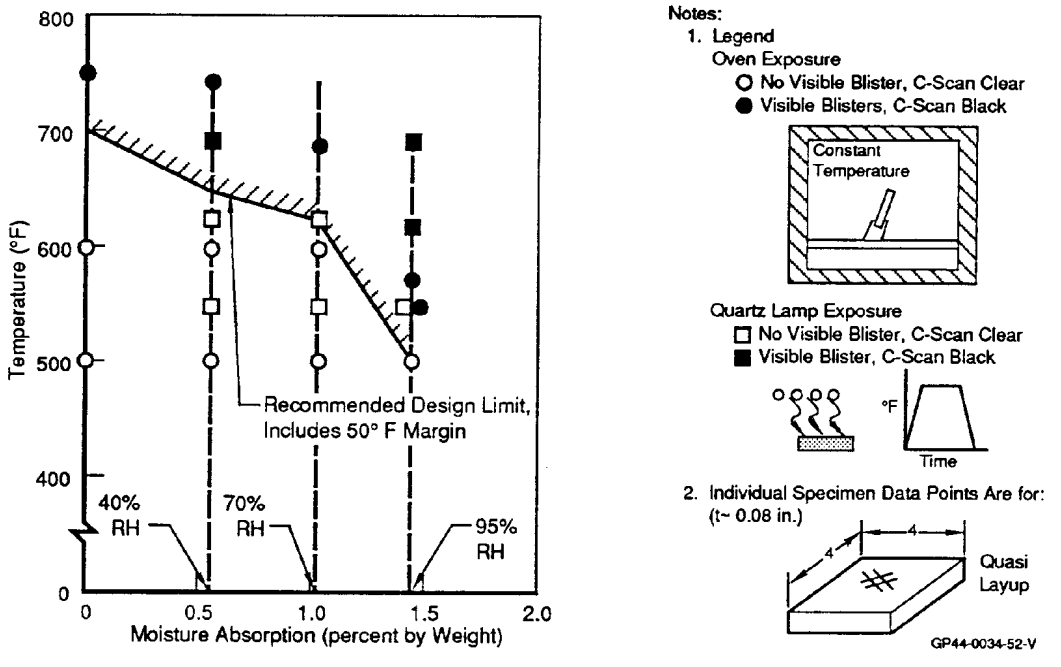


FIGURE 2.2.8.1(b) *Moisture content limits carbon/polyimide use temperature.*

The wet T_g is one of the indicators of a high temperature composite material's MOL. Humidity does affect the elevated temperature properties and can induce thermal blistering in a thick laminate cross section. Thermal blister resistance is a function of the moisture content and thickness of the part, and the heat up rate the part will encounter.

The other indicators of a high temperature composite material's MOL are the transverse microcrack (TVM) resistance and thermal oxidative stability (TOS) properties. TVM occurs due to thermal cycling of the laminate over a temperature range. TVM can develop because of the large difference in coefficient of expansion between the fiber and resin, and the relative low ductility of most high temperature resins. These thermal stresses can cause 90° ply failure, which occurs at the fiber-matrix interface. This degradation primarily affects the resin/interface dominated properties like compression strength, in-plane shear strength, and interlaminar properties. The magnitude of TVM that will occur depends on the application temperature range, maximum operating temperature, and number of thermal cycles.

TOS is a measure of the oxidation rate of materials, and is also an important property for high temperature composite systems. The thermal oxidation characteristics of a polymeric composite are a function of fiber, sizing, and resin. The constituents can be evaluated individually for thermal oxidative stability on a qualitative basis. The actual performance should be evaluated at the laminate level, since the fiber-matrix interface is the primary area that is degraded. All properties can be affected by TOS, although the interface dependent properties are most affected. The weight loss of a laminate is a good indication of the amount of thermal oxidation that has occurred for a particular system, although some mechanical property degradation may occur prior to significant weight loss. The TOS performance of a material is a function of the time, temperature, and oxygen flow rate/pressure.

There can be synergistic effects among TVM, TOS, and hot/wet exposures for high temperature polymeric composites. In order to get an accurate assessment of a material's MOL, it is recommended these effects be combined in a realistic manner that reflects the actual application environment. For short term applications, the amount of degradation can be determined experimentally by exposing laminates to combined conditions of thermal cycling, aging at temperature, and humidity conditioning to the part's specific mission life. Specimens can be machined and tested from this environmentally exposed material, and the residual strength of the material can be assessed.

For long term applications, it may be difficult to perform this environmental exposure in real time. Durability modeling and accelerated testing may be required in order to predict end-of-life properties for these applications. Durability modeling can be used to predict the amount of damage that is generated as a function of mission exposure conditions, and the subsequent residual strength properties. Mission exposure testing can be accelerated by aging the material at higher temperature or pressures, in order to accelerate the oxidation of the material. It is important that the accelerated tests produce realistic damage mechanisms that will be evidenced in real time exposures. For this reason, it is recommended that some limited real time exposure testing be done in order to confirm the damage mechanisms and also be used to confirm the durability model's accuracy.

2.2.8.3 Hot Wet Testing - Report Moisture Content at Failure

Laminate specimens to be static strength tested hot, wet are usually preconditioned to an equilibrium moisture content. Frequently, the test results report the equilibrium moisture content rather than the actual content at failure. This phenomena is illustrated in Figure 2.2.8.3. The sandwich beam specimens with the wet carbon/epoxy facesheet in compression were tested at room temperature (RT) and at 350 °F. The five RT beam compression specimens average moisture content at failure (1.25%) shown in Figure 2.2.8.3 is same as the moisture content before test since at RT there was no measurable dry out during testing. This was verified by cutting out a piece of the facesheet immediately after failure, measure the weight, drying out the piece, and calculating the moisture content at failure. The 350 °F test specimens which were at the test temperature for 9 minutes until failure, dried out ~ 0.5% from 1.25% to 0.75%. The moisture content for each specimen was determined by cutting out a piece of the facesheet immediately after failure, measuring the weight, drying out the piece, and calculating the moisture content at failure. One issue that must be understood is that at the initial moisture equilibrium condition of 1.25 %, every ply

through the thickness of the facesheet is at the 1.25 % moisture content. The 0.75 % moisture level at failure is an average moisture content for a facesheet that has a severe moisture distribution through the thickness (dry on surface, wet at the center).

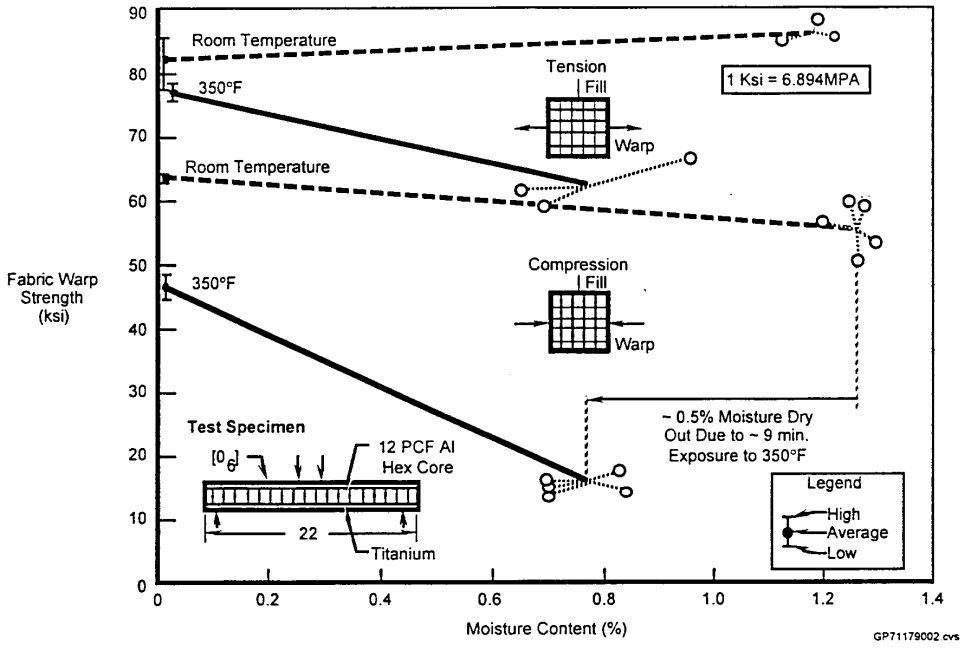


FIGURE 2.2.8.3 *Moisture dry out during hot/wet strength tests.*

The test goal should be to minimize dry out during testing and the potential severe moisture distribution at failure. There are several ways to minimize the moisture dry out during hot wet testing. If the strength tests are being conducted at a test temperature below 212 °F, the before test moisture equilibrium can be maintained (no dry out) by surrounding the specimen during test with a humidity cabinet at the same relative humidity used during the original moisture preconditioning. This method will not work if the test temperature is above 212 °F. Another method to minimize moisture dry out is to minimize the time at the elevated test temperature. Using contact heaters or quartz lamps (instead of forced hot air chambers) can minimize the time for the specimen surface temperature to reach the test temperature. When using accelerated heating devices, the mechanical load should not be applied until the center of the test specimen thickness has been stabilized at the test temperature. Finally, the moisture dry out can also be minimized by selecting a thicker specimen since the dry out occurs in the surface plies first.

Even though steps have been taken to minimize moisture dry out during hot wet testing, the moisture content at failure still must be determined and reported with the strength data. There are three approaches to obtaining the moisture content at failure. One approach is to use moisture monitoring specimens that mimic the material, lay-up, width and thickness of the test section of the test specimen. The monitoring specimen must follow the identical fabrication and moisture preconditioning steps as the test specimen. The monitoring specimen should be in the same test chamber as the specimen to follow the identical thermal history as the specimen during hot wet testing. As soon as the test specimen fails, the monitoring specimen must immediately be pulled out of the hot environment to preclude additional dry out. The specimen is weighed, dried out, re-weighed and the moisture content at failure is calculated. A second approach to obtaining the moisture content at failure is to predict the moisture dry out during hot wet testing using the documented time temperature history during test and subtract this moisture content from the moisture content before test to obtain the moisture content at failure. This approach assumes

that the moisture diffusion constant for the test temperature is known as well as the detail thermal history of the specimen during test. The third approach, which is the most accurate and preferred approach, is to cut a section out of the gage region of the specimen immediately after failure, weigh, dry out, re-weigh and calculate the moisture content at failure.

2.2.9 Nonambient testing

This section is reserved for future use.

2.2.10 Unidirectional lamina properties from laminates

Though feasible, it is frequently difficult to produce valid or reproducible results on mechanical tests of unidirectional single-orientation specimens, particularly at testing laboratories lacking the testing volume to dedicate technicians solely to preparation and conduct of such tests. An alternate approach tests a crossply laminate, usually from the [90/0]ns-family, and calculates via lamination theory an equivalent unidirectional lamina strength and stiffness. Crossply laminates have been found to be much more forgiving of troublesome secondary variations in specimen preparation and testing practice, often yielding higher mean strengths and lower data scatter. The material response of a crossply laminate is also believed by many to be more representative of a structural laminate. The basis of test data reduction for this approach is discussed in Section 2.4.2.

2.2.11 Data normalization

Data normalization is a post-test data manipulation process that attempts to eliminate unrealistic artificial variation in test data caused by local changes in fiber volume. The details of this process of adjusting fiber dominated results to a fixed reference fiber volume is summarized below and described in detail in Section 2.4.3.

Most material properties of composites are dependent on the relative proportion of reinforcement and matrix. In the characterization of properties of a continuously reinforced composite, a portion of the variation of a property value within a like sample population is simply due to locally changing fiber volume, rather than due to any variation in fiber, matrix, or fiber/matrix interface properties. For many composite properties measured in the direction parallel to reinforcing fiber[1], the relation between property and fiber volume is essentially linear. This makes possible a simple adjustment of certain measured properties to a fixed reference fiber volume, resulting in what is called a *normalized* property value.

While a minor variation in fiber volume content may be partly due to variation in the absolute amount of the fiber (and even, to void variation), most fiber volume variation is attributable to locally varying matrix content as a result of processing.

2.2.12 Data documentation

Planning data documentation requirements and methods prior to the start of a test program is a necessary step for the data to be fully useful for its intended purpose. Before deciding on the scope of data documentation, the initial purpose for acquiring the data and potential long-term uses of the data must be determined. Testing requirements may range from obtaining a quick estimate for preliminary material screening where one or more specimens may suffice to establishing long-term material property values for an organization's database and submission to MIL-HDBK-17 where large numbers of specimens may be needed. Different data documentation may be required at different stages of data recording and evaluation. In the laboratory, raw data, even raw transducer signals, may be stored. For evaluated and reviewed material properties, less detailed testing information may be needed, particularly if the information can be traced back to the original source. *Evaluation* is used in the sense defined by ASTM Committee E-49 as “the process of establishing the accuracy and reliability of property data” (Reference 2.2.12(a)). The expectation for MIL-HDBK-17 data is that documentation should be sufficient so that

[1]The so-called "fiber-dominated" properties.

1. Traceability and a historical record (identified by manufacture, testing, and evaluation dates) are established.
2. The material pedigree and associated process specification are well defined.
3. Testing procedures can be identified.
4. Variables that influence test results are identified.

Other programs that establish reference data should have documentation requirements at least as stringent as those in Table 2.5.6.

In any testing program, for future use of the data to be possible, data documentation should be complete enough so that the material and testing can be reproduced.

Documentation may be recorded in a computerized database for raw data or in a lab notebook or other hard copy form. The earlier computerization is implemented in the process of storing and evaluating data, the easier it is to maintain traceability and limit transcription errors. The use of computers does not eliminate the need for error checking and review. Regardless of how much computerization is involved in the process, each organization should have its own protocol for data recording and review. Considerations for such a protocol include who is responsible for recording particular information - material identification, processing, specimen preparation, inspection, testing, and archiving of failed specimens and contact information for each of these steps. It is quite common for each testing laboratory to have limited access to material identification information. In this case, responsibility for coordinating material identification information with testing laboratory data records must be established if any long-term use of the data is planned.

Guidance for establishing data documentation requirements is available from several sources. MIL-HDBK-17, Volume 1, Section 2.5.6 lists data documentation required for data submission to the handbook as well as a few items that are recommended for in-house recordkeeping. Two ASTM guides (References 2.2.12(b) and (c)) provide primary guidance for data recording and database development. ASTM E 1309 addresses identification of all composite materials. ASTM E 1434 provides guidance for test information and results for mechanical properties of continuous-fiber polymer matrix composite materials. These two documents should be used together in a modular approach. First the material is identified and then the test method and results information is recorded. Each data element has a level of importance established by the following categories:

- required for test validity
- required for material traceability
- recommended for test validity
- recommended for material traceability
- optional

These levels of importance divide data documentation requirements into two subsets: one for material traceability and one for test validity. A test laboratory can meet requirements for test validity without access to material identification information. Responsibility for the material traceability should be assigned to someone who does have access to the necessary information (as noted above). The approach used in developing these and similar ASTM guides is to provide assistance for data recording and for the contents of computerized databases without unduly limiting database structure.

These ASTM guides are implicitly included in the MIL-HDBK-17 data documentation requirements, since Table 2.5.6 specifies that all sections of the test method are to be followed unless deviations are reported. Several of the ASTM methods for mechanical properties refer to E 1309 and E 1434 for data recording. Consequently, the reference flows from Table 2.5.6 to the ASTM test methods to the data-recording guides.

Another source for guidance is the Composite Material Test Data Schema (CMTDS) developed within MacNeal Schwendler's MVISION™ database structure (Reference 2.1.12(d)). Focused on continuous-fiber polymer matrix composites, this schema establishes specific fields in a defined database structure

including field names, synonyms, and values sets for expected field entries without defining levels of requirements. A much larger range of potential properties, including thermal and physical properties, is incorporated. The recommendations of MIL-HDBK-17 and the ASTM guides were considered in the development of the schema, which functions both as a source of guidance and a specific implementation of more general recommendations. The released version addresses test data. A draft schema for property level information is under development. The experience of the CMTDS development is serving as feedback into revision of ASTM E 1309 and E 1434 currently underway.

General guidance on the development of material property databases is found in References 2.2.12(e) and (f).

2.2.13 Application specific testing needs

This section is reserved for future use.

2.3 RECOMMENDED TEST MATRICES

2.3.1 Material screening test matrices

The objective of the screening process is to reveal key mechanical property attributes and/or inadequacies in new material system candidates, while keeping testing to a minimum. The screening process identifies, for a particular composite material system, the critical test and environmental conditions as well as any other special considerations. Proper test matrix design enables comparison with current production material systems.

The general approach to screening test matrix design is selection of key static tests that provide sufficient data to assess mean values of stiffness and strength at both the lamina and laminate levels. The lamina-level tests provide intrinsic material stiffness and strength properties commonly used in classical lamination plate point stress analysis, including tension, compression, and shear loadings. Both the lamina-level tension tests and open-hole compression tests are also performed at key environments. The laminate-level tests provide screening strength data on application issues relating to stress discontinuities such as fastener holes, bolt bearing, or impact damage. Additional laminate level tests provide screening stiffness data to verify the use of lamina data with classical lamination plate theory for laminate stiffness predictions. Tests are generally performed at room temperature. Environmental effects are estimated from the key lamina-level tension and open-hole compression tests.

An example of a typical mechanical property screening test matrix is shown and discussed in Section 2.3.1.1. Under extreme environments, additional factors may have to be considered, as discussed in the example for high-temperature materials in Section 2.3.1.2. Sensitivity to exposure to operational fluids and other special issues may justify including additional special tests in the screening evaluation. An example of a fluid sensitivity screening test matrix is shown and discussed in Section 2.3.1.3. (Specific applications may require modifications to the above test matrices.)

2.3.1.1 Mechanical property screening

Table 2.3.1.1 shows a recommended mechanical property screening test matrix developed for epoxy-based resin systems but also useful for other material systems. In the screening test matrix, 0° axial tensile tests examine fiber dominant properties and 0° axial compression tests monitor fiber/matrix interactions;[1] both provide static strength and stiffness properties. The ±45° tensile test specimens are used to evaluate matrix characteristics, determining shear modulus and effective shear strength. Finally, damage resistance is assessed using compression after impact testing. The testing is conducted under three environmental conditions: cold temperature ambient (CTA), room temperature ambient (RTA), and elevated temperature wet (ETW). These test conditions are recommended based on results for current epoxy

[1]The 0° axial tensile tests may reveal fiber/matrix interaction in some materials at high strain rates.

resin systems that show the CTA environment as critical for fiber dominated properties and the ETW environment as the most severe condition for matrix dominated properties. The ETW specimens are conditioned to moisture equilibrium at the specified relative humidity.

TABLE 2.3.1.1 *Composite material static strength screening test matrix.*

Test	Number of Specimens			Evaluation Emphasis
	CTD	RTA	ETW	
Lamina:				
0° Tension	3	3	3	fiber
0° Compression		3		fiber/matrix
±45° Tension		3		fiber/matrix (0°/90° shear - lamina) (±45° - laminate)
Laminate:				
Open Hole Compression[1]		3	3	stress riser
Open Hole Tension[1]		3		stress riser
Bolt-Bearing[1]		3		bearing
Compression after Impact[2]		3		impact damage

[1]Fastener hole effects
[2]per NASA Reference Publication 1092.

2.3.1.2 Mechanical property screening for high-temperature material systems

Table 2.3.1.2 shows a typical mechanical property test matrix intended for high temperature polymer matrix composites. The changes were made to Table 2.3.1.1 in order to properly assess the durability of high temperature polymer matrix composites during the screening stages of an evaluation. The test matrix may vary depending on the purpose of the investigation, but it is important that all exposure conditions be evaluated.

Prior to the mechanical test evaluation, it is necessary to evaluate prepreg physical and laminate properties. Test laminates should be carefully inspected for porosity content, dry T_g, and wet T_g. The recommended mechanical tests cover fiber-dominated, interface/resin-dominated, and damage tolerance properties. The elevated test temperature static test conditions should be set below the wet T_g of the system.

The wet exposure condition is 160°F (71°)/ 85% relative humidity to an equilibrium weight gain. It is very important that specimen dry-out be measured and kept to a minimum during the elevated temperature wet tests.

TABLE 2.3.1.2 *Composite material test matrix for high temperature PMC's.*

Mechanical Property	Dry Test Temperature			Wet[1]	TOS[2]	Thermal Cycle[3]
	Minimum Temp	75°F (24°C)	ET1	ET1	ET1	ET1
Tension	3	3	3	-	3	-
Compression or OHC	-	3	3	3	3	3
In-Plane Shear	-	3	3	3	-	3
Mode I Fracture Toughness or CAI	-	3	3	-	3	3

1 - ET1 Elevated test temperature should be less than the Wet Glass Transition Temperature of the material
2 - Laminates are to be thermally aged at a temperature greater than ET1 but less than the dry T_g for a minimum of 1000 hours or an accelerated test condition that represents a 1000 hour exposure. Weight loss should be recorded as a function of time, i.e., 100, 250, 500, 750 and 1000 hours. Microscopy should be performed after exposure. Specimens are to be machined after exposure.
3 - Laminates are to be thermal cycled from Min Temp to a temperature greater than ET1 but less than the dry T_g. Laminates are to be cycled for a minimum of 500 thermal cycles. Microcrack density is to be measured after cycling. Specimens are to be machined from the laminate after exposure.

The thermal oxidative stability (TOS) test should be performed for a minimum of 1000 hours. Weight loss should be measured during testing at specified intervals of 100, 250, 500, 750, and 1000 hours. This test provides a measurement of the oxidation rate of the material.

Thermal cycling should be done for a minimum of 500 thermal cycles. The purpose of the test is to determine the rate of microcracking, not only if microcracking will occur. The minimum temperature should represent the minimum temperature of the potential application, for example, -65°F (-54°C) for aircraft.

The maximum exposure temperature for both the TOS and thermal cycling test should be between the wet T_g and dry T_g of the material system. If the exposure temperature is below the wet T_g, the test may not be discriminating enough and longer exposure times may be necessary. Exposures above the dry T_g of a material normally provide an unrealistic damage mechanism that does not occur below the dry T_g of the material. Prior to the machining of specimens, laminates should be non-destructively inspected for porosity and delaminations. Microscopy should also be done in order to understand the damage mechanism associated with the specific exposure. This will include the measurement of microcrack density.

2.3.1.3 Fluid sensitivity screening

Historically, the concern over exposure of structural composite materials to commonly encountered service related fluids other than water or moisture has not been a major concern. This is because the majority of structural composites have had an epoxy resin matrix which has inherently been very fluid resistant. With the epoxies, in general, the allowance for property degradation caused by absorption of atmospheric moisture has been sufficient to cover degradation which might be caused by other pertinent fluids, e.g., fuel, hydraulic oil, etc. Although epoxy resin systems are subject to accelerated degradation in the presence of highly acidic media, the majority of service fluids tend to be basic in nature, e.g., clean-

ing solvents and hydraulic fluids. The poor resistance of epoxies to methylene chloride, a common ingredient in paint strippers, is an exception. Methylene chloride also aggressively attacks other structural polymers. Consequently, the use of chemical paint strippers on polymer matrix composites is generally not allowed.

With due consideration of the above, it is still important to evaluate the resistance of new polymer materials to fluids with which they might come in contact. Many new epoxies have components, added to improve properties such as toughness, which might affect their solvent resistance. Many other polymers, which have different solvent sensitivities, are also now being used or are being considered for use. An example of a problem encountered in the past was that associated with the developmental evaluation of polysulfone thermoplastic structural parts and their abandonment due to poor resistance to phosphate ester based hydraulic fluids (Reference 2.3.1.3(a)). Some other structural thermoplastics, although they have excellent resistance to moisture and hydraulic oils, have poorer resistance to fuels. Fuels with higher aromatic content, e.g., JP-4 as compared to JP-8, seem to cause the worst problems (Reference 2.3.1.3(b)). In the referenced case, the fuel exposure seemed mostly to effectively lower the material's (PEEK) glass transition temperature (Reference 2.3.1.3(c)). The result was comparable lowering of the material's maximum use temperature.

Higher service temperature resin systems such as bismaleimides (BMI's) and polyimides are susceptible to degradation by fluids with high alkalinity. Both polymer formulations are susceptible to a cleavage of the functional imide rings in the presence of high concentrations of hydroxide ions. This is significant for two reasons. First, cleaning solvents and hydraulic fluids used by most airlines are alkaline by nature and second, hydroxide ions are produced locally at the resin boundary during galvanic coupling between carbon fibers and active metals and can cause degradation. The galvanic corrosion situation should be satisfactorily manageable with an attentive design. The incorporation of an isolation mechanism such as a resin/fiber ply between the carbon/resin and metal structure is one approach to mitigating the risk associated with the electrolytic driven degradation. Exposure can be lessened by providing drainage, etc. It is important that the laminate edges be well sealed if there is exposure in a sump area. In general, the exposure of these materials to alkaline solutions may be sufficiently incidental that this also may not be a problem.

The following evaluation procedure is suggested to assess the suitability of polymer resin systems for application where they might be exposed to a harmful fluid environment.

The evaluation should account for different exposure levels of aircraft structure to fluids. Two fluid exposure classifications are suggested, with example fluids cited for each group:

Group I

Fluids that have the potential for *pooling* or will contact the material for an extended period of time.

JP-4 Jet Fuel	MIL-T-5624
JP-5 Jet Fuel	MIL-T-5624
JP-8 Jet Fuel	MIL-T-83133
Hydraulic Fluid	MIL-H-5606
Hydraulic Fluid[1]	MIL-H-83282
PAO (Poly Alphaolefin) Cooling Fluid	MIL-C-87252
Engine Lubricating Oil	MIL-L-7808
Engine Lubricating Oil	MIL-L-23699
Ethylene Glycol/Urea Deicer (Class I)	SAE AMS 1432 (superseding MIL-D-83411)
Sump Water[2]	MIL-S-8802 section 4.8.15
Methylene Chloride[3]	ASTM D4701 (superseding MIL-D-6998)
SO2/Salt Spray[3]	---

[1] Monsanto low-density aviation hydraulic fluid, commercial.
[2] A mixture of SAE AMS 2629 Jet Reference Fluid and 3% sodium chloride/water solution.
[3] U.S. Navy requirement.

Group II

Fluids that are *wiped on and off* (or evaporate) or will not contact the material for an extended period of time.

Alkaline Cleaner (Types 1 and 2)	MIL-C-87936
MEK Washing Liquid	ASTM D740 (superseding TT-M-261)
Dry Cleaning Solvent (Type 2)	P-D-680
Hydrocarbon Washing Liquid	TT-S-735
Polypropylene Glycol Deicer (Type 1)	MIL-A-8243
Isopropyl Alcohol Deicing Agent	TT-I-735

More information on these fluids is found in References 2.3.1.3(d) - (t).

Exposure by immersion prior to test or to evaluate weight loss is also recommended, using a different exposure level for each group:

Group I Immerse material in fluid until it reaches equilibrium weight gain (saturation). (Except for the MIL-S-8802 Sump Water corrosion test.)

Group II Immerse material in fluid for 15 days to determine worst case effects. Then follow-up with tests that simulate a more realistic exposure including accidental extended exposure.

Both mechanical and physical testing should be done. Mechanical testing should include open hole compression tests on quasi-isotropic lay-up specimens and ±45° tension specimens. The open hole compression test has a meaningful relation to design values and is sensitive to matrix degradation. The use of a ±45° tension test is commonplace in industry for comparison of matrix properties. It is a sensitive test which will identify "potentially" harsh fluids. It provides an indication of whether necessary shear stiffness has been retained to ensure acceptable resin to fiber property transfer. While a material stiffness loss criterion is material and application specific, a 20-40% loss in shear modulus from that of the unexposed material is generally considered significant, and should be further investigated. A minimum of five specimens should be tested after exposure at room temperature and at the maximum use temperature. The results should be compared with unexposed controls.

A more economical alternative to open hole compression and ±45° tension testing is interlaminar or short beam shear tests. These specimens are easily fabricated, machined, conditioned, and tested. Although not as generally related to design properties, short beam strength tests are sensitive to matrix degradation and can be valuable indicators for material evaluation. As with the ±45° tension tests, results after exposure should be compared to unexposed controls at room and elevated temperature to obtain fluid exposure effects.

Physical testing should include weighing to measure weight change, photomicrographs to examine for microcracks, and, where practical, scanning electron microscopy to examine for surface crazing. Relative to the former, it must be warned that because a saturation condition has apparently been reached, it *does not* automatically follow that further degradation of properties has ceased. Especially where new resin systems are involved, test with long term exposure to critical fluids should be conducted. An example of such testing is given in Reference 2.3.1.3(u). Due to the long exposure times involved, these tests should be started early in the evaluation process.

It has been the procedure in the past that if water or moisture has been proven to be the most property-degrading fluid, then fluid exposure tests involving other than moisture conditioning were not included in subsequent design testing. Such a procedure for Group I pooling fluids is presented in Figure 2.3.1.3(a). In effect, if the properties of the material after fluid exposure are better than after moisture exposure, then subsequent testing accounts for moisture only. If a fluid other than water is more critical, then subsequent testing must include evaluation with that fluid.

In the case of Group II wipe on/wipe off fluids, the procedure is somewhat different since water is not a good comparison. Consequently, comparison to a resin that has an acceptable service history is recommended. This is illustrated by the decision tree in Figure 2.3.1.3(b) where comparison with the performance of 3501-6 epoxy resin system is suggested.

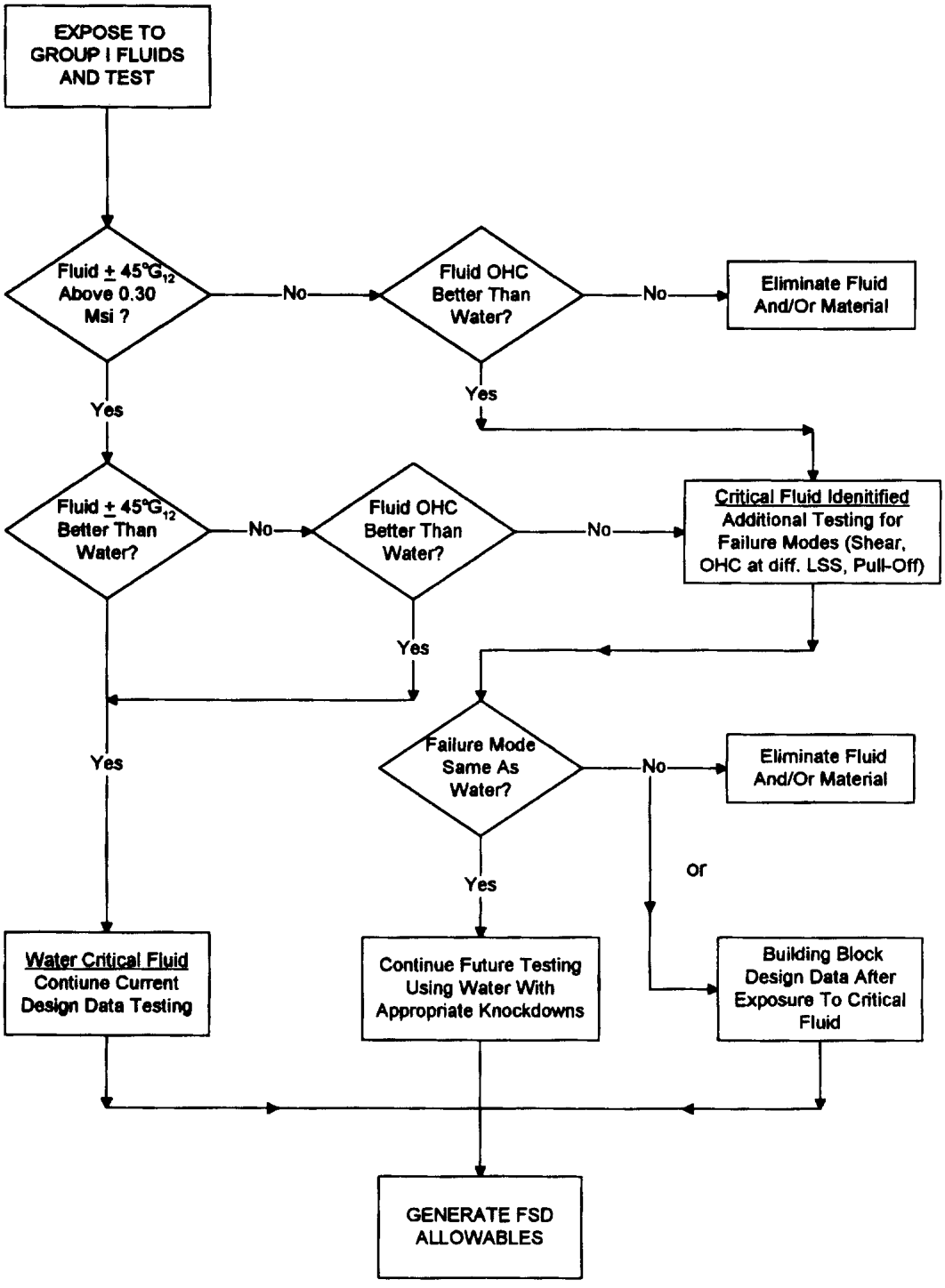


FIGURE 2.3.1.3(a) *Decision tree to closure - Group I pooling fluids.*

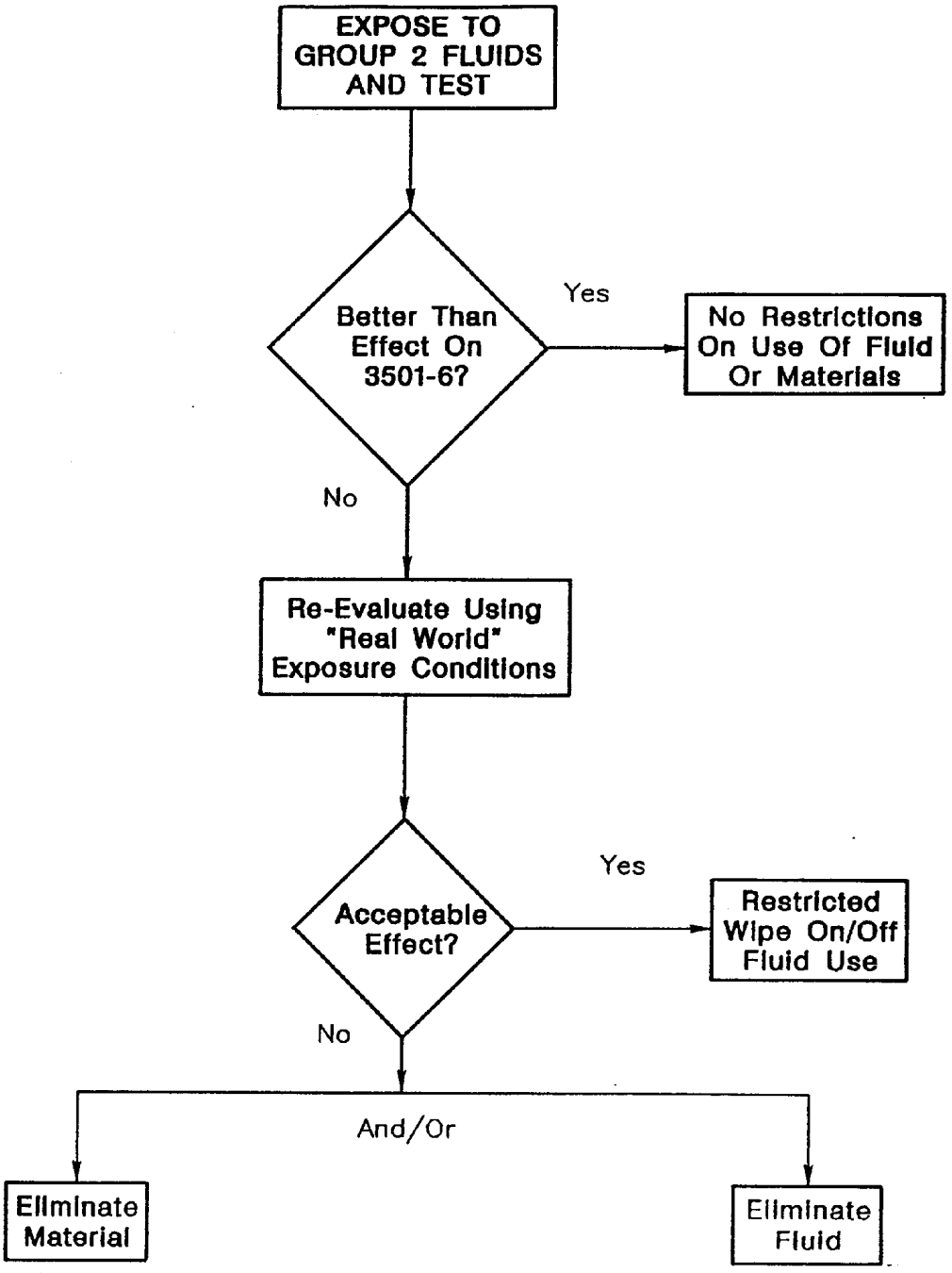


FIGURE 2.3.1.3(b) *Decision tree to closure - Group II fluids without long term exposure.*

2.3.2 Material qualification test matrices

2.3.2.1 Constituent test matrix

This section is reserved for future use.

2.3.2.2 Prepreg test matrix

The recommended test matrix for prepreg materials is shown in Table 2.3.2.2. The table is based on thermosetting matrices and requires modification for thermoplastic matrices.

TABLE 2.3.2.2 *Recommended physical and chemical property tests to be performed by material supplier and prime contractor.*

Test Property	Suggested Test Procedure[1]	Number of Tests per Batch[2]	Total Number of Tests
Resin Content	ASTM D 3529	3	15
Volatile Content	ASTM D 3530	3	15
Gel Time	ASTM D 3532	3	15
Resin Flow	ASTM D 3531	3	15
Fiber Areal Wt.	†	3	15
Moisture Content	†	3	15
Tack	†	3	15
HPLC (High Performance Liquid Chromatography)	†	3	15
IR (Infrared Spectroscopy)	†	3	15
DMA (Dynamic Mechanical Analysis, neat resin only)	†	3	15
DSC (Differential Scanning Calorimetry)	†	3	15
RDS (Rheological Dynamic Spectroscopy)	†	3	15

[1] Test procedures should be coordinated and agreed to prior to manufacture of prepreg material.
[2] Tests should be performed on each of the five batches of prepreg material.
†Test procedures to be described at a later date.

2.3.2.3 Lamina test matrices

Recommended physical and mechanical property test matrices for statistical evaluation of lamina-level materials are shown in Tables 2.3.2.3(a) and 2.3.2.3(b).

The mechanical test matrix shown in Table 2.3.2.3(b) is based on a minimum of thirty tests per condition per property (at least six replicates for each of at least five batches) to provide for parametric/nonparametric analysis when determining B-basis properties. Fewer replicates or batches may be acceptable if agreed to between the contractor and the procuring or certifying agency.

TABLE 2.3.2.3(a) *Cured lamina physical property tests.*

Physical Property	Suggested Test Procedure	Number of Tests Per Prepreg Batch[1]	Total Number of Tests
Fiber Volume	ASTM D 3171	3	15
Resin Volume	ASTM D 3171	3	15
Density	ASTM D 792	3	15
Cured ply thickness	-	10	50
Glass Transition Temperature (dry)[2]	-	3	15
Glass Transition Temperature (wet)[2]	-	3	15

1. Tests should be performed on each of the five batches.
2. Dry specimens are "as fabricated" specimens which have been maintained at ambient conditions in an environmentally-controlled test laboratory. Wet specimens are environmentally conditioned by exposing them in an elevated temperature humidity chamber until they attain an equilibrium moisture content agreed to by the contractor and customer, then packaged in a heat-sealed aluminized polyethylene bag until required for test. Tests should be performed in a manner which maintains the moisture content in specimens at the levels agreed to by the contractor and certifying agency.

TABLE 2.3.2.3(b) *Cured lamina mechanical property tests.*

Mechanical Property	Test Methods[1]	Test Condition[2] and Number of Tests Per Batch[3]			Number of Tests
	See Handbook Section	Min. Temp Dry	RT Dry	Max. Temp Wet	
0° Tension (warp)	6.7.4.4[4]	6	6	6	90
90° Tension (fill)	6.7.4.4[4]	6	6	6	90
0° Compression (warp)	6.7.5.4	6	6	6	90
90° Compression (fill)	6.7.5.4	6	6	6	90
In-plane Shear	6.7.6.4	6	6	6	90
0° Short Beam Shear	6.7.6.4	-	6	-	30
					480

1. MIL-HDBK-17 is not currently in a position to make exclusive test method recommendations, but the referenced Handbook sections identify methods that are currently deemed acceptable for data submittals to MIL-HDBK-17.
2. Minimum and maximum temperature tests should be performed within ±5°F (±2.8°C) of the nominal test temperature. Nominal test temperatures will be as agreed to by contractor and certifying agency. Dry specimens are "as-fabricated" specimens which have been maintained at ambient conditions in an environmentally-controlled test laboratory. Wet specimens are environmentally-conditioned by exposing them in a humidity chamber until they attain an equilibrium moisture content agreed to by the contractor and certifying agency, and then packaging them in a heat-sealed aluminized polyethylene bag until required for test. Tests should be performed in a manner which maintains the moisture content in specimens at the levels agreed to by the contractor and certifying agency.
3. Tests should be performed on each of the five batches.
4. For 0° and 90° tension, ASTM D 3039 and SACMA Recommended Method (SRM) 4-88 are acceptable test methods for MIL-HDBK-17 data submittals.
5. Short Beam Shear is for screening and quality control purposes only.

2.3.2.4 Filament-wound materials test matrix

The test matrix shown in Table 2.3.2.4 contains the suggested mechanical property tests for filament wound structures.

TABLE 2.3.2.4 *Filament-wound materials property tests.*

Mechanical Property	Suggested Test Procedure[1]	Test Condition and Number of Tests Per Batch[2]			Number of Tests
Condition[3]		Min. Temp Dry	RT Dry	Max. Temp Wet	
0° Tension	ASTM D 3039	6	6	6	90
90° Tension	ASTM D 5450	6	6	6	90
0° Compression	ASTM D 3410 (Method B)	6	6	6	90
90° Compression	ASTM D 5449	6	6	6	90
In-plane Shear	ASTM D 5448	6	6	6	90
Interlaminar Shear	ASTM D 5379	6	6	6	90540

1 Reader is referred to Section 6.7 Mechanical Property Tests for more information on these ASTM test methods

2 Tests should be performed on each of the five batches.

3 Minimum and maximum temperature tests should be performed within ±5°F (±2.8°C) of the nominal test temperature. Nominal test temperatures will be as agreed to by contractor and certifying agency. Dry specimens are "as-fabricated" specimens which have been maintained at ambient conditions in an environmentally-controlled test laboratory. Wet specimens are environmentally-conditioned by exposing them in a humidity chamber until they attain an equilibrium moisture content agreed to by the contractor and certifying agency, and then packaging them in a heat-sealed aluminized polyethylene bag until required for test. Tests should be performed in a manner which maintains the moisture content in specimens at the levels agreed to by the contractor and certifying agency.

The JANNAF Composite Motorcase Subcommittee has recommended to filament wind the flat laminates used for the test articles for ASTM D 3039, ASTM D 3410 and ASTM D 5379 for the uniaxial material properties used in the design and analysis of filament wound structures. However, there are no universal standards describing the process. Consequently there are numerous methods used by industry and Government to manufacture the flat laminates, (References 2.3.2.4(a) and (b)). At these two meetings filament winders, both industry and government, presented their techniques to prepare flat laminates for the purpose of testing for uniaxial mechanical material properties.

One main issue is whether to use a cylindrical or a rectangular winding mandrel. If a cylindrical mandrel is used, the diameter of the mandrel is a factor. The larger the diameter, the less the effects of shear when the laminates are removed from the winding mandrel and flattened for curing. If the mandrel is rectangular, the main concern is how tension of the fiber is maintained during winding.

The following issues have been identified as concerns for filament wound laminates:

- autoclave vs. non-autoclave cure
- cutting of fibers before or after cure
- whether to cure on the winding mandrel or to remove and cure on a separate fixture
- whether to use a caul plate
- whether to wind single layers, cut and stack versus winding entire thickness before cutting

Currently the winders appear to be using the technique that produces a panel that most closely simulates the process used on their finished part. The ASTM Task Group D30.04.05 is discussing and pursuing these same issues and developing a standard method to prepare filament wound laminates.

2.3.3 Material acceptance test matrices

This section is reserved for future use.

2.3.4 Alternate material equivalence test matrices

2.3.4.1 Qualification of alternate source composite materials

2.3.4.1.1 Introduction

These guidelines apply to the situation where one composite material system from a single supplier has been qualified and it is necessary or desirable to qualify an alternate system and/or supplier. The approach assumes the existence of a body of data and experience developed with the original material (none exists for the alternate system) from which the mechanical property basis values have been developed. It also assumes higher level tests have been performed to qualify a product and verify its performance.

A drastic change, such as switching fiber from E-glass to aramid, is not covered by this guideline. The focus is on materials that will meet the original material specification. A fiber class change, or comparable substitution, is considered a major revision or redesign. Processing and tooling changes are also considered beyond the scope of this section.

2.3.4.1.2 Goal and approach

The ultimate goal in qualifying an alternate material is to be able to exchange this material with the original system without compromising manufacturing or structural performance. To accomplish this goal it is necessary to define the key material parameters that govern performance during specific phases, such as processing, manufacturing, and service. The ideal is to perform this evaluation at the material constituent or composite lamina levels by measurement and comparison of parameters like chemical composition, fiber strength, matrix strength, and composite strength. This may be possible in the future, but is not adequate with current technology.

Successful qualification of an alternate material will not, in itself, be sufficient to permit mixing of this system with the original material within a given part. Intermixing of two different material systems within the same part is not recommended unless appropriate evaluations are carried out to demonstrate compatibility.

The focus of MIL-HDBK-17 is B-basis lamina properties. Adequate alternate material qualification may require going beyond this level of evaluation into more complex demonstrations involving analysis and tests. These may include laminate, specimen, element, and subcomponent tests such as open hole, filled hole, bolt bearing, low velocity impact, fatigue, and panel buckling. The general approach to be followed for qualification of an alternate material is as follows:

1. Identify the key material performance parameters and why they are crucial.

2. Define appropriate tests, measurements, or evaluations for each of the parameters. These must correspond exactly to the tests, measurements, or evaluations performed on the original material (for example: same specimen type and same conditions).
3. Define pass/fail (success) criteria for the tests, measurements, and evaluations.
4. Prepare a test plan and obtain necessary approvals.
5. Perform tests and document results.
6. Accept or reject.

2.3.4.1.3 Material compatibility

The extent to which an alternate material used in hardware applications must be evaluated to demonstrate equivalence, or superiority, with the original system is first, a function of its material compatibility and second, a function of hardware structural complexity and loading. Material compatibility is defined by the criteria shown in Table 2.3.4.1.3. The baseline system is a material from a single prepregger using a specific prepreg production line. For example, AS4/3501-6 produced from line 3 at Hercules, Inc. The most compatible alternate material, and the one requiring the minimum to demonstrate equivalence would be AS4/3501-6 produced from line 4 at Hercules, Inc. The least compatible material system would be one from a different prepregger with a different matrix and fiber. Thus, Fiberite C12K/934 is a less compatible system and requires more effort to demonstrate equivalence. Situations not included in Table 2.3.4.1.3 must be evaluated with respect to their appropriate compatibility scale.

2.3.4.1.4 Key material or structural performance parameters

Key material or structural performance parameters are those measurable quantities which, if compared to the original values, can be used to quantify any difference in manufacturing or structural performance parameters, are material and hardware dependent, and may change with design, tooling, manufacturing, and usage factors. However, five categories of parameters have been defined in Table 2.3.4.1.4. This table lists examples of typical performance parameters appropriate for each category.

2.3.4.1.5 Success criteria

The relative importance and completeness of performance parameters varies with the part design, loading, and application. In some cases it is sufficient merely to report a measured value. In other cases the value must meet or exceed the original measurement. And in some cases the value must not vary significantly either higher or lower than the original value. As an example, this is generally true for modulus, fiber areal weight, matrix content, and cured ply thickness.

Success criteria for each parameter must be defined at the beginning of the qualification program. Justification for each success criteria imposed must be provided. A tolerance on a given measurement should be part of the success criteria.

TABLE 2.3.4.1.3 *Material compatibility criteria.*

MOST COMPATIBLE						LEAST COMPATIBLE
MATERIAL FACTOR	1	2	3	4	5	6
Fiber Type	Y	N	Y	N	Y	N
Fiber Tow Size	Y	Y/N	Y	Y/N	Y	N
Resin	Y	Y	N	Y	N	N
Prepregger	N	Y	Y	N	N	N
Production Line	N	Y	Y	N	N	N

Y - remains the same in alternate material
N - changes in alternate material

a) Column 1 is a change in prepreg supplier and production line. This situation is becoming more common today as resin systems are licensed between prepreg manufacturers; for example, the Navy's A-6 re-wing and V-22 Osprey programs where Hercules 3501-6 is licensed to ICI Fiberite. This cooperative licensing allows competitive bidding for prepreg supplies and provides the customer with nearly identical prepreg for production usage.

b) Column 2 represents a change in fiber type based on a new fiber with properties similar to the originally qualified fiber. This situation may occur for economic reasons or in the event of discontinued fiber supply.

c) Column 3 is a change in resin. This would be justified by development of new resin systems by the prepregger that would offer improved pricing and/or properties, such as damage tolerance, for the customer's program.

d) Columns 4 and 5 represents a change in prepreg supplier, production line, and fiber or resin. This situation would occur when a customer needs and additional supplier but wishes to use the same fiber or resin due to second-source qualification budget constraints (assumes existing data base on the resin and/or fiber). Again, economic reasons justify this situation.

e) Column 6 involves qualifying a new source prepregger using a different fiber and resin system. An example of this situation is qualifying Fiberite C12K/934 to replace Hercules AS4/3501-6. This is the least compatible situation and would require the greatest effort to demonstrate acceptability.

TABLE 2.3.4.1.4 *Examples of key material or structural performance parameters.*

PHYSICAL	PROCESSING	MECHANICAL	MANUFACTURING	HARDWARE SCALE-UP
TACK	CURED PLY THICKNESS	LAMINA PROPERTIES	DRILLING	STATIC STRENGTH
RESIN CONTENT	CURE CYCLE	ENVIRONMENTAL EFFECTS	TOOLING	FATIGUE STRENGTH
AREAL WEIGHT	SENSITIVITY	DAMAGE TOLERANCE	NONDESTRUCTIVE INSPECTION	STIFFNESS
FLOW	FIBER VOLUME	INTERLAMINAR SHEAR	COST	FAILURE MODES
GLASS TRANSITION TEMPERATURE	THERMAL CYCLING	FLATWISE TENSION	LEAD TIME	QUALITY
FORM	DENSITY	FLAW GROWTH	AVAILABILITY	BEARING
OUT TIME	EXOTHERM	EFFECT OF DEFECTS	REPEATABILITY	CRIPPLING
SHELF LIFE	TOXICITY	PRESSURE BOTTLE TESTS	MACHINABILITY	OPEN HOLE TENSION
STORAGE REQUIREMENTS			UNIFORMITY	OPEN HOLE COMPRESSION
MOISTURE ABSORPTION				PANEL TESTS
SOLVENT RESISTANCE				FATIGUE TESTS

2.3.4.1.6 Lamina-level test matrices for alternate material assessment

Section 2.5 defines minimum requirements for B-basis lamina property values for MIL-HDBK-17 data, which can be quickly summarized as thirty specimens from at least five batches of material for each environment and property of interest. Since an alternate material qualification program is not intended to establish basis values, but rather to show compliance with them, a reduced number of lamina tests can be allowed for a second population of data to be compared to the original data. The actual number of equivalency tests needed depends on the degree of compatibility between the two material systems. Recommendations for test quantities and properties for tape and fabric material forms are shown in Tables 2.3.4.1.6(a) and (b). The equivalency check tests must be performed in the same way, and using the same test methods, as the tests used to determine the basis values. Following testing, (see Section 8.4.3) appropriate statistical analysis must be performed to evaluate the test results and assess equivalency.

TABLE 2.3.4.1.6(a) *Alternate material lamina test requirements - tape.*

	No. of Batches						Replicates						Environments[2]						Total					
Lamina Property	Compatibility[1]						Compatibility[1]						Compatibility[1]						Compatibility[1]					
	1	2	3	4	5	6	1	2	3	4	5	6	1	2	3	4	5	6	1	2	3	4	5	6
0° Tension	2	3	3	3	3	3	4	4	4	5	5	6	2	2	2	2	2	2	16	24	24	30	30	36
90° Tension	2	3	3	3	3	3	4	4	4	5	5	6	2	2	2	2	2	2	16	24	24	30	30	36
0° Compression	2	3	3	3	3	3	4	4	4	5	5	6	2	2	2	2	2	2	16	24	24	30	30	36
90° Compression	2	3	3	3	3	3	4	4	4	5	5	6	2	2	2	2	2	2	16	24	24	30	30	36
In-Plane Shear	2	3	3	3	3	3	4	4	4	5	5	6	2	2	2	2	2	2	16	24	24	30	30	36
																			80	120	120	150	150	180

[1] Compatibility is defined in Table 2.3.4.1.3.

[2] The environments should be RTD and the worst case.

Quality assurance tests must be performed per individual specification.

TABLE 2.3.4.1.6(b) *Alternate material lamina test requirements - fabric.*

	No. of Batches						Replicates						Environments[2]						Total					
Lamina Property	Compatibility[1]						Compatibility[1]						Compatibility[1]						Compatibility[1]					
	1	2	3	4	5	6	1	2	3	4	5	6	1	2	3	4	5	6	1	2	3	4	5	6
Warp Tension	2	3	3	3	3	3	4	4	4	5	5	6	2	2	2	2	2	2	16	24	24	30	30	36
Fill Tension	-	3	3	3	3	3	-	4	4	5	5	6	-	2	2	2	2	2	-	24	24	30	30	36
Warp Compression	2	3	3	3	3	3	4	4	4	5	5	6	2	2	2	2	2	2	16	24	24	30	30	36
Fill Compression	-	3	3	3	3	3	-	4	4	5	5	6	-	2	2	2	2	2	-	24	24	30	30	36
In-Plane Shear	2	3	3	3	3	3	4	4	4	5	5	6	2	2	2	2	2	2	16	24	24	30	30	36
																			48	120	120	150	150	180

[1] Compatibility is defined in Table 2.3.4.1.3.

[2] The environments should be RTD and the worst case.

Quality assurance tests must be performed per individual specification.

2.3.4.1.7 Laminate-level test matrices for alternate material assessment

The next higher level of testing that should be considered for qualification of an alternate material system is laminate mechanical properties. This level of testing confirms strength (strain) basis values for strategic design parameters and should be performed using the same laminate tested for the original material. The recommended tests are shown in Table 2.3.4.1.7(a). The extent to which the Table 2.3.4.1.7(a) tests are performed is governed by the material compatibility factor. The recommended number of tests is given in Table 2.3.4.1.7(b).

TABLE 2.3.4.1.7(a) *Extent of laminate testing.*

Material Compatibility Factor	Laminate Tests	Total	
		Tape	Fabric
1	Unnotched Laminates	12	12
2, 3	All Static Test, Two Environments	36	36
4, 5	All Static Test, Two Environments	36	36
6	All Required	42	42

2.3.4.1.8 Alternate material evaluation summary

Many of the handbook recommendations on key material performance parameters, such as physical and processing characteristics, are commonly included in material and process specifications. Other parameters are more application related and may be difficult to demonstrate at the material level. The reader should not infer from lack of discussion that a particular topic is unimportant; all key performance parameters for a specific project or product must be considered.

Guidelines for substantiating lamina and laminate material property requirements, given some change in the material system or process, were provided. Higher level mechanical element/subcomponent substantiation tests may also be required, depending on the degree of change in the key material or structural parameter, and on the application.

Statistical methods for comparing batches are discussed in Section 8.4.3.

2.3.4.2 Evaluation of changes made to previously qualified materials

This section defines guidelines for evaluating changes made by a material supplier to a material system provided as a qualified source. A drastic change is not covered herein. The focus is to meet original (existing) material specification requirements. Potential changes at all levels should be considered.

The goal of the recommended evaluations is to verify that intended changes do not compromise physical, structural, or manufacturing requirements. This guideline provides a list of potential changes and appropriate experiments/tests to evaluate the effects of a particular change. Specific evaluations are tailored to the nature and severity of proposed changes.

TABLE 2.3.4.1.7(b) *Number of suggested laminate tests.*

Design Property	Loading		No. of Laminate Types		No. of Environments[1]	Replicates[2]	Total Number of Specimens	
	Tension	Compression	Tape	Fabric			Tape	Fabric
Static								
Unnotched laminate, strength and stiffness	X	X	1	1	2	3	12	12
Open Hole		X	1	1	2	3	6	6
Filled Hole	X		1	1	2	3	6	6
Impact Damage	X	X	1	1	1	3	6	6
Double Shear Bearing	X		1	1	1	3	3	3
Single Shear Bearing	X		1	1	1	3	3	3
							36	36
Fatigue[3]								
Open Hole	-	-	1	1	1	3	3	3
Impact Damage	-	-	1	1	1	3	3	3
							6	6
							42	42

[1] Where two environments are required, they should be RTD and worst case. Where one is required, it should be RTD.
[2] One batch of material is sufficient.
[3] Repeated load and residual strength: constant amplitude, $R = -1$, $n = 1 \times 10^6$ cycles.

A documented quality plan is an assumed prerequisite for this procedure. It should describe the manufacturing process from raw materials receiving to final product shipment. This document should be kept current. It should be in accordance with ISO 9002 or Mil-Q-9858A. The quality plan should reference raw materials used, show key manufacturing steps in proper sequence, and list critical process control documentation as well as quality inspection or testing.

At the time of a proposed modification, a process analysis should be done to determine if the proposed change warrants further consideration. This can be done by an appropriate technical specialist. Guidelines for screening possible modifications should be established prior to embarking on an evaluation program. For example, routine or ongoing maintenance of equipment, changes in personnel, or upgrading control instrumentation would not normally require formal evaluation. Proposed changes in product formulation, elimination of process steps, changes in manufacturing equipment, or changes in sequence of operations are the types of significant modifications that would require formal evaluation.

The relative importance or category of a proposed process modification is determined by a logical system of in-depth process and product impact analysis. It is recommended that a process review team (PRT) be established to perform the process analysis. The process analysis must identify:

- Key process steps (including sequence)
- Key equipment used at each process step.
- Quality-critical processing parameters for each piece of equipment (time, temperature, rate, pressure).
- Quality-critical operating ranges for each critical process parameter.
- Quality-critical instrumentation used for monitoring and/or controlling each critical process parameter.

2.3.4.2.1 Modification categories

When a proposed process modification is identified for consideration, a comprehensive review of all related information should be conducted. This includes the rationale for making the process modification. An appraisal should be made as to the impact that the change could have on the next product user as well as upon the product's performance in the final application.

The foundation of the review is derived from the knowledge obtained from the product/process analysis described previously. Based upon this product impact review, the process modification will be placed into one of the following three categories:

Category 1: "No Impact"

The modification is minor in nature. It is known not to impact the product's quality, physical or chemical properties or performance. Additionally, the modification is not likely to cause operational or product performance deficiencies for subsequent customers. This type of process modification is therefore classified as "No Impact".

Category 2: "Unknown"

If upon review of available information there is not enough known about the proposed change, then the modification must be classified as "Unknown".

Category 2 is a temporary classification which is held until additional information is made available. No modification classified as Category 2 should be implemented. All Category 2 classifications must eventually become Category 1 or 3 before the modification is implemented.

Category 3: "Change"

If upon review of available information it is decided that the proposed modification may result in a significant change to the product's properties, quality, performance, or may have an impact on subsequent customers, then the modification must be classified as a "change".

2.3.4.2.2 Actions required for each modification category

Category 1 modifications should be formally approved. The change should be documented and an appropriate process change follow-up or monitoring file initiated. This releases manufacturing to implement the modification at an agreed upon schedule with appropriate monitoring for a specified time.

If the change is to a raw material ingredient, the "No Impact" classification can be applied if it is demonstrated as equivalent using a minimum of three lots of the ingredient both before and after the modification to that ingredient. The testing matrix used to demonstrate this should be agreed upon by the raw material manufacturer and the composite manufacturer as representing all significant characteristics of that material.

If the proposed modification is classified as "Unknown", additional information or testing should be identified for further review and action.

Manufacturing should not implement the proposed modification until the additional information or testing has been reviewed and status updated to either Category 1 or 3.

If the proposed modification is classified as a Category 3 "Change" then:

(a) The process modification is not implemented or
(b) An equivalency test plan is defined according to Tables 2.3.4.2.2(a) through (h).

When equivalency testing is performed, the data should be compared to the existing product data per statistical procedures given in Section 8.4.1. If the data analysis shows equivalency, the resulting data report should be submitted to the customer(s) for concurrence. If the data analysis shows that the modification resulted in non-equivalent products, the manufacturer will either:

(a) Not implement the change or
(b) Review the data documentation report with the customer to determine actions required for implementation.

2.3.4.2.3 Implementation

Category 1, "No Impact", process modifications can be implemented immediately based on the review approval. Normal acceptance testing should continue to be monitored to confirm that there has been no product impact.

Category 2, "Unknown", process modifications can not be implemented until additional information is available. Category 2 process modifications may only be implemented after conversion to and approval of either a Category 1 or 3 classification.

Category 3, "Change", process modifications require appropriate validation testing and written customer notification and concurrence prior to implementation or product shipment.

TABLE 2.3.4.2.2(a) *Validation requirements versus changes fiber.*

Change Description	Testing Requirements - Number of lots to be tested (A) (B)										
	Component Property		Prepreg Properties			Laminate Mechanical Properties					
	Level 1	Level 2	Physical	Process	Mechanical Accept	Comp ETW	±45 ETW	OHC ETW	OHT	CAI	(C)
	Table 2.3.4.2.2(d)		Table 2.3.4.2.2(f)	Table 2.3.4.2.2(g)	Table 2.3.4.2.2(g)						
New line	3	3	2	-	1	1	1	-	-	-	-
Precursor relocation	3	3	3	-	3	3	3	2	2	-	2
Sizing	3	3	3	1	3	3	3	2	2	-	2
Weaver	2	-	-	-	1	-	-	-	-	-	-
Relocation	2	-	-	-	1	-	-	-	-	-	-
Major on-line equipment	2	(D)	-	-	1	1	1	-	-	-	-
Process	2	(D)	-	-	1	1	1	-	-	-	-
Raw material	2	(D)	-	-	1	1	1	-	-	-	-

NOTES: (A) Prepreg tests made using most representative resin system.
(B) Chemical and physical tests use 3 specimens per sample. Mechanical tests use 5 specimens per sample.
(C) Fracture toughness or interfacial bonding test.
(D) Decision based on degree of change.

TABLE 2.3.4.2.2(b) *Validation requirements versus changes formulated resin.*

Change Description	Testing Requirements - Number of lots to be tested (A) (B)										
	Component Property		Prepreg Properties			Laminate Mechanical Properties					
	Level 1	Level 2	Physical	Process	Mechanical Accept	Comp ETW	±45 ETW	OHC ETW	OHT	CAI	(C)
	Table 2.3.4.2.2(e)		Table 2.3.4.2.2(f)	Table 2.3.4.2.2(g)	Table 2.3.4.2.2(g)						
Ingredient	3	3	2	1	2	2	2	2	-	1	1
Source for ingredient	3	3	1	1	1	1	1	-	-	-	-
Process	3	3	2	1	2	2	2	-	-	-	-
Equipment	3	3	2	1	2	2	2	-	-	-	-
Relocation	2	-	1	-	1	1	1	-	-	-	-

NOTES: (A) Prepreg tests made using most representative resin system.
(B) Chemical and physical tests use 3 specimens per sample. Mechanical tests use 5 specimens per sample.
(C) Fracture toughness or interfacial bonding test.

TABLE 2.3.4.2.2(c) *Validation requirements versus changes prepreg.*

Change Description	Testing requirements - number of lots to be tested (A) (B)								
	Prepreg Properties			Laminate Mechanical Properties					
	Physical	Process	Mechanical Accept	Comp ETW	±45 ETW	OHC ETW	OHT	CAI	(C)
	Table 2.3.4.2.2(f)	Table 2.3.4.2.2(g)	Table 2.3.4.2.2(g)						
Process/equipment	3	1	2	2	2	2	2	-	2
New line	3	1	2	2	2	2	2	-	2
Relocation	2	1	1	1	1	-	-	-	-
New fiber and/or resin	3	2	3	3	3	3	3	3	3

NOTES: (A) Prepreg tests made using most representative resin system.
(B) Chemical and physical tests use 3 specimens per sample. Mechanical tests use 5 specimens per sample.
(C) Fracture toughness or interfacial bonding test.

TABLE 2.3.4.2.2(d) *Fiber testing matrix*.

TEST	LEVEL 1	LEVEL 2
Tow Tensile	X	
Tow Modulus	X	
Density	X	
Mass per Unit Length	X	
Surface Characterization Such as ESCA/Interfacial Energy/Microscopic Evaluation		X

TABLE 2.3.4.2.2(e) *Neat resin testing matrix*

PROPERTY	LEVEL 1	LEVEL 2
HPLC	X	
Infrared		X
DSC		X
Gel Time	X	
Flexural Modulus		X
Glass Temperature, Dry and Wet		X
Viscosity		X
Moisture Absorption		X

TABLE 2.3.4.2.2(f) *Prepreg physical testing.*

PROPERTY	
Resin Content/Areal Weight Variability	X
Flow	X
Glass Transition Temperature, Dry and Wet	X
Moisture Absorption	X

TABLE 2.3.4.2.2(g) *Prepreg Processibility testing.*

Microcracking/Thermal Cycling of Cured Laminate	X
Morphology/Microstructure of Cured Laminate	X

TABLE 2.3.4.2.2(h) *Mechanical acceptance testing*.

PROPERTY	ROOM TEMPERATURE	ELEVATED TEMPERATURE DRY
Tensile Strength and Modulus	X	
Compression Strength	X	X
Shear, either SBS or ±45	X	X

2.3.4.2.4 Validation test matrices

Tables 2.3.4.2.2(a) through (h) define the validation testing recommended as a function of the type of change proposed. Table 2.3.4.2.2(a) provides the guidance for fiber changes. Table 2.3.4.2.2(b) is the overview and guidance for resin changes. Table 2.3.4.2.2(c) describes the recommendations for prepreg changes. Refer to the left hand column in each table for the change description which best represents the modification being proposed.

After the appropriate change description has been identified, the recommended testing is shown in the horizontal row to the right. The number of separate batches of material recommended for validation at each level are shown in Tables 2.3.4.2.2(a) through (c).

The test types are shown in Table 2.3.4.2.2(a) through (c) and are further delineated in the subsequent tables (Tables 2.3.4.2.2(d) through (h)).

All chemical and physical tests should use three specimens per sample. Five specimens per sample are recommended for all mechanical tests.

Prepreg testing can be performed with the most representative resin or fiber (whichever is independent of the change). That choice should be based on that material having the most credible data base. For example, if a change is being made to AS4 fiber, the validation could be performed by testing the fiber with 3501-6 resin, since that fiber/resin combination has the most complete data base.

2.3.5 Generic laminate/structural element test matrices

2.3.5.1 Introduction

A simplified flow chart, Figure 2.3.5.1, overviews the building-block flow of a typical material/structural qualification process. A series of evaluations is required to assess the adequacy of a material system for

production usage. These multi-purpose assessments, often performed in parallel, range from material performance to producibility and cost.

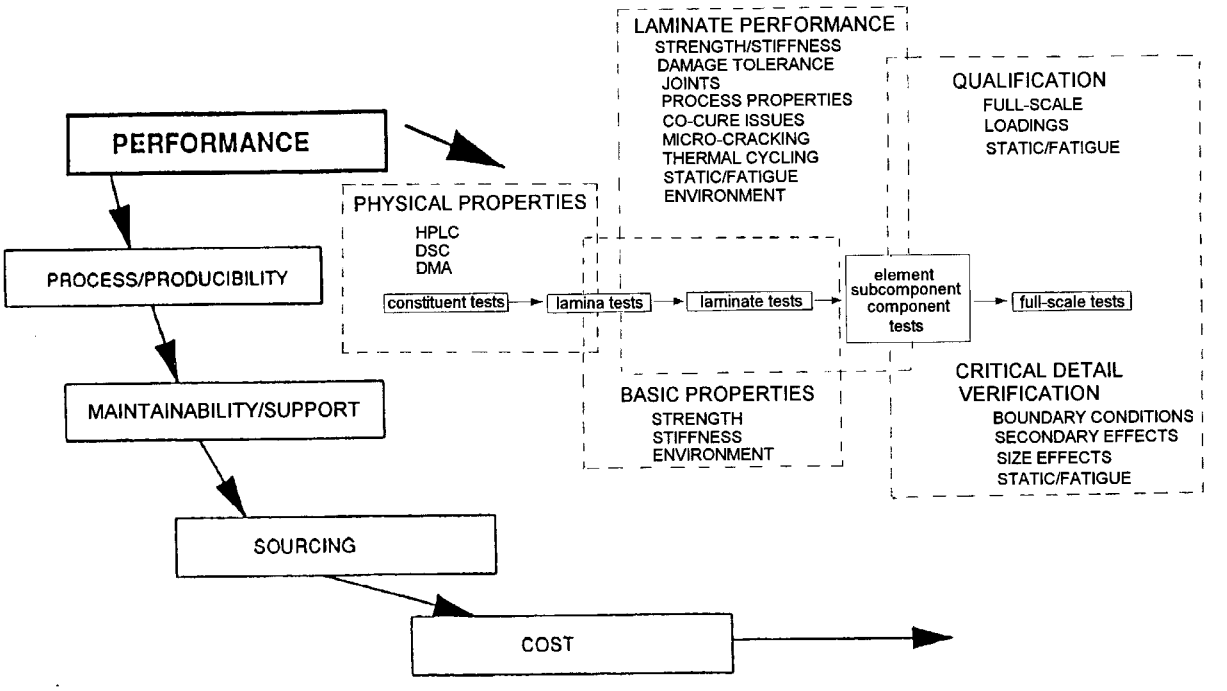


FIGURE 2.3.5.1 *Material/structural qualification.*

Depending on the sophistication of the application design concept (e.g., a flat monolithic panel versus an integrally cocured semi-monocoque fuselage section), an extensive, progressively more complex, building-block approach to certification testing may be required to evaluate and reduce material and structural design risks. It is recognized that the complete structural qualification of composite material systems for design allowables is often highly dependent on the application for which the material will be used. The historical lessons-learned dictate that composite production hardware design programs must evaluate and discover material, structural, and producibility design deficiencies early in the design development program to meet cost, performance, and schedule goals. Toward this end, it is extremely desirable on any program to establish early, with high confidence, the material design allowables. If this is successfully accomplished, the design development program can then focus on detail design, higher level design development tests, and producibility issues. The most *adverse* situation for any program to experience is a material development or reselection effort *in parallel* with detail design development.

This section addresses that part of the Figure 2.3.5.1 process which assesses the mechanical property characterization at the laminate level. The intent is to define a series of laminate level test matrices that complements both the ply level mechanical property characterization test matrix and the lamina/laminate screening test matrix, previously defined in Sections 2.3.2.3 and 2.3.1.1.

The basis for the test matrices of this section is that a significant number of similar, laminate level, specimen tests are performed in almost all hardware design development programs prior to extended production. The additional laminate level test data are necessary for theory/test correlations to substantiate mathematical models used to predict design allowables. Often these models employ lamina (ply) stiffness and strength input data (Section 2.3.2.3). Alternatively, laminate test data are needed to establish empirical trending where mathematical models do not exist or are deemed deficient. In either case,

some specimen laminate-level data have been historically required to substantiate or establish the design allowables essential to structural qualification. These costly and time-consuming tests are often repeated in each new application program. Because a significant number of these tests are performed at the specimen level, *the test data generated should, once generated, apply to a wide range of applications, and be acceptable to certifying agencies in other application programs*.

These generic characterization tests, once performed (with test matrices in Sections 2.3.2.3 and 2.3.1.1), are intended to further reduce the cost and time of new material characterization efforts, and *establish a generic database for the tested material* system *applicable to other proposed applications*.

2.3.5.2 Overview

Two laminate level test matrices are defined: (1) laminate strength, and (2) bolt bearing and bearing/bypass strength. Together, these test matrices should provide a statistically significant laminate-level database. The test matrices are defined for selective 3-batch assessments of either tape or fabric prepreg materials. Dependent upon the availability of *validated* analytic models for strength prediction, and the degree to which they use only ply-level strength and stiffness input data, batch effects may be accounted for at the lamina level and not require multiple batch testing in the following test matrices. Thus, with certification agency approval, a single-batch test plan variation may be proposed with replicates of 5 specimens per test condition as implied in Chapter 7. Additionally, it is noted that other load conditions, such as in-plane shear, may require additional testing at higher levels in the building-block assessment and are not covered by these test matrices.

2.3.5.2.1 Laminate strength test matrix

As detailed in Table 2.3.5.2.1, a series of selected orientation laminate unnotched strength tests are recommended for both tensile and compressive loadings at selective cold temperature dry (CTD), room temperature dry (RTD), and elevated temperature wet (ETW) test conditions. For two laminate configurations, three replicate tests are repeated for each of three batches of the material system. Two additional laminates are selectively tested with 5 specimens per test condition using one batch of material. The matrix emphasizes fiber dominant laminate evaluations at the extremes of material environmental capabilities (CTD and ETW) and provides baseline data at room temperature dry (RTD) test conditions. The intent is to provide data to permit a selective validation of stiffness and strength analytic models over a representative range of application relevant laminates and test conditions.

The limited number of tests implies that data pooling using regression analysis across test conditions will be employed (Section 8.3.5.3). The exact specification of critical temperatures and moisture conditioning is determined either by the minimum/maximum material operational capabilities (MOL) established for lamina level tests (Sections 2.3.1.1 and 2.3.2.3) or application considerations jointly established by the manufacturer and the procuring agency.

Four general laminate configurations are specified for tape characterization testing; three laminates for fabric material forms. As illustrated in the carpet plot of Table 2.3.5.2.1, the selection of the four laminates is intended to span the usual application range of structural laminates with emphasis on the fiber dominant orthotropic and quasi-isotropic laminate constructions. Additionally, the solid circles indicate the solely 0-degree, 90-degree, or ±45-degree (lamina level) evaluations specified in Section 2.3.2.3. For fabric characterization testing, the bold line in the carpet plot of Table 2.3.5.2.1 represents the reduced range of possible laminate constructions and the 50/40/10 and 40/40/20 tape laminates are replaced by a 40/20/40 fabric laminate construction in the test matrix of Table 2.3.5.2.1. Stacking sequences as specified in Section 7.2 on mechanically fastened joints are also recommended for laminates in this section.

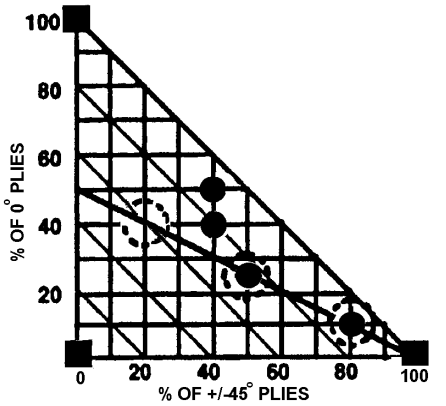
TABLE 2.3.5.2.1 *Laminate strength test matrix.*

OBJECTIVE:

Create a generic, laminate-level, database to assess an application range of unnotched laminates for correlation of laminate stiffness and strength analytic models and establishment of a selective, but statistically significant, database for empirical approaches.

Φ ORIENTATION OF 0 DEGREE PLIES TO LOAD DIRECTION

LAY-UP (%0,%±45,%90 degree plies)	THICK-NESS	LOAD ANGLE Φ	COMPR RTD	COMPR ETW	TENS CTD	TENS RTD
25/50/25	T1	0	9	9	9	9
	T2	0	9	9	9	9
	T1	22.5	-	5	5	-
50/40/10	T1	0	9	9	9	9
	T2	0	9	9	9	9
	T1	22.5	-	5	5	-
40/40/20	T1	0	-	5	5	-
10/80/10	T1	0	-	5	5	-
SUBTOTALS =			36	56	56	36
TOTALS =						184



NOTES:

Assumes 0, +45, -45, & 90 degree family of ply orientations; balanced & symmetric
9 implies 3 specimens/batch; 3 batches
5 implies 5 specimens/batch; 1 batch
T1 represents laminate thickness of approximately 2.0-6.0 mm (.08-.25 inch)
T2 represents second laminate thickness; *may be optional* depending on upper bound of application laminates
For fabric — 40/40/20 replaces 50/40/10 **laminate and 40/40/20 is deleted**

■ PLY (LAMINA) LEVEL TEST POINTS (VOL. I, SECT. 2.4)

● LAMINATE LEVEL TEST POINTS (THIS TEST MATRIX)
RTD - room temp., dry test condition
CTD - cold temp., dry test condition
ETD - elevated temp., dry test condition

◌ FABRIC TEST POINTS

The test matrix also requires two of the laminates to be tested at 22.5 degrees off the principal material axis to assess off-axis material behavior at the critical environmental test conditions. Additionally, if the application range of thickness exceeds significantly (say by a factor greater than two) the basic "T1" thickness range of 0.08-0.24 inches (2-6 mm), a second three-batch series of "T2" laminate thickness tests is specified in Table 2.3.5.2.1 for all test conditions. However, if the application range of laminate thickness are contained within a 0.16 inch (4 mm) variation, it is believed only one test matrix thickness is required (perhaps different from the "T1" range suggested in Table 2.3.5.2.1). This would reduce the matrix to a total of 114 tests for tape laminates (104 for fabric). If the range of application thickness is significantly broader, the second series (T2) of laminate thickness tests should also be performed. This would result in a total of 184 tests for tape laminates (174 for fabric.

2.3.5.2.2 Bolt bearing and bearing/bypass strength test matrix

The test matrix, detailed in Table 2.3.5.2.2, is intended to provide strength data to assess the effects of under bearing and bearing/bypass strain concentrations on laminate strength. In addition to generating design data to establish allowables for composite bolted joint analysis, the filled-hole strength can be used as a reference strength for the effect of manufacturing anomalies and impact damage on laminate strength. The test recommendations in this section include all tests found in Chapter 7, Tables 7.5.2.4(a), 7.4.4, and 7.5.5.3. However, there are additional tests required by Table 2.3.5.2.2 because of the desire to empirically obtain laminate design allowables directly from these data. This necessitates selective testing of three batches of material at the worst environments.

Three of the laminate constructions previously tested under the unnotched laminate strength matrix of Section 2.3.5.2.1 are also specified for testing tape and fabric materials under tensile and compressive bolt bearing and bearing/bypass loadings. Single fastener joint evaluations cover a range of fastener load transfer test conditions including filled hole (100% bypass), bearing/bypass interactions (75% and 50% bypass), and pure bearing (0% bypass). The test matrix is designed to emphasize critical material environmental conditions with most test data collected at either the hot-wet (ETW) condition for matrix-dominated failure modes or the cold-dry (CTD) condition for fiber-dominated failure modes. Sufficient data are also specified to establish a room temperature dry baseline condition. Tension and compression load conditions are specified. Pure bearing tests are only performed under tensile loadings.

The test philosophy is to first evaluate the effect of hole size on laminate strength. Three fastener diameters which span the range of application hardware are specified. Tensile and compressive strength data are gathered for filled hole tension/compression, bearing/bypass, and pure bearing test conditions. Specimens detailed in Figures 7.4.2, 7.5.3.2(d), and 7.5.3.2(a) of Section 7.5, respectively, should be employed. The fastener diameter which represents the majority of application fastener diameters should be selected as the baseline diameter D1 in Table 2.3.5.2.2. The remaining two fastener diameters (D2 and D3 of Table 2.3.5.2.2) should bound all other application fastener usage. Based on historical aerospace industry practice with carbon/epoxy, a baseline joint geometry of width/diameter (W/D) of 6 and edge-distance/diameter (e/D) of 3 is recommended for almost all test specimens (two additional W/D ratios tested for the 25/50/25 lay-up; these values may change as carbon/epoxy systems evolve or for other material systems). The fastener head style, protruding or countersink, that represents the majority of application usage should also be selected as the baseline (H1 in Table 2.3.5.2.2) for all specimen configurations. The "T1" laminate thickness, discussed in Section 2.3.5.2.1, is used as the baseline thickness for all specimens1. If necessary to cover application design variations, a second series of laminate specimens, of thickness T2 in Table 2.3.5.2.2, may be required. Stacking sequences as specified in Section 7.2 on mechanically fastened joints are also recommended for laminates in this section.

The initial testing should be performed on the isotropic (25/50/25) laminate specimens for the 100% bypass and pure bearing (0% bypass) load conditions at RTD environment. *This should permit initial correlation (or calibration) of analytic or empirical models used for strength prediction of laminates under tensile or compressive loadings*. It is anticipated that these calibrated strength prediction models will then be used to predict, prior to test, the results to be obtained from the remaining bearing/bypass tests of the iso-

[1]T1 is specified as 0.2 in. (5 mm) in Section 7.2.5.3

tropic laminate and the full range of tests on the remaining two orthotropic laminates (tape - 50/40/10 and 10/80/10, and fabric - 40/20/40 and 10/80/10). These initial evaluations would be followed by the full range of environmental (CTD or ETW) tests and provide the statistical validation or basis for establishing critical design condition allowables. These same tests, compared to RTD test results, would verify consistency and types of laminate failure modes. Finally, if required, the second series of laminate thicknesses (T2 and T3) and fastener head style (H2) tests would be performed at the critical environmental test conditions to establish additional calibration of analytic models or empirical "knockdown" factors for design allowables.

Based on typical design considerations, pure bearing tests under tensile loading conditions provide conservative strength values and similar failure modes as compared to pure bearing tests under compressive loading, as long as the specimen edge distance/diameter (e/D) ratio is specified as 3 or greater. Accordingly, this test matrix requires pure bearing tests (0% bypass) only under tensile loading conditions. Similarly, design values based on the intermediate 50% level of bearing/bypass load interactions under compressive loading are generally conservative compared to design values based on the 75% level or intermediate levels of tensile loading. Thus, in some cases, only tests of 50% bearing/bypass load interaction under compressive loading may be sufficient and the compressive tests at 75% levels and the tests of 50% bearing/bypass conditions under tensile loading may be eliminated. For current composite material systems, this is felt to be realistically conservative for both the acknowledged nonlinear behavior under compressive bearing/bypass load conditions and the relatively linear material behavior under tensile bearing/bypass load conditions. Should material behavior or design weight goals require a less conservative approach, more experimental evaluation would be necessary and other bearing/bypass ratio test conditions should be tested. The reader is referred to Section 7.5.3 for additional guidance.

As for the unnotched laminate test matrix of Section 2.3.5.2.1, the limited number of tests implies that data pooling using regression analysis across environmental test conditions will be employed (see Section 8.3.5). The exact specification of critical temperatures and moisture conditioning is determined either by the minimum/maximum material operational capabilities (MOL) established for lamina level tests (Sections 2.3.1.1 and 2.3.2.3) or application considerations jointly established by the manufacturer and the procuring agency. To assure upper limits of temperature effects are understood for application laminates, an additional set of "ETW+ DT" tests are performed selectively on the matrix sensitive open hole compression test specimens.

A total of 395 tests are specified if only one laminate thickness and one fastener head style are required to cover application design variables. As in Section 2.3.5.2.1, for two laminate configurations, three replicate tests are repeated for each of three batches of the material system at critical environmental test conditions. Two additional laminates are selectively tested with 5 specimens per test condition using one batch of material. An additional 124 tests are recommended to cover a second fastener head style, and a further set of 206 tests are recommended if a second (191 tests) and third (15 tests) thickness evaluation is required.

TABLE 2.3.5.2.2 *Bearing/bypass*

LAY-UP	THICKNESS	HOLE SIZE DIAMETER	FASTENER HEAD TYPE	COMPRESSION BEARING/BYPASS						
				100% BYPASS			75% BYPASS		50% BYPASS	
				RTD	ETW	ETW+ΔT	RTD	ETW	RTD	ETW
25/50/25	T1	D1	H1	5	9	9	5		5	5
25/50/25	T1	D2	H1	5	9					5
25/50/25	T1	D2,W/D=8	H1	5						
25/50/25	T1	D3	H1	5	9					5
25/50/25	T1	D3,W/D=4	H1	5						
25/50/25	T1	D1	H2	5	9					5
25/50/25	T2	D1	H1		9	9				5
25/50/25	T2	D2	H1							
25/50/25	T2	D3	H1		9					5
25/50/25	T3	D1	H1							
25/50/25	T3	D2	H1							
25/50/25	T3	D3	H1							
50/40/10	T1	D1	H1	5	9	9	5		5	5
50/40/10	T1	D2	H1	5	9					5
50/40/10	T1	D1	H2	5	9					5
50/40/10	T1	D3	H1	5	9					5
50/40/10	T2	D1	H1		9	9				5
50/40/10	T2	D2	H1							
50/40/10	T2	D3	H1		9					5
10/80/10	T1	D1	H1	5	5		5		5	
10/80/10	T1	D1	H2	5	5					
10/80/10	T2	D1	H1		5					
TOTALS				60	123	36	15	0	15	60

NOTES:
1. T1, D1, and H1 are the primary values of laminate thickness, fastener diameter, and fastener head type. T2, T3, D2, D3 and H2 *may be optional* depending on the range of laminate thicknesses and fastener geometries.

laminate strength test matrix.

TENSION BEARING/BYPASS								NUMBER OF TESTS
100% BYPASS		75% BYPASS		50% BYPASS		0% BYPASS		
CTD	RTD	CTD	RTD	CTD	RTD	RTD	ETW	
9	5		5		5	5	9	76
9	5					5	9	47
	5							10
9	5					5	9	47
	5							10
9	5					5	9	47
9						5	9	46
						5	5	10
9							9	32
						5		5
						5		5
						5		5
9	5		5		5	5	9	76
9						5	9	42
9	5					5	9	47
9							9	37
9						5	9	46
						5	5	10
9							9	32
5	5		5		5	5	5	50
5	5					5	5	30
5							5	15
123	50	0	15	0	15	80	133	725

2. "9" represents 3 specimens/batch with 3 batches tested; "5" represents 5 specimens/batch with only 1 batch tested.

2.3.6 Alternate approaches to basis values

2.3.6.1 Lamina mechanical property test matrix for regression analysis

The test matrix of Table 2.3.2.3(b) can be modified for use with regression analysis. Regression analysis allows the pooling of data obtained at different environmental parameters such as temperature, potentially improving the understanding of intermediate temperature effects. It has the added benefit, when used with a suitably sized population of 90 or more datapoints over five or more material batches, of allowing calculation of A-basis statistics for a property, over an environmental range, with a smaller total test population than would otherwise be required.[1] The approach is also particularly useful when an application design temperature changes over the life of the product design, resulting in smaller amounts of data at each of several temperatures.

However, one should be aware of several fundamental assumptions made in statistical regression analysis of strength data, including:

- the failure mode remains constant over the change in the parameter,
- variation remains essentially unaffected by the parameter, and
- parameters that are not included as independent variables (such as moisture content in a regression on temperature) are fixed.

The example regression analysis lamina test matrix shown in Table 2.3.6.1 differs from the point specific test matrix of Table 2.3.2.3(b) in that the "maximum temperature wet" condition has been replaced with three elevated temperature test conditions, providing a more uniform distribution of test data over the temperature range. ET2 represents the maximum operating temperature of a given application. ET1 represents an intermediate elevated temperature above room temperature but below ET2, while ET3 represents an upper end temperature of the material system, such as the MOL. All temperatures are less than either the dry T_g for dry testing or the wet T_g for wet testing. All temperatures represent either a dry material condition or a wet material condition; dry and wet material conditions are not mixed within a given regression analysis. Specific examples of distributed test temperatures include:

!	(350°F epoxy)	-65°F, 73°F, 180°F, 220°F, and 250°F (-50°C, 23°C, 80°C, 100°C, and 120°C)
!	(450°F BMI)	-65°F, 73°F, 250°F, 350°F, and 400°F (-50°C, 23°C, 120°C, 180°C, and 200°C)
!	(600°F polyimide)	-65°F, 73°F, 350°F, 450°F, and 550°F (-50°C, 23°C, 180°C, 230°C, and 290°C)

For data submission for handbook publication the standard population sampling and data documentation requirements discussed in Section 2.5 remain in effect.

[1]This assumes the data have a coefficient of variation no higher than 15%. If the CV is larger, more data points will need to be added to the test matrix in order to calculate an A-basis value. If B-basis values are to be calculated, only 30 data points are needed over the temperature range to achieve the same confidence level. Each batch should be distributed over the temperature range as uniformly as possible, and at least three batches must be represented at any one test condition.

TABLE 2.3.6.1 *Cured laminate mechanical property test matrix designed for regression analysis.*

A-basis level matrix - 5 batches/90 data points per property

Mechanical Property	Test Methods[1]	Test Condition[2] and Number of Tests Per Batch[3]					Number of Tests
	See Handbook Section	Min	RT	ET1	ET2	ET3	
		Temp					
0° Tension (warp)	6.7.4.4[4]	3	4	3	4	4	90
90° Tension (fill)	6.7.4.4[4]	3	4	3	4	4	90
0° Compression (warp)	6.7.5.4	3	4	3	4	4	90
90° Compression (fill)	6.7.5.4	3	4	3	4	4	90
In-plane Shear	6.7.6.4	3	4	3	4	4	90
0° Short Beam Shear[5]	6.7.6.4	-	6	-	-	-	30
							480

1. MIL-HDBK-17 is not currently in a position to make exclusive test method recommendations, but the referenced Handbook sections identify methods that are currently deemed acceptable for data submittals to MIL-HDBK-17.
2. Minimum and maximum temperature tests should be performed within ±5°F (±2.8°C) of the nominal test temperature. Nominal test temperatures will be as agreed to by contractor and certifying agency. Dry specimens are "as-fabricated" specimens which have been maintained at ambient conditions in an environmentally-controlled test laboratory. Wet specimens are environmentally-conditioned by exposing them in a humidity chamber until they attain an equilibrium moisture content agreed to by the contractor and certifying agency, and then packaging them in a heat-sealed aluminized polyethylene bag until required for test. Tests should be performed in a manner which maintains the moisture content in specimens at the levels agreed to by the contractor and certifying agency.
3. Tests should be performed on each of the five batches.
4. For 0° and 90° tension, ASTM D 3039 and SACMA Recommended Method (SRM) 4-88 are acceptable test methods for MIL-HDBK-17 data submittals.
5. Short Beam Shear is for screening and quality control purposes only.

Important Note: This matrix is intended for the generation of dry coupon data. Wet data can be generated by duplicating this test matrix in the wet condition or by generating hot/wet data at the application specific temperature.

Other important notes:
Min Temperature is normally -65°F
ET2 is the maximum application temperature
ET3 should be less than T_g temperature of the material,
dry T_g if testing is done dry, wet T_g is the testing is done wet.

2.3.7 Data substantiation for use of basis values from MIL-HDBK-17 or other large databases

To reduce development costs for new composite applications, designers and manufacturers need to make use of basis values and properties from large, existing composite materials databases without having to perform tests that essentially duplicate the database. To do this, the user must demonstrate the equivalency of the properties of the composite material processed per their processing parameters and in their design configuration to the properties of the original database material. The demonstration of equivalency is a crucial step in the concept of shared databases. If unable to establish equivalency, the user will not likely be able to use the larger, shared material database for certification purposes without significantly increased amounts of testing.

To use basis values from MIL-HDBK-17 (or from other databases) in design, the using organization should demonstrate the ability to consistently produce the same material as that evaluated during the material testing program. As a minimum, the substantiation tests identified in Table 2.3.2.3(b) should be conducted for this purpose. A total of six specimens per loading condition are required, either using two independently processed material batches or two panels from a single material batch, processed independently. This amounts to twelve specimens per condition. Other test matrices may be acceptable (for example, see Reference 2.3.7) if replication is sufficient to evaluate critical mechanical properties. The statistical procedures used to validate that the data are from the same population as that for which the original basis value was determined are summarized in Section 8.4.1. The use of basis values from any MIL-HDBK-17 data class depends on agreement between the manufacturer and the certifying agency. Deviations from the recommended lamina-level substantiation testing, for example, a reduction or increase in the number of loading conditions evaluated, also depend upon such agreements.

The recommended test matrices and statistical procedures to demonstrate material equivalency are only applicable to the following specific situations:

(a) An identical material processed by the same part manufacturer using identical fabrication process at a different location,
(b) An identical material processed by a different part manufacturer using a process that is equivalent to the original database process,
(c) An identical material processed by the same part manufacturer using a follow-on process that is slightly different from the original process,
(d) Minor changes by the material supplier in the prepreg constituent(s) and/or constituent manufacturing process, or
(e) Combinations of the above.

The specific types of changes to the follow-on material system and/or process that may be considered as minor changes include but are not limited to:

(a) Increasing the cure pressure or vacuum level for the follow-on process. This includes changing from oven curing (vacuum only) to autoclave curing. Decreasing the cure pressure or vacuum level for the follow-on process, however, is generally considered a major change.
(b) Minor change in cure parameters such as dwell time and heat-up rate.
(c) Prepreg tack.

The types of changes to the follow-on material system that are considered as major changes, which are not covered by this section but are addressed in Section 2.3.4 Alternate Materials, include:

(a) Change of fiber (for example, changing from AS4 to T300 or IM7 fibers)
(b) Change of resin (for example, changing from 3501-6 to E7K8 resin)
(c) Fabric weave style (for example, changing from 8 harness satin weave to plain weave)
(d) Tow size of fabric (for example, changing from 6K tow to 3K tow)

Further evaluation or testing may be required depending on the extent of the changes. For example, increasing the prepreg tack may result in higher volatile content. Higher volatile content has been known

to cause higher void content and lower glass transition temperature in the cured laminate. Sections 2.3.4, 2.5.3.4 and 8.4.2 provide further guidance on this subject.

A successful material equivalency demonstration does not imply that the follow-on material and/or follow-on process will also yield equal properties at laminate, element and sub-component levels, as the manufacturing complexity of a particular application may result in different properties. Tests at these levels are typically needed to fulfill the remaining parts of the structural substantiation requirements.

Engineering judgment is a critical element of the equivalency demonstration process. If a mechanical property at one temperature does not show statistical equivalence, the importance of that property and the size of the discrepancy should be investigated before declaring that the material is not equivalent to the shared database material. For example, for fiber dominated laminates tensile strength and modulus and elevated temperature, wet compressive strength and modulus are examples of properties that are usually design critical and more importance should be placed on the statistical test results of these properties.

In addition to the use of material equivalency testing to take advantage of shared databases, similar testing is used to determine whether the natural variations in the material or process alter key properties. Another form of equivalency testing is the material batch acceptance testing using to control materials. Such continuous sampling is performed to ensure the material meets specification requirements, and, when used with a statistical process control system, that the material properties are not changing over time.

2.4 DATA REDUCTION AND DOCUMENTATION

2.4.1 Introduction

This section is reserved for future use.

2.4.2 Lamina properties from laminates

The mechanical properties of composites have increased markedly as materials have evolved. Carbon fiber composite tensile strengths and strains at failure, for example, nearly doubled during the 1980's (Reference 2.4.2(a)). As properties have improved, however, some test methods that were adequate for previous generations of composites are no longer suitable for characterizing the full capabilities of high strength advanced material systems.

The most serious problems relate to accurate determination of basic lamina (ply) tension and compression strengths which, traditionally, have been characterized using unidirectional test specimens for tape and similar form composites. As material capabilities have advanced, the deficiencies associated with these specimens have been greatly amplified. While it is possible in some cases to generate acceptable strength data with unidirectional specimens, extreme care is required in their design and fabrication, thus adding significant cost. As an alternative, data from the testing of crossply[1] laminates have been used by an increasing number of workers to indirectly calculate lamina properties by classical lamination theory.

There are numerous arguments that support this approach. The most frequently claimed advantages are higher (more realistic) strength values with lower data scatter, both of which have been demonstrated by a number of investigators (e.g., References 2.4.2(a) and (b)). Higher values are attributed to reduction or elimination of premature failures stemming from various causes which are discussed later. Lower variability is associated with less sensitivity to specimen quality fluctuation and small manufacturing defects. This reduction in sensitivity reflects more closely the response of structural configurations.

[1]The term “crossply” is used as defined in Section 1.7, which differs from other definitions used in the industry. Here it is synonymous with “angleply” and “multi-directional,” and is not restricted to laminates of the [0/90] family.

Perhaps the most compelling reason for using crossply testing is that it is more closely representative of application laminates used in actual structural components. Since, in general, a ply may respond to loads differently when adjacent to plies of different orientation than when in isolation (or adjacent to plies of the same directionality), it makes sense to characterize ply properties in their end-use setting. In this way, ply values used in laminate analysis will be more representative of properties expected of the ply in the laminate being analyzed, not those of a ply in isolation.

Although this approach is not a panacea for all testing difficulties, it is becoming quite common in the advanced composites industry, and standardization activities are in progress. The method does offer advantages and should be considered when planning test programs. For additional information the reader is referred to References 2.4.2(c) and (d).

2.4.2.1 Methodology

The general approach for determining longitudinal lamina strength is to select, fabricate, and test a suitable multidirectional laminate, and then calculate 0° ply tensile or compressive strength using classical lamination theory. This methodology makes some assumptions:

1. The laminate fails by the same mechanism and at the same strain as the plies in a unidirectional specimen that does not fail prematurely.

2. The stress-strain curves for both the laminate and lamina are essentially linear elastic to failure (a methodology for use when this does not hold is briefly discussed).

3. The values of $E1$, $E2$, and $\nu 12$ for the ply used in the equations are valid at incipient failure.

4. Effects of ply residual stresses and damages such as ply cracks are negligible.

Given these assumptions, it is clear that not all laminates are suitable. A family of laminates that has been found useful, and for which the bulk of test data exists, is the $[0x/90y]_{ns}$. In this family the $[0/90]_{ns}$ is most widely used. While this laminate is not commonly used for actual structure, it does provide an environment where adjacent plies are of different orientation. In addition, the calculated factor (discussed below) is reasonably low. Quasi-isotropic laminates have also been used successfully, but the factor is almost twice as high as for the 0°/90° laminates, giving somewhat less confidence. Laminates with so many ±45° plies as to cause $\nu 12$ for the laminate to exceed $\nu 12$ for the lamina are not preferred because the strain to failure may not be as great as for the unidirectional specimens. Some composites with very brittle resin matrices do not permit the fabrication of quality 0°/90° laminates due to splitting during cool-down after cure. In such cases some ±45° plies must be included. Within a family of laminates, stacking sequence will have an effect. Laminates with several plies of the same orientation stacked together (thick layers) will generally yield lower compressive strength values than more homogeneous lay-ups (Reference 2.4.2.1). Obviously, symmetric laminates must be used in all cases to preclude bending.

The third assumption presumes that $E1$, $E2$, and $\nu 12$ have been obtained from other tests (most likely, unidirectional specimens). This does not present a serious problem, since the shortcomings of unidirectional specimens do not affect modulus measurements to the same degree as strength measurements. It can be argued that $E2$ (and to some degree $E1$) is not linear to failure, and is usually calculated significantly below the failure load. However, as discussed in detail later, this is not a significant issue due to the rather low sensitivity of this methodology to variation in $E2$.

To calculate lamina strength, the measured test laminate strength is multiplied by a crossply factor (CPF) generated from classical lamination theory:

$$F_1 = CPF \bullet F_x \qquad 2.4.2.1(a)$$

For the $[0_x/90_y]_{ns}$ family of laminates this factor, based on the assumption of uniform strains in each ply, is calculated according to the following formula:

$$CPF = \frac{E_1\left[mE_2 + (1-m)E_1\right] - (\nu_{12}E_2)^2}{\left[mE_1 + (1-m)E_2\right]\left[mE_2 + (1-m)E_1\right] - (\nu_{12}E_2)^2} \qquad 2.4.2.1(b)$$

where m is the fraction of 0° plies in the laminate (E_1, E_2, and ν_{12} are for tension or compression as appropriate).

For $[0/90]_{ns}$ laminates (equal numbers of 0° and 90° plies), the formula reduces to:

$$CPF = \frac{E_1\left(\frac{E_1+E_2}{2}\right) - (\nu_{12}E_2)^2}{\frac{(E_1+E_2)^2}{4} - (\nu_{12}E_2)^2} \qquad 2.4.2.1(c)$$

As stated above, these equations are not very sensitive to variability in E_2, and show very little response to changes in ν_{12}. For $[0/90]_{ns}$ laminates with E_1 = 20 Msi, a 20% change in E_2 results in less than 2% change in the factor, and a 20% change in ν_{12} has negligible effect. Therefore, E_2 and ν_{12} do not have to be quantified with great accuracy (the precise effect on the factor will, of course, depend on the actual ratio of E_1 to E_2). The shear moduli of a ply are a function of stress. Since these moduli affect stability, and hence compressive strength, there may be some difficulties with soft matrix materials.

Many times a value for E_2 may not be available at all. If this is the case, there is an alternate approach, which may be preferable even if E_2 has been determined. This method involves measuring only E_1 from a unidirectional specimen, and E_x of the crossply laminate being tested. Under assumption 1 that the test laminate fails at the same strain as a unidirectional specimen, the lamina strength may be calculated as follows:

$$F_1 = \frac{E_1}{E_x}F_x \qquad 2.4.2.1(d)$$

Very good agreement has been reported between the E_1/E_x ratio and the factor, F, obtained as described above (Reference 2.4.2(a)).

All of the methodology described thus far assumes linear stress-strain behavior to failure. If this is not the case (as in some crossply glass/epoxy laminates, for example), the fiber direction lamina strength can be calculated as follows:

$$F_1 = E_1\left(\varepsilon_x + \nu_{21}\varepsilon_y\right)/\left(1-\nu_{12}\nu_{21}\right) \qquad 2.4.2.1(e)$$

where the strains (ε) in the x and y directions are those measured at failure.

If Poisson effects can be neglected, the above equation reduces to:

$$F_1 \approx E_1\varepsilon_x \approx F_x\frac{E_1}{E_x^*} \qquad 2.4.2.1(f)$$

where E_x^* is the secant modulus of the laminate at failure. This equation is useful when and E_2 are not known.

2.4.2.2 Tension strength tests

Well designed and fabricated unidirectional tensile specimens can give good results for advanced composites, but this is generally the exception rather than the rule. One major problem is premature failure at the tips of adhesive bonded tabs, particularly when they are tapered gently rather than square cut and gripped over their entire length. The higher loads required to test advanced composites often result in high peel forces at the tab ends and subsequent interlaminar tension failure of the first ply of the composite. Once this has occurred, most of the load is taken by this outer ply, which then fails in tension and results in tab loss. Since lower loads are required to test crossply specimens, this is much less likely to happen.

Rawlinson (Reference 2.4.2(a)) and others have investigated various laminate stacking sequences. Both 0/90 and 0/±45 balanced laminates yielded mean strength values comparable to those measured from the best quality unidirectional specimens, and had significantly less data scatter. In addition, it has been demonstrated that some of these laminates can be tested successfully without bonded tabs using hydraulic grips, thus offering additional testing economy. What have sometimes been referred to as "tab-less" specimens actually require an interlayer between the grips and the specimen: for example, a sheet of emery paper with the abrasive side in contact with the specimen, or an abrasive-coated wire mesh with a sheet of plastic to protect the jaws of the test machine. If bonded tabs are not used, 0° plies should not be on the outside surfaces of the laminate since damage may be inflicted by the grips. Thus, for crossply testing without bonded tabs, a $[90/0]_{ns}$ laminate would be preferred over $[0/90]_{ns}$. It should be noted that surface strain measurements are more sensitive to matrix cracking of the outer 90° plies in the $[90/0]_{ns}$ configuration. If bonded tabs are used, the stacking sequence of the tab material is important to consider (Reference 2.4.2(b)) (see Section 6.7.4). For coupons with tabs there appears to be little difference between results from $[90/0]_{ns}$ and $[0/90]_{ns}$ laminates.

2.4.2.3 Compression strength tests

As in tensile testing, the high loads needed to test advanced composites cause problems in compression testing of unidirectional specimens. In compression, end "brooming" and longitudinal splitting are common modes of premature failure. Occurrence of these modes is greatly reduced or eliminated by crossply specimens, which tend to fail in microbuckling or ply buckling (Reference 2.4.2.3). Furthermore, low sensitivity to methods of loading and end constraint has been reported for quasi-isotropic laminates (Reference 2.4.2.3). The same result has been reported by others for $[0/90]_{ns}$ laminates. This suggests that the capability of the material is being evaluated, not the capability of the test method.

There is currently no consensus regarding the "best" laminate stacking sequence to be used, although $[0/90]_{ns}$ has been commonly employed. These specimens are reported to give high strength values and low data scatter. Data from several sources (yet unpublished) indicate that $[90/0]_{ns}$ laminates yield higher mean values than $[0/90]_{ns}$. The reason for this increase has not been conclusively established, but has been attributed to several factors. First, there is speculation that the 90° outer plies act to protect the load bearing 0° plies from damage which might be inflicted during specimen fabrication or testing. Such damage could provide sites for initiation of premature failure if inflicted on 0° plies. Second, it is thought that the presence of the outer 90° plies enhances the stability of the otherwise outer 0° plies. If this is true, structural analysts will have to determine if design properties derived from $[90/0]_{ns}$ laminates are appropriate for specific applications where outer 0° plies are aligned with the primary compressive load direction. Third, it is known that 0° outer plies increase stress concentrations at the ends of the gage area for tabbed test specimens, and this is suspected to contribute to premature failure. Fourth, outer 0° plies might split as a result of transverse tensile stresses induced by Poisson effects.

2.4.2.4 Other properties

Transverse strengths of unidirectional composites have always been difficult to characterize because of premature failures due to extreme notch sensitivity. In an effort to improve this situation, a few studies using crossply laminates have been undertaken, but these are not well documented. There is relatively low interest in pursuing this since, for the analysis of most structure, the accuracy of these strength values does not significantly affect the result unless the transverse strength used in the analysis is so low as to cause a false prediction of a "first ply" failure.

$[+45/-45]_{ns}$ laminates tested in tension have commonly been used to derive [0/90] (matrix-dominated) in-plane shear strength and modulus properties. This method generally produces a strength result that is a lower bound of the true material shear capability. See Section 6.7.4 of this volume for more detail.

2.4.3 Data normalization

Data analysis is performed on mechanical test data for a variety of reasons that include determination of multi-batch statistics and statistically based property values (allowables), comparison of materials from

different sources, material selection, evaluation of processing parameters, and quality assurance evaluation. Such calculations or direct comparisons may not be valid if test specimens having different fiber volume contents were tested. Normalization is a procedure for adjusting raw test values to a single (specified) fiber volume content. The following sections discuss the theory, methodology, and practical application of normalization.

2.4.3.1 Normalization theory

Mechanical properties that are dominated by the properties of the reinforcing fiber are dependent on the volume fraction of fiber in the laminate. In the commonly used "rule of mixtures" model, 0° tensile strength of a unidirectional laminate, for example, is assumed equal to the matrix tensile strength at 0% fiber volume, and equal to the fiber strand tensile strength at 100% fiber volume. Neglecting the effects of resin starvation at high fiber contents, the relationship between fiber volume fraction and ultimate laminate strength is, therefore, linear over the entire range of fiber/resin ratios. This follows from the fact that volume percent fiber is the same as the area percent fiber in the specimen cross-section. Tensile modulus is expected to follow the same behavior. Thus, test specimens having different fiber volume contents have fiber-dominated properties that vary linearly with fiber volume fraction.

Two factors can cause laminate fiber volume fraction to vary: (1) the amount of matrix resin present relative to the amount of fiber (resin content), and (2) the amount of porosity (void volume). These factors give rise to changes in fiber volume fraction from material to material, batch to batch, panel to panel, and even specimen to specimen within a panel. In order to perform data analysis that compares materials, batches, panels, or specimens, the data for fiber-dominated properties must be adjusted to a common fiber volume fraction. If this is not done, an additional source of variability will be included in the data that might lead to erroneous conclusions. The process of data normalization attempts to remove or reduce this source of variability in fiber-dominated properties.

2.4.3.2 Normalization methodology

Since, in theory, fiber-dominated strength and stiffness properties vary linearly with fiber volume fraction, an obvious first approach would be to determine the actual fiber volume fractions of the test specimens by an appropriate method (matrix digestion, ignition, optical techniques, etc.), and to adjust raw data values by the ratio of a common fiber volume fraction (chosen or specified) to the actuals as shown in Equation 2.4.3.2(a).

$$\text{Normalized value} = \text{Test value} \times \frac{FV_{normalizing}}{FV_{specimen}} \qquad 2.4.3.2(a)$$

where

$FV_{normalizing}$ = chosen common fiber content (volume fraction or %)
$FV_{specimen}$ = actual specimen fiber content (volume fraction or %)

Although this would appear to be the most direct approach, it has limitations. The most serious deficiency is that fiber volume is not commonly measured for each individual test specimen. At best, representative pieces from each test panel are used to estimate the average panel fiber volume fraction. Since resin content might vary significantly within a panel (due to resin movement during processing and other factors), the fiber volume fraction might not be the same for all specimens cut from the panel. As a result, accurate normalization of each individual specimen is not possible. In addition, digestion methods can be problematic with some material systems, and considerable skill is required for accurate, repeatable results (see Section 6.4.6 for information on fiber volume methods).

A preferred method of data normalization employs an approach that accounts for the fiber volume variation between individual test specimens. The basis of this method is the relationship between fiber volume fraction and laminate cured ply thickness. As stated earlier, laminate fiber volume fraction is a function of resin content and void content. At a given void content, laminate fiber volume fraction is entirely dependent upon resin content. Furthermore, for a given void content and fiber areal weight, panel

thickness (and hence cured ply thickness) is also dependent only upon resin content. Thus, it follows that cured ply thickness is solely dependent upon fiber volume fraction for constant fiber areal weight and void content. This dependency permits normalization of each individual test specimen by its ply thickness (total thickness divided by number of plies). An example of this relationship between cured ply thickness and fiber volume fraction (which is virtually linear within the 0.45 to 0.65 fiber volume fraction range of usual interest for structural composites) is shown in Figure 2.4.3.2.

The following describes the derivation of an equation for normalizing each individual test specimen. Using the relationships discussed in the previous paragraph, expressions for $FV_{normalizing}$ and $FV_{specimen}$ are developed and substituted into Equation 2.4.3.2(a). For illustrative simplicity compatible units of measure are assumed.

The first step is to define an equivalent thickness of fiber which would result if the fiber material could be shaped into a solid sheet of uniform thickness with no air space between filaments:

$$t_f = \frac{FAW}{\rho_f} \qquad 2.4.3.2(b)$$

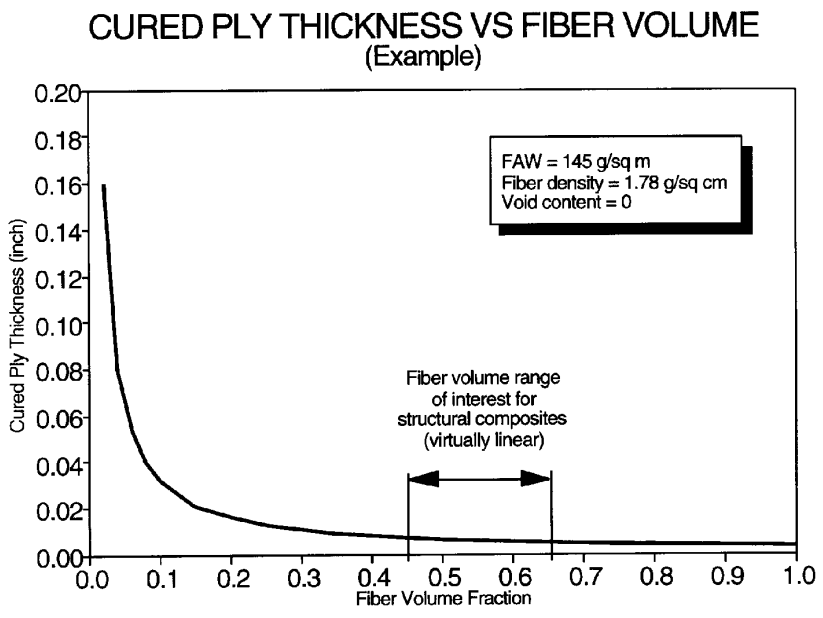


FIGURE 2.4.3.2 *Example of correlation of cured ply thickness with fiber volume fraction.*

where

t_f = equivalent thickness of a solid layer of fiber
FAW = reinforcement fiber areal weight
ρ_f = fiber density

The fraction of fiber in a laminate is then the thickness of this fiber layer divided by the total laminate thickness:

$$FV = \frac{t_f}{CPT} \qquad `2.4.3.2(c)$$

where

FV = fiber volume fraction

CPT = laminate cured ply thickness

From Equations 2.4.3.2(b) and 2.4.3.2(c) it follows that

$$FV = \frac{FAW}{\rho_f \, x CPT} \qquad 2.4.3.2(d)$$

This is the equation that was plotted for the example in Figure 2.4.3.2. It then follows that

$$FV_{normalizing} = \frac{FAW_{nominal}}{\rho_f \, x \, CPT_{normalizing}} \qquad 2.4.3.2(e)$$

and

$$FV_{specimen} = \frac{FAW_{specimen}}{\rho_f \, x \, CPT_{specimen}} \qquad 2.4.3.2(f)$$

where

$FV_{normalizing}$ = fiber volume fraction specified or chosen for normalizing
$FV_{specimen}$ = fiber volume fraction of the specimen
$FAW_{nominal}$ = nominal fiber areal weight from a material specification or other source
$FAW_{specimen}$ = specimen actual fiber areal weight
$CPT_{normalizing}$ = cured ply thickness corresponding to normalizing fiber volume fraction
$CPT_{specimen}$ = actual specimen ply thickness (specimen thickness divided by number of plies)

Combining Equations 2.4.3.2(e) and 2.4.3.2(f), the following is obtained:

$$\frac{FV_{normalizing}}{FV_{specimen}} = \frac{FAW_{nominal}}{FAW_{specimen}} \, x \, \frac{CPT_{specimen}}{CPT_{normalizing}} \qquad 2.4.3.2(g)$$

and substituting 2.4.3.2(g) into 2.4.3.2(a) produces:

$$\text{Normalized value} = \text{Test value} \, x \, \frac{FAW_{nominal}}{FAW_{specimen}} \, x \, \frac{CPT_{specimen}}{CPT_{normalizing}} \qquad 2.4.3.2(h)$$

Thus, each specimen can be normalized by multiplying the test value by the ratios of fiber areal weight and cured ply thickness shown. The normalizing cured ply thickness is calculated by rearranging Equation 2.4.3.2(e) as follows:

$$CPT_{normalizing} = \frac{FAW_{nominal}}{FV_{normalizing} \, x \, \rho_f} \qquad 2.4.3.2(i)$$

While Equation 2.4.3.2(h) is illustrative of the model initiated in Equation 2.4.3.2(a), it is not necessary to calculate $CPT_{normalizing}$ if Equation 2.4.3.2(h) is transformed to:

$$\text{Normalized value} = \text{Test value} \, x \, \frac{FV_{normalizing} \, x \, CPT_{specimen} \, x \, \rho_f}{FAW_{specimen}} \qquad 2.4.3.2(j)$$

The value for $FAW_{specimen}$ is defined as the actual fiber areal weight for each individual specimen, but this measurement is not made on a specimen basis. However, since fiber areal weight does not usually vary greatly within a batch of material, the batch average (or roll average, if available) fiber areal weight is generally sufficient for normalization. In the case of laminates made by resin transfer molding (RTM) or other non-prepreg processes, lot or roll average areal weights for the fabric or preforms should be used. With this assumption that batch fiber areal weight approximates specimen fiber areal weight within a batch, Equation 2.4.3.2(j) becomes:

$$\text{Normalized value} = \text{Test value} \, x \, \frac{FV_{normalizing} \, x \, CPT_{specimen} \, x \, \rho_f}{FAW_{batch}} \qquad 2.4.3.2(k)$$

In actual practice, fiber areal weight is commonly reported in g/m^2 and fiber density in g/cm^3, while ply thickness may be in inches or millimeters. For these units, Equation 2.4.3.2(k) requires a conversion factor of 25,400 in the numerator if ply thickness is in inches, or a factor of 1000 if in millimeters. With these factors included, Equation 2.4.3.2(k) becomes:

$$\text{Normalized value} = \text{Test value} \, x \, \frac{25{,}400 \, x \, FV_{normalizing} \, x \, CPT_{specimen} \, x \, \rho_f}{FAW_{batch}} \qquad 2.4.3.2(l)$$

or

$$\text{Normalized value} = \text{Test value} \times \frac{1000 \times FV_{normalizing} \times CPT_{specimen} \times \rho_f}{FAW_{batch}} \qquad 2.4.3.2(m)$$

where

$FV_{normalizing}$ = fiber volume fraction specified or chosen for normalizing
$CPT_{specimen}$ = actual specimen ply thickness (specimen thickness divided by number of plies), inch (Equation 2.4.3.2(l)) or mm (Equation 2.4.3.2(m))
ρ_f = fiber density, g/cm^3
FAW_{batch} = batch average fiber areal weight, g/m^2

As stated earlier, void content affects fiber volume fraction. If porosity is "added" to a laminate, the thickness will increase and the fiber volume fraction will decrease. However, for a given fiber areal weight, the change in fiber volume fraction will be the same regardless of the source of a thickness change (resin content change or void content change). Thus, when normalizing using Equation 2.4.3.2(l) or 2.4.3.2(m), there is no need to make any adjustment for void volume. This assumes, of course, that the void content is not so large or localized that basic load carrying capability is reduced.

A hybrid method uses both individual specimen thickness and fiber volume data obtained by experimental methods (matrix digestion, ignition, optical techniques, etc.). This approach is shown by Equation 2.4.3.2(n):

$$\text{Normalized value} = \text{Test value} \times \frac{CPT_{specimen}}{CPT_{batchavg.}} \times \frac{FV_{normalizing}}{FV_{batchavg.}} \qquad 2.4.3.2(n)$$

where

$CPT_{specimen}$ = actual specimen ply thickness (specimen thickness divided by number of plies)
$CPT_{batch\ avg.}$ = batch average cured ply thickness calculated from a number of panel or specimen thickness measurements
$FV_{normalizing}$ = fiber volume fraction specified or chosen for normalizing
$FV_{batch\ avg.}$ = batch average fiber volume fraction calculated from a number of experimental fiber volume determinations from panels within the batch

In Equation 2.4.3.2(n), the test value is first adjusted by specimen ply thickness to an average batch ply thickness. This essentially normalizes the data to a common fiber volume fraction, presumably the batch average fiber volume fraction. The second ratio in Equation 2.4.3.2(n) then makes a further adjustment from the batch average fiber volume fraction to the normalizing fiber volume fraction. This method can be useful when fiber areal weights are not available. However, this approach requires another assumption: that the specimens used to experimentally determine batch average fiber volume fraction had an average ply thickness equal to $CPT_{batch\ avg.}$. This is not generally the case, since batch average cured ply thickness may be determined from many measurements over a number of panels, while batch average fiber volume fraction may be obtained from comparatively few specimens. If fiber volume specimens are selected carefully so they are representative of batch ply thickness, this method may be used successfully.

2.4.3.3 Practical application of normalization

Common practice is to normalize fiber-dominated lamina and laminate strengths (both unnotched and notched) and moduli for laminates fabricated from tapes, fabrics, and rovings. Although fiber volume effects on various matrix-dominated properties (in-plane and interlaminar shear, for example) have been observed, there is no clear model for these effects, and such properties are not normalized. In Volume 2 of this Handbook, normalized values are presented for all mechanical strength and stiffness properties *except*: 90° (transverse) tension and compression of unidirectional laminates, interlaminar (3- or z-direction) tension, interlaminar shear, in-plane shear, short beam strength, bearing, bearing/bypass, strain energy release rate, and Poisson's ratio.

Laminates fabricated from rovings and similar forms using a winding process present a unique situation relative to normalization. Such constructions do not have plies in the usual sense: the wound "ply" thickness depends upon tow band width, wind spacing, and tow spread during winding. Since nominal ply thickness and fiber areal weight are not directly applicable, normalization by ply thickness and fiber areal weight is not possible. Test data for these materials must be normalized using the ratio of normalizing fiber volume fraction to the average measured panel fiber volume fraction (Equation 2.4.3.2(a)).

When fiber-dominated properties are normalized, data scatter should decrease compared to the unnormalized values since variability due to fiber volume fraction differences is being reduced. Thus, coefficients of variation should be lower after normalization. However, this is not always observed, and there are a number of reasons why the reduction in scatter expected from normalization is not invariably realized:

1. If measured cured ply thicknesses are close to the normalizing thickness and fiber areal weight is close to nominal, correction factors will be small, and may be nearly the same magnitude as errors in measuring these quantities.

2. The mode of failure initiation may change as a function of fiber volume. As an example, measured (unnormalized) compression strength may increase as fiber volume fraction increases over a given range. However, at some point additional fiber may not increase strength because the ability of the matrix to support the fibers has been exceeded, and a stability failure occurs on a macro scale. In this case, the relationship between strength and fiber volume breaks down, and data scatter is not necessarily reduced by normalization.

3. Flaws in test specimens might cause premature failures. If some specimens fail because of flaws and others at the true material limit, results of normalization will not be predictable.

4. If the coefficient of variation is already small (less than 3%, for example), further reduction as a result of normalization should not be expected, since this level of variability is about the minimum usually observed for most composite properties.

No change in data scatter after normalization is usually not a cause for concern. However, if data scatter *increases* significantly after normalization, the reason should be investigated.

2.4.4 Dispositioning of Outlier Data

Detection of outlier data points (observations which are much lower or much higher than other observations in a data set) is part of statistical analysis and is discussed in Volume 1, Section 8.3.3. Although Section 8.3.3 cautions against discarding outlier data for which no clear cause for its erroneous nature has been found, there are cases where outliers can (and should) be removed based on judgment. The following paragraphs attempt to attach some degree of structure to the judgment process so that outliers that should be retained are not casually discarded, and those which should be deleted are not retained.

For purposes of this discussion, it is assumed that the objective of testing is to characterize the properties of a material when processed, conditioned, and tested in accordance with specified procedures and parameters. If this is the case, variability within the test data should (ideally) reflect only material variability (raw material quality, constituent process variability, mix ratios, etc.), processing parameter variability (within the control ranges for the specimen fabrication process), variability in the environmental history of the specimens prior to test (within control limits), and variability within the tolerances of the test machine parameters. In reality there is also unavoidable random variability due to unknown and uncontrollable factors. However, beyond these inevitable and acceptable sources of variability are sources of error that inflate the observed data scatter. This additional variability may be due to inferior fabrication practices, process parameters that exceed allowed control limits, test fixture or test machine deficiencies, and any number of other factors both detectable and undetectable. The task in dealing with outlier data is to determine (based on physical evidence and judgment) whether the data variability is from a source that re-

flects accepted material and process variability (in which case the outlier data is retained), or from an erroneous external source (in which case the outlier data is discarded).

When outlier data is detected (either by visual inspection of the data or by statistical tests), the first action should be to identify the cause through physical evidence. The following list gives some examples of conditions that could be used as the basis for discarding outlier data (the list is not exhaustive):

1. The material (or a constituent) was out of specification
2. One or more panel or specimen fabrication parameters were outside the specified tolerance range
3. Test specimen dimensions or orientation were outside the specified tolerance range
4. A defect (not under study) was detected in the test specimen
5. An error was made in the specimen preconditioning (or conditioning parameters were out of specified tolerance ranges)
6. The test machine and/or test fixture was improperly set up in some specific and identifiable manner
7. The test specimen was improperly installed in the test fixture in some specific and identifiable manner
8. Test parameters (speed, test temperature, etc.) were outside the specified range
9. The test specimen slipped in the grips during test
10. The test specimen failed in a mode other than the mode under test (loss of tabs, unintended bending, failure outside the gage section, etc.)
11. A test was purposely run to verify conditions suspected to have produced outlier data
12. Data were improperly normalized

Once the search for physical causes has been completed without success, the judgment process begins. There are many approaches to assessing outlier data for which no physical cause has been identified. The following is suggested as one possible process, and follows the flow chart shown in Figure 2.4.4.

When an outlier is detected, it may or may not be a cause of concern. If its inclusion in the data does not significantly affect calculated basis values and does not raise other engineering issues, it may simply be retained without further consideration.

If an outlier data point was detected in a single batch or set of data, and if additional data sets are available (same fabrication, conditioning, and test conditions), there are a number of considerations that can be used to support a judgment call. If, on the other hand, additional data sets are not available, or if the outlier was detected only after combining (pooling) several data sets (see Volume 1, Section 8.3.1), there are fewer options to consider and judgment is more subjective.

In the case of a single data set outlier where additional data sets are available, the first consideration is to determine if the outlier in question is within the range of the non-outliers of the other data set(s). If it is within the non-outlier range of the other data, it is recommended that the outlier be retained.

If a single data set outlier is outside of the non-outlier range of other data, the next option is to obtain retest data using specimens from the same part or panel as the original data that contained the outlier. If the retest data refutes the outlier data, the retest data set may be used to replace the entire original data set. The replacement set is then combined with the other data sets. If outliers still exist in the retest data, the original data is retained and combined with the other data sets. The retest data set may also be added to the body of data. Regardless of whether the original data was replaced or not, the combined set is then tested for outliers. If no outliers are detected in the combined set of data, no deletions from the combined set are made.

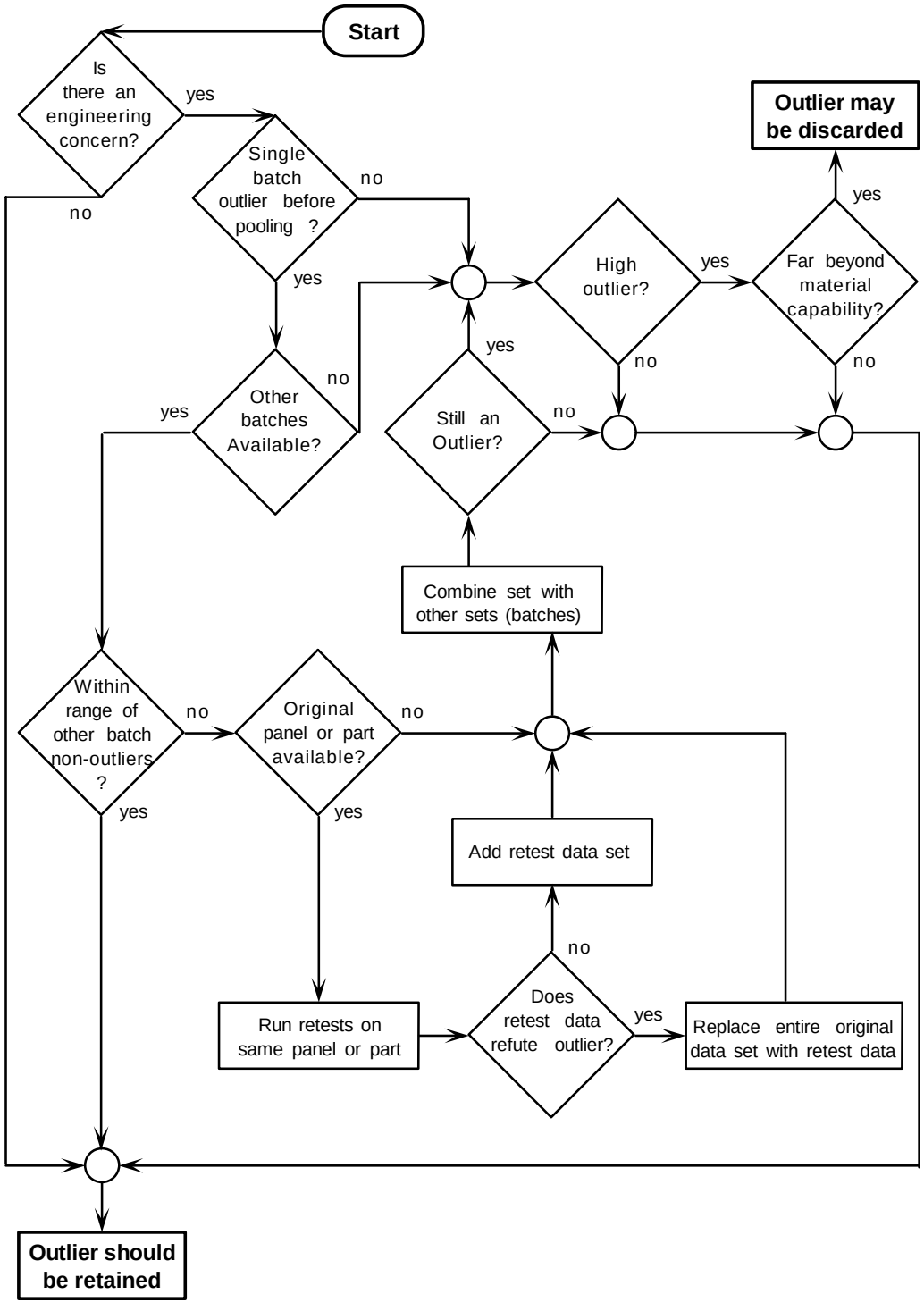


FIGURE 2.4.4 *Judgment process for dispositioning of outlier data without identified cause.*

If there are outliers in the combined data set (or in a single set where no additional sets were available), the judgment process becomes even more subjective. In this case it is recommended that only high outliers be considered for deletion. Generally, if a high outlier is so high that, based on experience and similar test results from other sources, it is clearly beyond the known or expected capability of the material, it may be discarded. If low outliers exist in single data sets and no additional sets are available, additional batches should be tested and the judgment process repeated. Retests of the same single batch are not considered sufficient without additional batch information.

Note that the judgment process does not specifically identify a cause, and does not really prove that the questionable data resulted from erroneous variability. The judgment process attempts to build a body of information that leads to the conclusion that it is highly likely that erroneous variability (caused by something other than expected material, process, or testing variations) was responsible for the outlier. This approach to outlier disposition should only be used after all attempts to identify and quantify physical causes have failed. The rationale for any data deletions made by the judgment process should be fully documented.

2.4.5 Data documentation

This section is reserved for future use.

2.5 MATERIAL TESTING FOR SUBMISSION OF DATA TO MIL-HDBK-17

2.5.1 Introduction

Section 2.5 describes the requirements for publication of material property data in MIL-HDBK-17 Volume 2. A Data Source Information Package is available from the MIL-HDBK-17 Coordinator or Secretariat to aid data suppliers in submitting data to the Handbook. This package provides recommendations on data preparation and transfer and a diskette containing ASCII text and spreadsheet files containing suggested formats for specimen, batch, and material information. The overall data submittal and review process is described in Section 1.5 and summarized in Figure 2.5.1.

Material property data sets submitted for possible publication are classified by one of the MIL-HDBK-17 data classes described below, and are examined to see that material and process (Section 2.5.2), sampling (Section 2.5.3), conditioning (Section 2.5.4), test methods (Section 2.5.5), and data documentation (Section 2.5.6) requirements are met for the properties discussed in Sections 2.5.7-2.5.11. B-basis values are presented in the handbook only for B and A data classes. (If sufficient data are available, an A class designation is used and both A- and B-values are presented). The MIL-HDBK-17 data classes are:

- A75 – Robust Sampling Data
  Statistically-based material properties that meet the most stringent handbook level of population sampling, data documentation and test method requirements. A- and B-values are presented in the handbook. The upper-case letter A is used for summary tables.

- A55 – Reduced Sampling Data
  Statistically-based material properties that meet the most stringent handbook level of data documentation and test method requirements with reduced sampling appropriate for certain applications (See Volume 3, Chapter 4, [Building Block Approach]). A- and B-values are presented in the handbook. The lower-case letter a is used for summary tables.

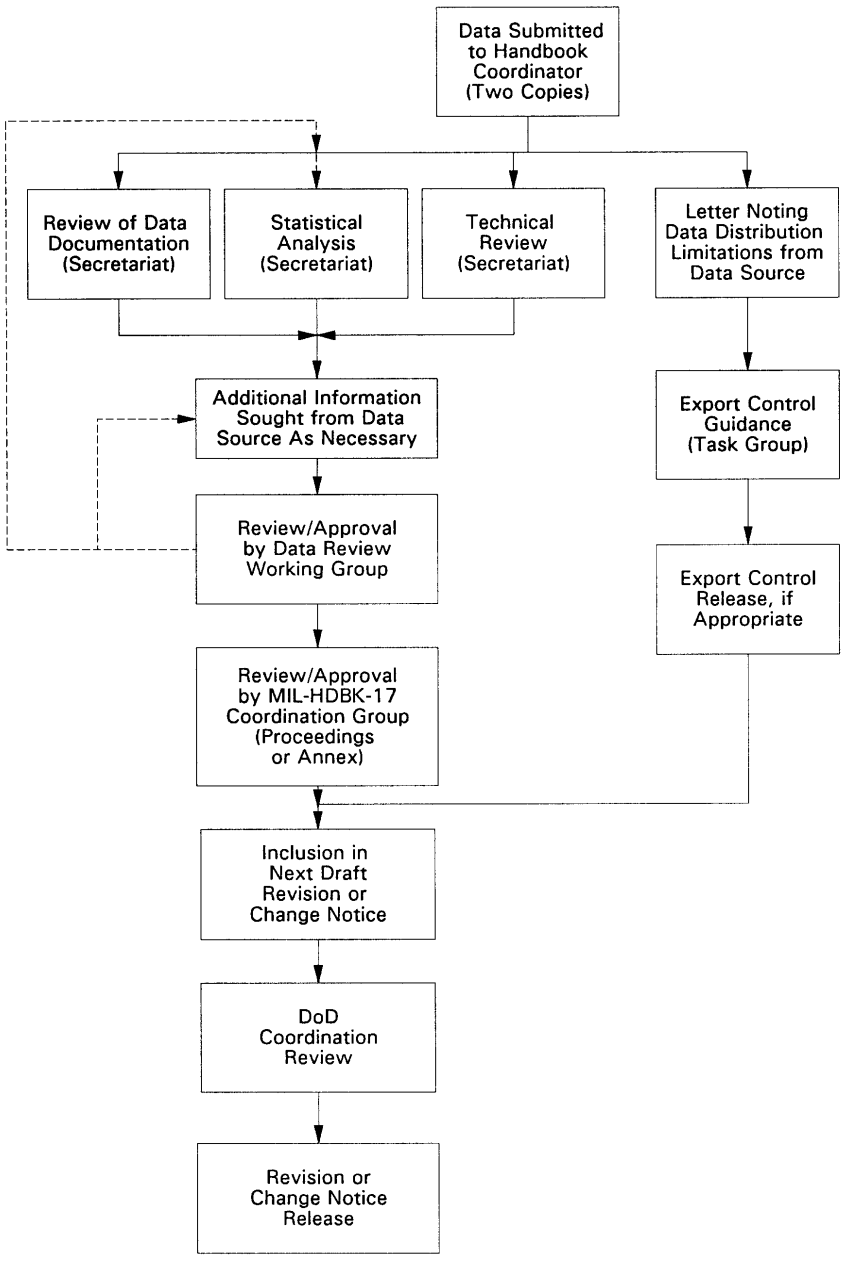


FIGURE 2.5.1 *Data submittal and review process.*

- B30 – Robust Sampling Data[1]
  Statistically-based material properties that meet the most stringent handbook level of population sampling for B-values, data documentation and test method requirements. B-values are presented in the handbook. The upper-case letter B is used for summary tables.

[1] The B30 data class corresponds to the Fully Approved class in MIL-HDBK-17 versions B through E.

- B18 – Reduced Sampling Data
  Statistically-based material properties that meet the most stringent handbook level of data documentation and test method requirements with reduced sampling appropriate for B-values for certain applications (See Volume 3, Chapter 4 (Building Block Approach)). B-values are presented in the handbook. The lower-case letter b is used for summary tables.

- M – Mean Data
  Mean material properties that meet the most stringent handbook level of data documentation and test method requirements. This data class generally applies to modulus and Poisson's ratio data and other properties for which basis values are not typically used. The upper case letter M is used for summary tables.

- I – Interim Data
  Data that do not meet the specific sampling or data documentation requirements required of B and A data classes. Interim data can be subdivided into two categories:
  1. Data that meet data documentation requirements for B and A data classes, but for which insufficient batches or replicates were tested. These data may potentially be pooled with other data to create a properly-sampled population that meets the B and A data requirements.
  2. Data which fail to meet the data documentation requirements for B and A data, even if the population sampling is adequate for those data classes. Such data cannot be used for subsequent pooling.

- S – Screening Data
  Data representing fewer than three batches, or data resulting from a test method limited to the screening level of approval. The screening data class is intended to provide for rapid inclusion in the handbook of data for new materials and other information that is useful even with a limited data set as described in Section 2.1.2.2 and as illustrated by the recommended test matrix of Table 2.3.1.1.

Note that, for uses other than inclusion in MIL-HDBK-17, selection of a material data class for use in an application is subject to agreement between the contractor and the certifying agency.

2.5.2 Material and process specification requirements

All materials submitted to the handbook should be manufactured in accordance with a material specification that imposes requirements on key physical and mechanical properties and should be processed in accordance with a process specification that adequately controls key processing parameters.

2.5.3 Sampling requirements

As noted in Section 2.2.5.1, the magnitude of a basis value is a function of the amount of data obtained, the number of batches represented, and the uniformity of the batches produced. Basis values are presented in the handbook only for B and A data classes. The minimum sampling requirements for each class are shown in Table 2.5.3.

The essence of documentation requirements is complete traceability and control of the database development process from material production, through procurement, fabrication, machining, environmental conditioning, gaging, testing, data acquisition, data normalization, and final statistical interpretation. The key items of information from this process for lamina/laminate mechanical testing are summarized in Table 2.5.6 and should be documented as part of any such material property determination. The items marked (•) should be included with any data submitted to the Secretariat. The items marked (⊗), as well as all items marked (•), must be included in the submission in order for submitted data to qualify for B, A, and M data classes. All other information should be traceable and available to the Secretariat for validation of statistical outliers. This list is based on the information necessary for lamina/laminate level me-

chanical property testing. Individual documentation items or documentation groups are not required where they are not applicable[1].

TABLE 2.5.3. *Minimum sampling requirements for MIL-HDBK-17 data classes.*

			Minimum Requirements	
Designation	Symbol	Description	Number of Batches	Number of Specimens
A75	A	A-Basis – Robust Sampling	10	75
A55	a	A-Basis – Reduced Sampling	5	55
B30	B	B-Basis – Robust Sampling	5	30
B18	b	B-Basis – Reduced Sampling	3	18
M	M	Mean	3	18
I	I	Interim	3	15
S	S	Screening	1	5

2.5.3.1 Additional requirements for B and A data classes

The prepreg batches should be prepared by the material supplier using production facilities. The first prepreg batches, up to five, should each be made using distinct fiber and matrix constituent lots (not required for batch numbers greater than five). For each condition and property, batch replicates should be sampled from at least two different test panels covering at least two separate processing cycles. Test panels should be nondestructively evaluated using ultrasonic inspection or another suitable nondestructive inspection technique. Test specimens should not be extracted from panel areas having indications of questionable quality. A test plan (or report) should document laminate design, specimen sampling details, fabrication procedures (including material traceability information), inspection methods, specimen extraction methods, labeling schemes, and test methods.

2.5.3.2 Data pooling

The ability to pool multiple similar but not identical data sets is desirable in order to obtain sufficient data to calculate material property basis values. Data sets for pooling may be available for materials from different fabricators, different locations of a single fabricator, or slightly different processes from the same fabricator.

Decisions on suitability of pooling will be made by the MIL-HDBK-17 Data Review working group, which will examine all tested properties for batch-to-batch variability (Section 8.3.2.2). Advance approval of the MIL-HDBK-17 Data Review working group is recommended before starting a new testing program that relies on pooling. However, MIL-HDBK-17 Data Review approval of a specific pooling process will not guarantee that the material data sets will, when testing is completed, be found to be poolable. Preliminary investigations into poolability are recommended before committing significant resources to large-scale testing.

[1] For example, fastener type and torque-up conditions are applicable to the bolt-bearing test but not to a tension test. Consequently, the reporting of this information is required for the bearing test and is not required for the tension test.

MIL-HDBK-17 Data Review has already pre-approved a pooling process for the case when several different fabricators wish to jointly develop B-basis data for MIL-HDBK-17 submission. Standard material and process specifications must be used and available. Sampling requirements are a minimum of three fabricators, each producing panels from at least three different batches of material. The minimum of nine batches must be sampled from five distinct prepreg batches, as discussed in Section 2.5.3.3. The batch replicate, processing, inspection, planning, and reporting requirements of Section 2.5.3.3 also hold.

2.5.4 Conditioning requirements

This section is reserved for future use.

2.5.5 Test method requirements

Specific test method criteria apply when submitting data to MIL-HDBK-17 for consideration for inclusion in Volume 2 of the Handbook, based on the following concepts. Ideally, a test method should have undergone a rigorous review of its applicability, precision and bias by an independent voluntary consensus standards organization that may include representatives from material suppliers, end-users, academia, or government. This review, and the test method, should be available in a referenceable, open-literature publication, and include interlaboratory (round robin) testing. Many times test methods meeting the above criteria are not available, and methods which meet less rigorous criteria (2 or 3 below) must be selected for data submittal.

The MIL-HDBK-17 Coordination Group has identified specific test methods, based on the material's structural complexity level (Section 2.1.2.1) and property, to be used when submitting data for consideration for inclusion in Volume 2 of the Handbook. These methods are designated or described in Chapters 3 through 7, and meet one or more of the following criteria:

1. Methods, applicable to advanced composites and in common use, which have completed the following:
   - Round robin testing under sponsorship of a recognized standards-making organization
   - Rigorous review of precision and bias
   - Publication in the open literature of a recognized standards-making organization

2. "Common practice" methods, which have not been standardized as in (1) above, but which are in common usage in the composite materials industry, are available in referenceable, open-literature publications, and have begun the process toward formal standardization.

3. Where no standards meeting the above criteria exist for specific structures or process/product forms, other test methods may have been selected by consensus of the MIL-HDBK-17 Coordination Group. Such methods may have been developed within the MIL-HDBK-17 Working Groups, or by other organizations, and will have begun the process toward formal standardization.

The test methods used for data submittal to the handbook must meet the handbook recommendations, summarized in Table 2.2.4, at the time the tests were performed. Fully Approved test methods are required for B and A data classes. Interim test methods are acceptable for I class data and Screening test methods are acceptable for S class data.

2.5.6 Data documentation requirements

This section outlines data documentation requirements necessary for the inclusion of data in MIL-HDBK-17 Volume 2. Data must meet the data documentation requirements that are in effect on the date of submission to the handbook. The data documentation requirements in effect at the time of publication of the handbook are provided in Table 2.5.6. Note that these requirements are subject to subsequent modification and that the latest authoritative data documentation requirements, which may differ slightly from Table 2.5.6, must be obtained from either the Secretariat or the Coordinator.

TABLE 2.5.6 *Documentation requirements*

Material identification - required for all composite materials
● material identification ● material class (e.g., C/EP) ▼ material procurement specification
Matrix material - required for all composite materials
● commercial designation ● manufacturer ● date of manufacture, earliest and latest ● lot number for each lot ● nominal density and test method
Reinforcement - required for all composite materials
● precursor type (i.e., PAN, Rayon) ● commercial designation ● manufacturer ● date of manufacture, minimum and maximum ● lot number for each lot ● surface treatment (Y/N) ▼ surface treatment type ● surface finish (sizing) identification and amount ● density (average per lot) and test method ● nominal filament count ● twist
Preform
● preform architecture ● preform identifier ● preform manufacturer ● preform method of manufacture - molded, stitched, RFI, etc. ● number of preform layers 2-D Fabric ▼ fabric manufacturer/weaver ● fabric family (weave pattern) ● fabric standard style number (particularly for glass fabrics) ● fabric sizing identification ● fabric sizing content ● fabric warp and fill tow count per inch ● fiber areal weight per batch[1] ● fabric fill fiber (if different) 3-D Woven Materials (including triaxial fabric) ● interlock description ● warp fiber filament count ● weft fiber filament count ● angle fiber filament count ● weaver yarn filament count ● percentage of warp yarn ● percentage of weft yarn ● angle of angle yarn (positive with respect to axial yarn) ● percentage of angle yarn ● percentage of weaver yarn

[1] See Part Description, fiber areal weight

● percentage of through-thickness yarn ● pitch length ● warp end count ● weft end count Stitching Information ● stitch type ● stitch thread ● stitch axial pitch ● stitch row spacing ▼ stitch denier ● stitch filament count ● bias yarn end count ● bias yarn angle Braiding Information ● braid description ● axial fiber type ● braid fiber type ● axial fiber filament count ● braid fiber filament count ● braid angle ● percentage of axial yarn ● percentage of braid yarn ▼ axial yarn spacing in braids Winding Description ● winding description
Prepreg
● ply manufacturer ● date of manufacture ● material lot number ● commercial designation ● material form - tape/fabric ● fiber areal weight per batch[1] ● total resin content per lot ▼ volatile content ● scrim material class ● scrim fabric style
Processing - required for all composite materials
▼ process specification ● lay-up schematic (including bagging, scrim, bleeder, etc.) ⊗ part manufacturer ● date of manufacture (date completed) ● reinforcement application process (how the fiber/preform was put together) - see Volume 2, Table 1.4.2(b) ● cure process type (how the part was cured/molded) - see Volume 2, Table 1.4.2(b) ▼ tackifier common name ▼ tackifier material class (e.g., epoxy) ▼ tackifier form - aerosol/liquid ▼ tackifier manufacturer

[1] See Part Description, fiber areal weight

Process Description - appropriate group required for all composite materials
Autoclave/oven/press cure
● near-net or extra resin process
● temperature for putting uncured part into autoclave/oven/press (including range)
● ramp rate to cure conditions
● cure conditions - temperature, pressure, duration,
● ramp rate to postcure
● postcure conditions - temperature, pressure, duration,
● cooling rate
● part removal temperature
● other critical control parameters
RTM (not applicable to RFI)
● degas steps on the resin prior to injection
● initial tool temperature
● preform insertion temperature
● heat-up rate, soak time and temperature before injection
● vacuum used (Y/N) and inches Hg
● injection rate (cm^3/min), temperature, and pressure
● cure temperature, pressure, and duration
● cooling rate and part removal temperature
● additional postcure (Y/N) - temperature, duration, in-tool/free-standing
Part Description- required for all composite materials
● form (panel, tube, etc.)
● ply count
● lay-up code
● fiber areal weight[1], nominal, by batch or part, and test method
● nominal fiber volume[1] and test method
▼ resin content (weight or volume), nominal and test method
⊗ void content, nominal, by batch or part, and test method
● density, nominal, by batch or part, and test method
● ply thickness, nominal, by batch or part, and test method
● glass transition temperature (wet and dry, nominal) and test method
Specimen preparation- required for all composite materials
● specimen orientation
⊗ tab adhesive curing temperature (nominal)
Mechanical testing- required for mechanical testing of all composite materials
● number of specimens
● test procedure (citing *all* deviations from standard procedures including reporting requirements. It is assumed that, other than the deviations reported, the test method was followed.)
● date of applicable standard
● date of testing
● specimen thickness for each specimen
● specimen conditioning standard method
● conditioning temperature[2]
● conditioning humidity
● conditioning time
● conditioning environment (if not lab air), standard designation of fluids if available

[1] Fiber volume or fiber areal weight (FAW) for each batch or panel is required. For prepregs, batch or roll average FAW is acceptable. For other materials, lot or roll average FAW of the assembled reinforcement (fabric, braid, or preform) is acceptable. If additional out-of-plane reinforcement, such as stitching is used, the lot or roll average FAW can be obtained for the reinforcement assembly prior to the out-of-plane reinforcement (e.g., unstitched fabric).
[2] If multi-step conditioning method was used, provide conditioning information for each step.

●	equilibrium (Y/N)
⊗	moisture content, specify whether moisture content or uptake
●	test temperature
●	soak time at test conditions prior to load initiation
●	fastener type and torque-up conditions (bearing, mechanically fastened joint (MFJ), filled hole)
●	hole diameter (open/filled hole, bearing, MFJ)
▼	hole clearance, countersink angle and depth (filled hole, bearing, MFJ)
●	nominal thickness, width, and material for each member (bearing, MFJ)
●	edge distance (bearing, MFJ)
●	fixture torque-up (e.g., SACMA RM-1)
●	shear strain at which test was truncated (shear)
●	failure mode identification and location
●	all non-normalized (raw) data
●	method of calculating modulus and Poisson's ratio
●	method of finding offset strength (bearing)
●	method of finding proportional limit (bearing
●	method of calculating fracture toughness (fracture toughness)
●	method of finding proportional limit (bearing)
●	method of calculating fracture toughness (fracture toughness)

● Required for submission to Secretariat
⊗ Required submission to the Secretariat for B and A data classes
▼ Requested for submission to the Secretariat, presented if available

General recommendations on data documentation are provided in Section 2.2.12. The essence of documentation requirements is complete traceability and control of the database development process from material production, through procurement, fabrication, machining, environmental conditioning, gaging, testing, data acquisition, data normalization, and final statistical interpretation. The key items of information for mechanical testing of composite materials are summarized in Table 2.5.6 and should be documented as part of any such material property determination. The items marked (●) should be included with any data submitted to the Secretariat. The items marked (⊗), as well as all items marked (●), must be included in the submission in order for submitted data to qualify for B and A data classes. Items marked (▼) are requested for submission to the Secretariat; this information will be presented in Volume 2 if available. Recommendations for in-house documentation are discussed in Volume 1, Section 2.2.12. All reasonable information should be traceable and available to the Secretariat for validation of statistical outliers.

This list is based on the information necessary for mechanical property testing. The documentation requirements are grouped by possible forms at various stages of fabrication. Groups required for all composite materials are identified. Material Identification, Matrix Material, and Reinforcement groups are required for all types of materials. The items in the Preform and Prepreg groups are required based on additional steps in fabricating the material as indicated for each fabrication form. For example, a (2-D) fabric prepreg would require the 2-D Fabric portion of the Preform group and the Prepreg group. The appropriate section within Process Description should be used. The remaining groups apply to all mechanical property testing. Note that items are grouped by whether or not they are needed to include relevant information for a particular stage of fabrication. For most items, this also is the fabrication form from which the information is obtained. There are a few exceptions to the latter grouping. For example, information on scrim is included with Prepreg since that is the form which requires scrim information. Fiber areal weight should be measured at the most appropriate stage of fabrication. Individual documentation items are not required where they are not applicable[1].

[1] For example, fastener type and torque-up conditions are applicable to the bolt-bearing test but not to a tension test. Consequently, the reporting of this information is required for the bearing test and is not required for the tension test.

The information required for other types of tests or material levels is similar. For instance, prepreg property testing would require the prepreg, reinforcement, matrix, and possibly fabric information, as well as appropriate information on specimen preparation and testing procedures.

2.5.7 Data normalization

Certain types of data should be normalized to provide consistent presentation of properties and to allow for reasonable material comparison. For mechanical properties, data are normalized by the Secretariat for lamina/laminate strength and stiffness properties *except* 90° (transverse) tension and compression of unidirectional laminates, interlaminar (3- or z-direction) tension, interlaminar compression, interlaminar shear, in-plane shear, short beam strength, bearing and bearing/bypass, strain energy release rate, and Poisson's ratio. The procedures from Section 2.4.3 should be used for normalization of handbook mechanical data, in the following order of preference:

1. By fiber volume as measured on the test specimen as shown in Equation 2.4.3.2(a),
2. By specimen cured ply thickness and batch average fiber areal weight as shown in Equation 2.4.3.2(k)
3. By specimen cured ply thickness and batch average fiber volume as shown in Equation 2.4.3.2(n)

Data for unidirectional tape are normalized to 60% fiber volume and data for fabric are normalized to 57% fiber volume unless another value is considered more appropriate by the Data Review working group. Normalization procedures for other properties have not yet been approved.

2.5.8 Statistical analysis

All data for the handbook are analyzed according to the flowchart in Section 8.3.1. Where batch-to-batch variability can be neglected (based on Section 8.3.2) the data model used is the first data model with an observed significance level greater than 0.05. Models are considered in the following order - Weibull, normal, lognormal, and nonparametric. Selection of statistical approach including consideration of pooling (Section 2.5.3) is subject to review and approval by the Data Review working group.

2.5.9 Mechanical properties of laminae and laminates

Handbook values for mechanical properties of each material will be listed in the data summary in Volume 2.

2.5.9.1 Unidirectional properties from laminates

A laminate "backing-out" approach for unidirectional material lamina mechanical properties is documented in Section 2.4.2. Data by this approach will be considered for inclusion in the handbook according to the procedures in Figure 2.5.1. While the Section 2.4.2 approach is applicable to many lay-ups and other possibilities continue to be explored, to date only [90/0]ns laminates have been considered acceptable by the MIL-HDBK-17 Coordination Group.

2.5.9.2 Strength and strain-to-failure

Handbook values for strength, and strain-to-failure should meet the sampling requirements in Section 2.5.3 for each property and at each condition. For the data to be included in the population, failure modes must be considered acceptable in accordance with the test method used. Strengths will be normalized according to Sections 2.4.3 and 2.5.7. Strengths and strains-to-failure will receive the full statistical treatment described in Section 8.3.1 including outlier detection, data pooling testing, determination of distribution, and B-basis value calculation.

2.5.9.3 Elastic moduli, Poisson's ratios, and stress/strain curves

Handbook values for elastic moduli (Young's moduli or shear moduli) and Poisson's ratios, calculated over a fixed strain range, should meet the sampling requirements in Section 2.5.3 for each property and at each condition. The elastic moduli should be normalized according to Sections 2.4.3 and 2.5.7 and all results receive the statistical analysis outlined in Section 8.3.1. Minimum, average, maximum, and coefficient of variation (CV) values will be tabulated for moduli, and the average value tabulated for Poisson's ratio. The report should include the calculation method and strain ranges for each property. If stress/strain data are provided an average stress/strain curve will be calculated using the procedures described in Section 8.4.4 and reported as shown in Volume 2, Section 1.4.2.

2.5.10 Chemical properties

This section is reserved for future use.

2.5.11 Physical properties of laminae and laminates

Handbook values for physical properties (at 73±5°F (23±3°C), if available) will be listed in the data summary for each material. Additional values as a function of temperature or other parameters, if available, will be presented graphically.

2.5.11.1 Density

The handbook value for density should be determined at a specified temperature (in the absence of a specific requirement use 73±5°F (23±3°C)) from the average of a minimum of three specimens for each batch used in the determination of any mechanical properties.

2.5.11.2 Composition

This section is reserved for future use.

2.5.11.3 Equilibrium moisture content

Handbook values for equilibrium moisture content should be determined for specified relative humidity and temperature values (in the absence of a specific requirement, use 85%RH, 180°F (82°C)) from the average of a minimum of three specimens at each condition. If additional information is available for equilibrium moisture content as a function of temperature and relative humidity, those values will be presented graphically.

2.5.11.4 Moisture diffusivity

Handbook values for moisture diffusivity should be determined for specified temperatures (in the absence of a specific requirement use 180°F (82°C)) from the average of a minimum of three specimens at each temperature. If additional information is available for moisture diffusivity as a function of temperature and relative humidity, those values will be presented graphically.

2.5.11.5 Coefficient of moisture expansion

Handbook values for moisture expansion coefficient should be obtained and will be reported in the same way as those for thermal expansion coefficient (Section 2.5.12.1).

2.5.11.6 Glass transition temperature

Handbook values for glass transition temperature should be determined for dry and wet material conditions from the average of a minimum of three specimens at each condition. Guidelines for glass transition temperature testing and maintenance of a wet condition are discussed in Section 6.4.3.

2.5.12 Thermal properties

Thermal property room temperature values will be listed in the data summary. Additional values as a function of temperature, if available, will be presented graphically in a single figure according to Volume 2, Section 1.4.3. Each property should be determined for a specified temperature or temperature range. Default values, to be used when temperatures are not otherwise specified, are provided for different matrix materials in Table 2.5.12. The room temperature default value for all materials is 73°F (23°C). The tolerance on all default temperatures is ±5F° (±3C°).

TABLE 2.5.12 *Default temperatures for handbook thermophysical data.*

Matrix Material Family	Default Elevated Temperature		Default Temperature Range	
	°F	°C	°F	°C
Epoxy	220	104	73 - 275	23 -135
Bismaleimide	350	177	73 - 450	23 - 232
PEEK	220	104	73 - 250	23 - 121
Polyimide	550	288	73 - 600	23 - 315

2.5.12.1 Coefficient of thermal expansion

Handbook values for average coefficient of linear thermal expansion (CTE) should be determined for specified temperature ranges (in the absence of a specific requirement, use the default temperature range for the appropriate matrix material family in Table 2.5.12) from the average of a minimum of five specimens for each temperature range. The reference temperature for thermal expansion should be clearly noted.

2.5.12.2 Specific heat

Handbook values for constant pressure specific heat should be determined at specified temperatures (in the absence of a specific requirement use the room temperature default) from the average of a minimum of three specimens for each temperature.

2.5.12.3 Thermal conductivity

Handbook values for average thermal conductivity should be determined for specified temperature ranges (in the absence of a specific requirement, use the default temperature range for the appropriate matrix material family in Table 2.5.12) from a minimum of three specimens for each temperature range.

2.5.12.4 Thermal diffusivity

Handbook values for thermal diffusivity should be determined for specified temperatures (in the absence of a specific requirement, use the default elevated temperature for the appropriate matrix material family in Table 2.5.12 as the median temperature) from the average of a minimum of three specimens for each temperature.

2.5.13 Electrical properties

This section is reserved for future use.

2.5.14 Fatigue

Fatigue is defined as the change in property as a result of repeated mechanical loading in the appropriate environmental conditions. This is not normally considered a design limiting property for PMC structures that have been designed with fiber dominated lay-ups and have minimal out-of-plane loading conditions. The exceptions to this generalization are high cycle fatigue components like rotor blades, propeller blades, and engine fan blades. These structures encounter a high number of cyclic loads (up to 5x10^8) and should not be sized for static and damage considerations alone. In these applications, fatigue is an important property. The "building block" design/validation approach using coupon, element, and component testing can be used to assure adequate fatigue life.

Fatigue should be addressed at various levels of development:

- Basic material property screening- Primary purpose is to compare various materials to aid in the selection process.
- Design allowables-Purpose is to characterize the specific material selected with sufficient replicate testing and conditions to ensure adequate performance of the completed design by analysis. An additional use of this level of testing is to define fatigue enhancement factors for conducting higher level (more complex) element and component tests.

There is no established practice of using laminae fatigue data to construct laminate fatigue allowables, so it is recommended that application specific laminate based allowables be generated. ASTM D3479, "Tension-Tension Fatigue Oriented Fiber, Resin Matrix Composites" is a generalized coupon testing method that can be used as a guide for a fatigue test method. Note, this published test method approach is for tension-tension fatigue. Compression-compression and tension-compression fatigue are typically more critical in composites.

The following are some general guidelines for specimen fatigue testing. Fatigue data is generated at the design critical test conditions, i.e. -65F, room temperature, or hot/wet. Design critical test conditions are very application dependent. For hot/wet testing, specimens should be brought to their desired moisture level prior to testing, and maintained at this moisture level during the actual fatigue test. The desired moisture level is again application dependent. It may be an equilibrium moisture level for a specified relative humidity, a percentage of an equilibrium level, or some other condition defined by the specific application environment, part geometry, and projected service life. As an example, Reference 2.5.14(a) (J. Rouchon) contains the methodology used by one certifying agency.

Like the generation of static design allowables, notches can be used for fatigue testing as well. This approach could be non-conservative, in that a notched specimen has a greatly reduced volume of material at the maximum stress cross-section when compared to an unnotched specimen. If the purpose of the specimen test is to account for material and process variation effects on fatigue performance, the unnotched specimen is a more appropriate configuration. Unnotched specimen failures will initiate at random failure sites that are inherent in the specimen, whereas notched specimen failures are constrained to initiate at the manufactured notch.

If the purpose of the fatigue testing is to provide a structural level assessment of process variation, the use of actual manufacturing anomalies may be more appropriate. Composite structure based manufacturing anomalies, such as delaminations, disbonds, fiber breaks, are important in a cyclic load environment because it must be shown they will not grow significantly in the no growth damage tolerance approach or they will not grow to a critical length during the life of the component or before they are detected by inspection in the growth approach. Element, component, and sub-component level fatigue tests are usually recommended to substantiate allowable manufacturing anomaly limits and also to establish in-service inspection requirements.

Fatigue data for in-plane material property performance can be evaluated with three R-ratios: R=0.1 (tension-tension), R=10.0 (compression-compression), and R=-1.0 (tension-compression). Design fatigue loading conditions may not dictate the full spectrum of loading conditions, but with these three R-ratios a Goodman diagram (Reference 2.5.14(b)) can be constructed to aid in predicting fatigue at any given R-ratio. For interlaminar properties, it is recommended two R-ratios be run, R=0.1 and R=-1 .0. R=10.0 is not necessary for flatwise (through the thickness) fatigue, since this is not typically a viable failure mode. The loading conditions are the same for R=10.0 and R=0.1 for interlaminar shear fatigue, so R=10.0 tests are not performed.

Tests are typically run in load control. Strain control tests are not generally necessary because of PMC's linear elastic behavior. Unless test frequencies are set to be the same as in-service frequencies, test frequencies should be set so that any temperature rise in the specimen is limited to 5F. Generally, test frequencies of 5-10 hertz are used. All specimens should be thermo-coupled to ensure specimen heating is insignificant during all fatigue testing. The use of in-service frequencies during testing can be important, since it has been shown that increased test frequencies can increase fatigue life in some materials/test configurations.

Failure mode evaluation of fatigue test specimens are just as important as for static test specimens. Erroneous data can be generated if an improper failure mode occurs. The typical undesirable failure modes to avoid include, tab debonding, non-gauge section failures, and fiber splitting in geometry transition areas. Fatigue specimen design and fabrication quality are very significant in achieving the proper gauge section failure modes in specimen fatigue tests.

Stress levels selected for testing should provide good data spacing on a semi-log fatigue stress vs. cycles plot (S/N plot), and provide failure points in the cycle regime of interest. An example of a cycle range for a low cycle fatigue (LCF) 40,000 cycle structure, would be to generate failure points from 10^3 to 10^5 cycles. The number of data points required per curve varies depending on its desired use. For materials screening, 8 data points are usually sufficient to establish the fatigue performance. For design allowables, 15 data points are recommended over three batches. Regression analysis or Sendeckyj's method (References 2.5.14(c) and (d)) are efficient approaches for analyzing this data and establishing B-basis and A-basis design allowable lines.

The fatigue design allowable curves can be used for two purposes. The first is to establish maximum stress values for a specific number of cycles by using a B-basis or A-basis value. The other purpose is to use the data distribution from the fatigue data of the critical design property to establish a load/life enhancement magnification factor used for element or component testing. Since only a small number of elements or components are usually fatigue tested, the load/life of the test must be enhanced to account for material and process scatter. Even though there are empirically based techniques to predict the life of PMC structures, the element or component fatigue test is required for verification because of feature dependent effects like ply drops and manufacturing complexities not accounted for in the specimen testing or the design analysis. The load/life enhancement approach is outlined in the FM report (Reference 2.5.14(e)) and provides a sound statistical approach to ensure application durability with a limited number of test articles. Load/life enhancement approach may also be used to account for environmental effects that are difficult to reproduce in element or component tests.

As discussed previously, most fatigue concerns in composites apply to high cycle applications. Most aircraft structure is subjected to a range of loads with various amplitudes and maxima (spectra loading). These structures are not characteristically high cycle. The spectrum loading introduces another complexity that has proved difficult to account for in life prediction. Cumulative damage approaches developed for metallic structure have been shown to be ineffective in composites. This has necessitated an increased reliance on empirical procedures for life verification rather than life prediction.

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