CHAPTER 6 LAMINA, LAMINATE, AND SPECIAL FORM CHARACTERIZATION

6.1 INTRODUCTION

The use of composite materials continues to increase as new performance, reliability, and durability requirements drive hardware designs to higher levels of structural efficiency. Additionally, government requirements are becoming more stringent to ensure proper levels of structural integrity are maintained. These design drivers, among others, have resulted in a growing recognition that certification or qualification of aerospace structure requires an extensive combination of analysis, testing, and documentation.

Further, because of the large number of design variables inherent to composite structure, analytic models are even more necessary than for metallic structure to ensure completeness of the hardware qualification process. Inherent in all structural analysis models are material, physical, and mechanical property characterization data. Ideally, these analytic models would permit analysts to predict full-scale structural response (e.g. stability, deflections, strength, life) directly from a generic (lamina) material database. In truth, test data is required at design development (element, subcomponent, component) and full-scale article test levels as well as the generic (coupon) levels of evaluation.

The purpose of Chapter 6 is to provide guidelines of testing procedures for characterization of physical and mechanical lamina (ply) and laminate properties.

A laminate is a product made by bonding together two or more layers of material or materials, and a lamina is a single ply or layer in a laminate. The material forming each layer typically consists of a carbon, glass, or organic (polymeric) fiber reinforcement embedded in a thermoplastic or thermosetting resin matrix. While retaining their identities in the composite, the constituents combine to provide specific characteristics and properties.

Many techniques are used to characterize the chemical, physical, and mechanical properties of composite materials. The purpose of this chapter is to provide information on techniques that may be used to analyze and evaluate these properties. The test methods discussed in each section may not be appropriate for all types of composite materials. Currently, more studies are being conducted to investigate how variations in reinforcement and resin chemistry and morphology may affect the physical properties and long term performance of composites. Where possible, the limitations of existing test methods are discussed.

6.2 SPECIMEN PREPARATION

6.2.1 Introduction

This section provides general recommendations for the fabrication and preparation of the test specimens detailed in this document. These recommendations cover specimen traceability, test article[1] fabrication, specimen location, configuration, and machining.

The validity of material properties used in design of structure is dependent on the quality of the specimens being tested. If the objective of the testing is to provide comparative information of different materials, it is crucial that variability due to specimen preparation be kept to a minimum. If the data being generated are intended to be used to generate allowables, the goal is to reflect the interaction of the base material and processing which is expected to occur in production. In either case care must be taken in the specimen preparation process to minimize the variation which naturally occurs during the process. Specimen fabrication should be performed in compliance to ASTM D 5687 (Standard Guide for Preparation of Flat Composite Panels with Processing Guidelines for Specimen Preparation). Even test articles that are not flat can benefit from the ASTM guide.

[1] A test article is any construction from which individual specimens are extracted. Such a test article may be a flat panel fabricated specifically to develop material properties, or it may be a production part set aside for test purposes.

6.2.2 Traceability

All specimens should be traceable to the material batch, lot, roll, process and test article. The requesting organization may choose to require traceability of each specimen to its location within the test article.

The specification, or purchasing paperwork, should require batch, lot, roll traceability and lot acceptance test information. It is recommended that when uncured material is purchased it be required that all available traceability information, including vendor certifications and material receiving inspection data of acceptance test results, be delivered with the material. The organization conducting the investigation should review the information to ensure there is enough traceability information to proceed with test article and specimen fabrication.

All prepreg material that is stored before fabrication should have a storage history record. Information such as accumulated time in and out of refrigeration should be recorded.

For the test article, the prepreg batch number, lot number, roll number, and processing information should be recorded. Another piece of information which needs to be maintained throughout specimen fabrication is ply orientation. One method by which this may be accomplished is through the use of a witness line, as discussed in the next section.

6.2.3 Test article fabrication

The following is a list of important items that should be considered when fabricating test articles:

a. Test articles should be built according to engineering drawing requirements or sketches. The drawing requirements or sketches should specify: ply materials, test article reference orientation, ply orientation, material and process specifications or equivalent process document, and inspection requirements.

b. Vital material and process identification, such as prepreg batch number, lot number, roll number, autoclave run, press, or other consolidation method and lay-up stacking sequence, should be recorded. This information is stored to maintain traceability of the test articles. This same traceability should be maintained on any excess material left after the specimens have been removed.

c. The test article identification code and witness line should be permanently identified on each test article. A witness line should be established on the fabrication tool to act as a reference to the fiber orientation on the test article. For hand lay-up methods a witness line which will be maintained during the lay-up and curing process must be identified as the reference orientation. The angular tolerance between the plies put down and this line depends on the processing specification by which the material is being processed. In automated processes some other method of establishing the reference orientation must be established. Once established, the witness line should be transferred to the test article, and maintained throughout specimen extraction.

d. It is generally recommended that for cured test articles at least 1 in. (25 mm) of material be trimmed from the edges. One of the machined edges of the test article may be used to permanently maintain the reference orientation on the article.

e. The requesting organization (or if required, the appropriate quality assurance organization) should inspect test articles. This inspection should be done before the specimens are fabricated to ensure they meet all requirements in the controlling process specification or appropriate equivalent document. If the test article does not meet all requirements, the requesting organization and, when applicable, the customer representative, should provide the final disposition of the test article.

6.2.4 Specimen fabrication

The following is a list of important items that should be considered when fabricating specimens.

a. Specimens should be extracted from test articles in the region that meets all process, engineering drawing, and specimen drawing requirements.

b. Specimens should be located on the test article according to the cutting diagram provided by the requesting organization. If a test article does not pass the inspection criteria, the requesting organization may choose to cut specimens relative to identified test article defects to make the effect of the defects on the specimen response representative of the full-scale item.

NOTE: When defining specimen locations, allow for material removed in the cutting operation.

c. A specimen identification code should be defined in the test plan, referenced in the test instructions, and recorded in the data sheets. The specimen identification code should be permanently marked on each specimen. Care should be taken to keep the code outside the failure area of the specimen.

d. For specimens too small to mark the complete code, mark only the unique serial number on the specimen. It is recommended that care be taken to place small specimens in bags properly labeled with that specimen's full identification.

e. If it is required that the location of the specimen on the test article be known, specimens should be labeled before being extracted. This labeling should allow all specimen and excess material locations to be known after cutting.

f. The reference edge of the specimen should be aligned within the specified orientation using the witness line. In instances where a smaller subtest article is machined and used to make several specimens at once, a reference line or edge should be transferred to this subtest article from the witness line. This transferred line should be orientated within ±0.25° with respect to the witness line.

g. Before cutting, the specimen location and orientation should be verified by the requesting organization or an independent reviewer.

h. Specimens should be extracted from the fabricated test articles according to the appropriate machining procedure as specified. Specimens may be machined with a variety of machining tools. In general the final cutting tool should have a fine grit, be hardened, and run at a high tool speed without wobble. The cut itself should be executed to minimize excess heating of the laminate.

i. The added cost and manufacturing associated with tabbed specimens should be considered when selecting specimen type. The limitations and problems associated with the tabbing of specimens is stated in each individual test method. If bonded tabs are required, the cure of the adhesive should be evaluated to determine if it is compatible with the composite system and tab material (if different). If the tab configuration produced in the bonding process is not within the geometry requirements of the specimen configuration, further machining of the tabs may be required.

j. Holes in specimens should be drilled in accordance to the applicable process specification.

k. Any fasteners that are required should be installed in accordance to the applicable process specification.

l. Completed specimens should be inspected prior to testing to ensure conformance with the standards being used. Variations in individual specimen thickness should be within the applicable test method tolerances. Larger variations may cause improper loading when used with close tolerance test fixtures. These variations may indicate that the specimen was fabricated improperly (e.g., ply drop-off or resin bleed).

6.3 CONDITIONING AND ENVIRONMENTAL EXPOSURE

6.3.1 Introduction

Conditioning is the process of exposure of material to a potentially property-altering environment prior to subsequent test.[1] This section focuses on conditioning of materials subjected to moisture exposure (immersion in all types of fluids, but especially humid air). There are, of course, many other types of conditioning environments. An incomplete list includes: subambient (moderately low temperatures), cryogenic (very low temperatures), elevated temperature (dry), oxidizing, low-Earth orbit simulation (including exposure to monatomic oxygen), and exposure to various types of radiation. Conditioning issues in these other environments will not be explicitly discussed in this section. A related, but much more difficult, extension of material conditioning is associated with the issue of *long-term aging* (for example, 10,000 to 80,000 or more hours of exposure), which for practical engineering purposes requires development of procedures for accelerated conditioning. While some very limited and restricted guidelines for acceleration of basic moisture conditioning are discussed in the following subsections, acceleration of long-term aging processes is a state-of-the-art topic that is beyond the scope of this section.

Most polymeric materials, whether unreinforced resin, polymeric composite matrix, or a polymer-based fiber, are capable of absorbing relatively small but potentially significant amounts of moisture from the surrounding environment.[2] The physical mechanism for moisture mass change, 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 is discussed in Section 6.4.8). Fickian moisture diffusion into or out of the interior occurs relatively slowly; many orders of magnitude slower than heat flow in thermal diffusion. Nevertheless, given enough exposure-time in a moist environment, a significant amount of moisture may be absorbed into the material. This absorbed moisture may cause material swelling, and, particularly at higher temperatures, may soften and weaken the matrix and matrix/fiber interface, which is deleterious to many mechanical properties that are often design drivers for structural applications. Absorbed moisture effectively lowers the maximum use temperature of the material (see Sections 2.2.7 and 2.2.8). The effect is demonstrated by a lowering of the glass transition temperature (thus the particular interest in T_g test results).

The two main types of basic moisture conditioning of materials are: *fixed-time conditioning*, where a material specimen is exposed to a conditioning environment for a specified period of time; and *equilibrium conditioning*, where a specimen is exposed until the material reaches equilibrium with the conditioning environment. While fixed-time conditioning is still in common use when screening materials, it usually results in a material condition that is substantially non-uniform through the thickness; subsequent test results are, therefore, considered only a qualitative assessment rather than a quantitative result. Except for certain screening-level purposes, or as part of application-specific structural-level tests, fixed-time conditioning as summarized in Section 6.3.2 is not considered sufficient or representative; only equilibrium conditioning as discussed in Section 6.3.3 provides a true assessment of comparable material response.

When absorbed moisture is a potential design concern, a material testing program should evaluate both the moisture absorption material properties (diffusion rate and equilibrium content) and the effect of absorbed moisture on key design properties after equilibrium moisture exposure. An ASTM moisture absorption conditioning/material property test method, ASTM D 5229/D 5229M (Reference 6.3.1), has been created to define the conditioning parameters and procedures needed to assure that uniform through-

[1]Nonambient testing is another subject, and, for mechanical testing, is covered in Section 6.5.3.
[2]While certain polymers, like polybutadiene, resist water vapor absorption to the point that humidity conditioning may not be required, these materials are still considered rare exceptions. On the other hand, most reinforcements, including those of 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 water vapor absorption is limited to the polymer matrix.

thickness equilibrium[1] is obtained during conditioning. ASTM D 5229/D 5229M also defines how to determine the moisture absorption properties, and its use for this purpose is discussed in more detail in Section 6.6.8.

6.3.2 Fixed-time conditioning

As stated above, fixed-time conditioning is only of limited usefulness[2], it cannot generally provide the desired uniform moisture condition through the thickness of the material. The shortcomings of the fixed-time approach are illustrated in Figure 6.3.2 for a simulated 30-day exposure of IM6/3501-6 carbon/epoxy at 140°F (60°C) and 95% RH. Using known values for moisture diffusivity and moisture equilibrium content, the calculated average moisture content of various laminate thicknesses is plotted and shown as a smooth curve. From this curve, it can be seen that the maximum laminate thickness that can reach equilibrium at this temperature during this fixed, though fairly lengthy, conditioning exposure, is 0.035 in. (0.89 mm). For greater thicknesses, the moisture distribution through the thickness will *not be uniform,* as the interior moisture levels will be below equilibrium moisture content. This is further illustrated by an example in Section 6.3.3.

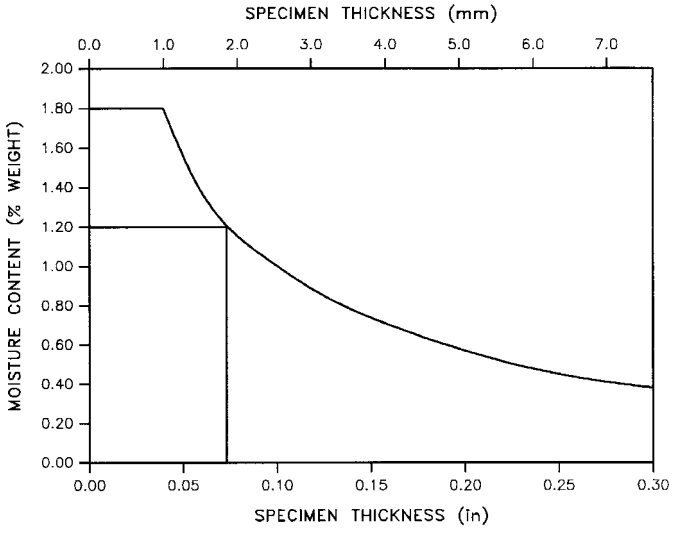


FIGURE 6.3.2 *Two-sided moisture absorption of carbon/epoxy laminate after 30 days exposure at 140°F (60°C)/95% RH.*

As will be discussed in Section 6.3.3.1, with lower target relative humidity levels, it is common to try to accelerate conditioning by subjecting the material to a higher relative humidity level for a shorter period of time. The objective is to introduce the same average moisture content in the material as would be seen in equilibrium conditioning at the lower relative humidity level, although the distribution of moisture content distribution will be less uniform through the thickness. Using a single-humidity level, fixed-time conditioning example, again illustrated by Figure 6.3.2, equilibrium at 78% RH (1.2% equilibrium moisture content for this material) can be approximated only at a thickness of 0.070 in. (1.8 mm). For a thickness greater than 0.070 in. (1.8 mm), the average moisture content will be insufficient, and for a thickness less than

[1]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.
[2]Examples of fixed-time conditioning methods that should specifically be avoided include: ASTM D 618 (Reference 6.3.2(a)), ASTM D 570 (Reference 6.3.2(b)), and SACMA RM 11-88 Method I (Reference 6.3.2(c)).

0.070 in. (1.8 mm), the moisture content will be higher than desired. Again, the fixed-time conditioning approach is inadequate.

As seen from the examples above, total moisture content resulting from fixed-time conditioning is thickness dependent. However, since fluids diffuse through different materials at different rates, fixed-time conditioning cannot produce a uniform material condition for all materials,[1] even if thickness is held constant. Therefore, test results based on fixed-time conditioning should not be used for design values, and generally should not even be used in qualitative comparisons between different materials. However, fixed-time conditioning can serve a purpose when combined with a flexure test (which is sensitive to surface exposure) for qualitative aerospace fluids assessment, as discussed in Section 2.3.1.3.

6.3.3 Equilibrium conditioning

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 (hereinafter assumed equivalent to equilibrium at the design service relative humidity). The preferred conditioning methodology uses ASTM D 5229/D 5229M (Reference 6.3.1), a test method that includes procedures for conditioning as well as for determining the two Fickian moisture material properties: moisture diffusivity and moisture equilibrium content (weight percent moisture).

ASTM D 5229/D 5229M is a gravimetric test method that exposes a specimen to a moisture environment and plots moisture mass gain versus the square-root of elapsed time. The early portion of the mass/square-root-time relationship is linear, the slope of which is related to the moisture diffusivity. As the moisture content of the material near the surface begins to approach equilibrium, the slope of this curve becomes increasingly smaller. Eventually, as the interior of the material approaches equilibrium, the difference between subsequent weighings will be very small and the slope will be nearly zero. At this point the material is said to be at equilibrium moisture content. This process is illustrated in Figures 6.3.3(a) and (b). Figure 6.3.3(a) shows the total mass gain versus square-root-time during specimen moisture exposure; the different curves illustrate the difference in response due to different temperatures. For the 150°F condition (the diamonds in Figure 6.3.3(a)), Figure 6.3.3(b) shows the moisture profile through the thickness of the specimen for several early time periods, illustrating the rapid moisture uptake near the surface together with the relatively slow update of moisture in the middle of the specimen.

A similar, but more limited and not fully equivalent, procedure for conditioning and equilibrium moisture content (but not diffusivity) is documented by SACMA RM 11R-94 (Reference 6.3.3(b)), which first brings three specimens to moisture equilibrium at 85% RH.[2] The actual SACMA conditioning process on test specimens is then subsequently conducted, and terminated when the weight gain of the conditioned specimens reaches 90% of the moisture equilibrium content, resulting in a lower moisture content in the test specimen as compared to that resulting from ASTM D 5229/D 5229M. As an example, a 0.1 in. (2.5 mm) thick laminate with a diffusivity of 1.6E-09 in^2/s (1.0E-06 mm^2/s) and a true (very long-term) equilibrium moisture content of 1.50%, when evaluated by the two approaches, would reach effective equilibrium at 1.45% in 24 days (ASTM), or at 1.43% in 21 days (SACMA). In subsequent conditioning, the ASTM procedure would reproduce the same 1.45% moisture content in 24 days, while the SACMA conditioning procedure would produce a moisture content of 1.29% (0.9 x 1.43) in 13 days.

[1]Including a specific material system produced at different resin contents.

[2]While the 1988 version of SACMA RM 11 used a different definition of equilibrium, the 1994 edition adopted the ASTM definition, with one difference: the reference time period (minimum weighing time interval for equilibrium) was fixed at 24 hours. For sufficiently high diffusion rates there is no difference. For example, for the SACMA RM 11R-94 preferred thickness of 0.040 in. (1 mm), the two definitions begin to deviate when the moisture diffusivity is slower (smaller in value) than 3.6E-10 in^2/s (2.3E-07 mm^2/s). As the rate of diffusion slows below 3.6E-10 in^2/s (2.3E-07 mm^2/s), the SACMA calculated equilibrium moisture content will begin to deviate from the ASTM value. This diffusivity crossover point is a function of thickness; for the maximum SACMA thickness of 0.080 in. (2 mm), the crossover point increases to a diffusivity of 1.4E-09 in^2/s (9.3E-7 mm^2/s). When determining the moisture equilibrium content of low diffusivity materials, the ASTM definition, which is sensitive to both diffusion rate and coupon thickness, should be used.

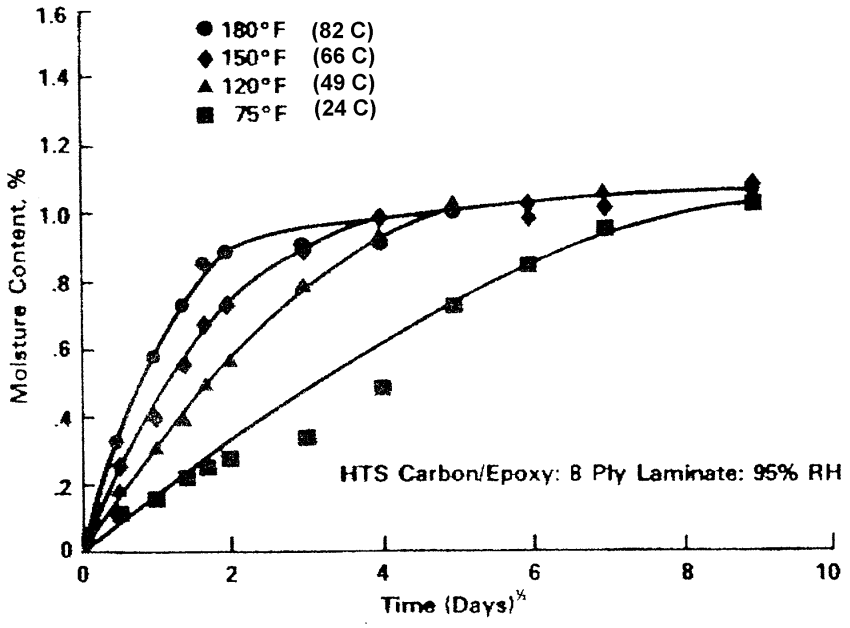


FIGURE 6.3.3(a) *Typical moisture absorption response (Reference 6.3.3(a)).*

The relative humidity level to be used when moisture conditioning is application dependent. As discussed in more detail in Section 2.2.7.3, 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. Accepted design service moisture levels for other applications have not yet been established.

6.3.3.1 Accelerating conditioning times

Because equilibrium moisture conditioning can take a very long time, there is a strong desire to attempt to accelerate the process. While certain two-step, accelerated conditioning cycles are considered acceptable, such as use of an initial high-humidity step (95+% RH) to speed up moisture gain, followed by completion to equilibrium at a lower final humidity level (85% RH), one must be careful not to select an accelerating environment that changes the material, alters the physics of diffusion, or both. Since the moisture diffusion rate is so strongly dependent on temperature, there is a temptation to accelerate the process by increasing the conditioning temperature.[1] However, long exposures to high temperatures combined with moisture may alter the chemistry of the material.[2] 350°F (177°C) cure epoxy-based materials are typically not conditioned above 180°F (82°C) in order to avoid this problem; materials that cure at lower-temperatures may need to be conditioned below 180°F (82°C). And while an initial high relative humidity step is acceptable, the extreme cases of exposure to pressurized steam or immersion in hot/boiling water are *not* accepted methods of accelerating humidity absorption, as they have been found to produce different results from that of 100% humidity.[3]

[1]As an example, for the material illustrated by Figure 2.2.7.1(a), increasing the temperature from 150°F (65°C) to 180°F (82°C) increased the moisture diffusivity of the material from 4.5E-10 in^2/s (2.9E-07 mm^2/s) to 9.8E-10 in^2/s (6.3E-07 mm^2/s), resulting in substantially reduced conditioning times.

[2]The definition of "high" temperature, is, of course, relative to the material system in question, and cannot properly be addressed here.

[3]The differences reported in the literature are probably due in part to excessively-high conditioning temperatures, but even at moderate temperatures water immersion appears to produce a different response in many polymers than water vapor. In some cases, matrix components have been known to dissolve into the water.

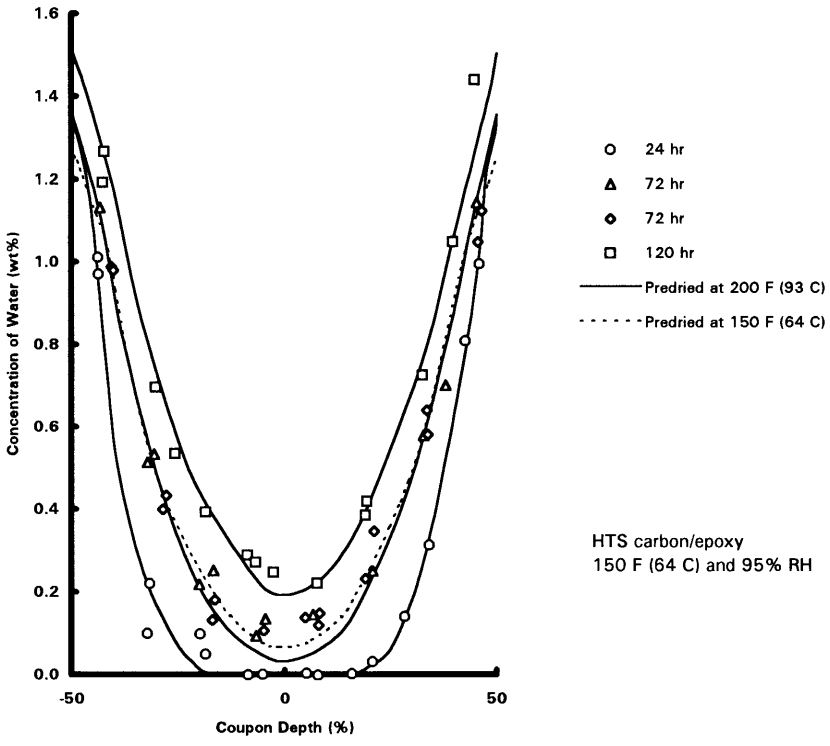


FIGURE 6.3.3(b) *Through the thickness moisture profile versus time (Reference 6.3.3(a)).*

6.3.3.2 Procedural hints

While the procedural description and requirements for ASTM D 5229/D 5229M are fairly complete, the following items justify emphasis:

1. It is highly recommended that some knowledge of the material moisture response be obtained prior to starting conditioning, either from the literature, or from prior test.
2. In moisture property measurement the actual specimen must be initially dry, and the precision and timing of early mass measurements are critical. But for material conditioning needs, knowledge of the initial moisture content may not be important, or may adequately be separately determined from other specimens in parallel. Therefore, it is common not to begin moisture conditioning with a material dry-out step. Moisture conditioning also does not require the repetitive, precise weighings early in the exposure process that are needed to determine the moisture diffusivity. Thus, conditioning without simultaneous determination of the moisture absorption properties is faster and less labor intensive.
3. If the moisture properties are desired, it is faster and less labor intensive to create two other sets of specialized moisture property specimens: a “thin” set that will reach equilibrium quickly, and a “thick” set from which a stable slope to the moisture weight gain versus square-root-time curve can be reliably obtained with minimum test sensitivity. This process is discussed in more detail in Section 6.6.8.

While the procedures for both moisture property determination and equilibrium moisture conditioning are similar, there are some practical reasons why simultaneous determination of moisture properties during a moisture conditioning phase is rarely desirable.

Moisture content measurements are taken either by weighing the actual specimens, or by weighing in their place “travelers,” which are material conditioning specimens cut from the same panel and conditioned at the same time as the specimens. Travelers are required when the specimen is either too small, too large, or includes other materials, such as specimens with tabs, or sandwich specimens. A traveler, when used, accompanies the specimen, or group of related specimens, throughout all subsequent conditioning history.

Because the weight gain of typical polymeric composites is relatively small (on the order of 1%), mass measurement equipment must be selected accordingly. For larger specimens (>50 g), a balance accurate to 0.001 g is generally adequate. For smaller specimens with mass down to 5 g, a precision analytical balance capable of reading to 0.0001 g is required. Direct moisture mass monitoring of coupons weighing less than 5 g is not recommended; a traveler should be used instead.

Near the end of conditioning, minor weighing errors or small relative humidity excursions of the environmental chamber, particularly slight depressions in relative humidity, may artificially cause the material to appear to have reached equilibrium, when, in fact, the material is still absorbing moisture. The lower the temperature (lower the diffusion rate), the more important these errors become. Despite the literal definition of equilibrium expressed by ASTM D 5229/D 5229M, in view of the likely possibility of these experimental errors, the prudent engineer should do the following:

1. Even after the material satisfies the definition of equilibrium, review the chamber records to ensure that a depression in chamber relative humidity did not occur during the reference time period (weighing time interval). If such a depression is found to have occurred, continue the exposure until the chamber has stabilized, then go to item 2.
2. Even after the material satisfies the definition of equilibrium, maintain the exposure, and show satisfaction of the criterion for several consecutive reference time periods.

If the required reference time period does not match a reasonable human time schedule for weighing, then a more regular time interval may be adopted and the ASTM D 5229/D 5229M requirement (less than 0.01% mass change over the reference time period) pro rated to the adjusted time interval. For example, if a required reference time period for equilibrium is determined to be 115,000 s (32 hours), the coupons may be weighed at either 24 hour intervals or 48 hour intervals, with the mass change requirement adjusted from 0.01% to either 0.0075% (24/32 x 0.01) or 0.015% (48/32 x 0.01), respectively.

While many newer models have solid-state controls, a great many environmental chambers control the chamber humidity via monitoring of “dry-bulb” (actual) and “wet-bulb” (moisture depressed) temperatures, which are converted to equivalent relative humidity via a table or algorithm supplied by the manufacturer. The ability of these chambers to control relative humidity is dependent on the accuracy of the thermometer readings. Particularly important in these chambers is regular cleaning of the water reservoir, replacement of the wick, and maintenance of a proper contact between the wick and the wet-bulb thermometer (Reference 6.3.3.2). Chambers that control the dry-bulb temperature and the *differential* between the dry-bulb and wet-bulb temperatures generally have improved control of chamber relative humidity over those that control the dry-bulb and wet-bulb temperatures.

If a drying step is included, whether as an initial step prior to moisture conditioning, or has part of an oven-dry experiment, care should be taken to avoid excessively high drying temperatures and high thermal excursions that may induce thermal cracking in the material.

A variant of equilibrium conditioning uses equilibrium conditioning test data, for a specific material and relative humidity, to establish a table or plotted-curve of minimum exposure time required to achieve equilibrium versus laminate thickness. This approach requires some up-front testing and calculation, but eliminates much of the repetitive weighing otherwise required. A continuous record of the chamber environment must be maintained to prove that proper exposure was achieved.

6.4 INSTRUMENTATION AND CALIBRATION

6.4.1 Introduction

The ability to accurately and repeatably measure deformation and displacement is critical to the testing and characterization of composite materials. This section will discuss the various types of instrumentation used to make strain measurements, and provide guidelines to help determine the appropriate methods for various test types, material forms, test conditions, and data requirements. Only those extensometers which can be classified as ASTM E 83 Class B-2 or better are acceptable for generating data to be included in MIL-HDBK-17 (Reference 6.4.1).

6.4.2 Test specimen dimensional measurement

6.4.2.1 Introduction

Virtually all mechanical property testing requires that dimensional measurements of the test specimen be made. The types of measurements vary depending upon the particular specimen geometry and test requirements, and may include specimen length, width, thickness, gage length, hole diameter, and fastener diameter. Required precision is usually specified by the test method or specification, but generally depends on how a measurement will be used. Some measurements are simply informational, while others are used in calculations (to convert load to stress, for example), and still others are needed to verify conformance to a required geometry. The following five sections discuss (in order of decreasing precision) the various devices commonly used to measure specimen dimensions. Following this is a section on special hole diameter measuring devices. The final section discusses calibration of dimensional measurement devices.

6.4.2.2 Calibrated microscopes

Microscopes with calibrated scales in their eyepieces can provide an extremely accurate means for measuring small specimen dimensions. Resolutions down to 0.0001 inch (2.5 μm) can routinely be attained using magnifications in the range of 50x - 200x. Although this technique is usually more time consuming than micrometer measurement, there are some instances where optical methods may be the only practical option. For example, the thickness of a tabbed specimen may be in question after destruction of the gage section during test. Thickness may be measured and/or verified by optically measuring the thickness of the laminate remaining intact under the bonded tabs. Under the calibrated microscope the laminate thickness between the adhesive bondlines of the tabs can easily be seen and measured (although there is a bias on rough textured specimens). Except for such special cases, however, direct micrometer measurement is usually preferable.

6.4.2.3 Micrometers

Micrometers are precision instruments that are most commonly used for measuring small dimensions. Although some models are available for measurements up to several inches, or even several feet, they generally can only measure continuously over a one inch (25 mm) interval, and require extension rods for different intervals. For this reason calipers are often more convenient for measuring dimensions larger than one inch.

The standard one inch micrometer (25.4 mm)[1] is the most popular instrument for measuring specimen thicknesses. For wide specimens, deep reach micrometers are available for making thickness measurements several inches or more from the specimen edges. The readout may be a scale engraved around the barrel (optionally with a vernier scale), a mechanical digital display, or an electronic digital display. Most instruments indicate in 0.0001 inch graduations and digital models often estimate a fifth decimal place.

[1]Note that the SI equivalent dimensions provided in this section are Asoft≅ conversions, that is SI dimensions for measuring instruments and gradations are provided but sizes are not necessarily converted to SI standard sizes.

Several styles of measuring faces are available. These generally fall into four categories: flat, spherical, blade, and pointed. Both faces on a given instrument may be the same style or different (one face flat and one spherical, for example). Pointed faces are not recommended for use with composites, as they may penetrate the surface (pointed faces are typically used to measure the root diameter of threads). Blade (knife edge) faces are convenient for measuring specimen thickness between bonded tabs on short gage section length specimens. However, such specimens should be carefully inspected for the presence of tab bonding adhesive in the gage section. If adhesive is present the measured laminate thickness will be erroneously inflated.

Flat and spherical (ball) faces are appropriate for most specimen width and thickness measurements, but laminate surface texture should be considered when choosing between these two face styles. For "glass smooth" surfaces, double flat, double ball, or ball-flat faces are all appropriate. However, if the surface is textured (due to coarse weave fabrics, or from use of peel ply during processing, as examples) a flat face will contact the "hills" of the texture, and the resulting measurement will be falsely inflated. A ball face, which will settle somewhat into the "valleys" of the texture or compress the "hills," is therefore preferred. Although the percentage error can vary with specific surface conditions, it is usually not significant for thick specimens. However, for thin (2-3 ply) specimens, measurements may be significantly biased since differences of 0.0015 to 0.0030 inch (0.038 to 0.076 mm) may typically be observed between measurements made with double ball and double flat micrometers. Test specimens that are smooth on one surface and textured on the other may be evaluated by a ball/flat micrometer.

In addition to "stand-alone" micrometers, some testing machines have micrometers integrated into their systems, permitting direct electronic input of specimen dimensions. The system generally prompts the user to position the specimen in the micrometer for width, thickness, and possibly other measurements, and later uses these measurements for calculations. Since the measuring faces fall into the same categories as discussed above, the same considerations apply.

6.4.2.4 Scaled calipers

Scaled calipers are devices with parallel, jaw-like measuring faces and a scale for reading the distance between the stationary face and the movable face. Although models are available for measuring dimensions up to several feet, 6 inch and 12 inch (15 cm and 30 cm) lengths are most common for measuring composite test specimens. The scale may be engraved along the length of the caliper, or may take the form of a dial or digital electronic readout. Although an engraved scale (with auxiliary vernier scale) and the digital readout have 0.0001 inch (2.5 μm) resolution, accuracy is more commonly limited to ±0.001 inch (0.025 mm).

Calipers are convenient for measuring specimen lengths and widths, particularly in the range of 1 - 12 inches (2.5 - 30 cm), since this range exceeds the capability of the common 1 inch micrometer. In addition, some calipers have measuring tips (nibs) designed in such a way that internal as well as external measurements may be made. With this design, calipers may be used to measure hole diameters (in open hole tension and compression specimens, for example). Typically, nibs designed for internal measurement can fit into a 0.25 inch (6.35 mm) or larger hole. Some can read an internal dimension as small as 0.125 inch (3.18 mm).

Calipers may not be particularly suited for measuring specimen thicknesses, especially if the specimen surface(s) is textured. For such measurements a ball-faced instrument is generally preferred (see Section 6.4.2.3 above) as opposed to calipers (which have flat or blade shaped measuring faces).

6.4.2.5 Precision scales

Precision scales are available in various lengths, with 6 inch and 12 inch (15 cm and 30 cm) being common. These tools are similar to rulers, but are usually made of steel and are more precisely and finely graduated. Each instrument typically has four scales, one along each edge of each side. The finest graduations are commonly 1/64 inch or 1/100 (0.01) inch (0.4 mm or 0.25 mm). Reading to 1/100 inch

(0.25 mm) generally requires use of a magnifying glass to discern the graduations clearly. While precision scales may be used for any measurements requiring this resolution, calipers or other instruments are usually easier to use.

6.4.2.6 Rulers and tape measures

These tools are commonly marked in 1/16 inch (1.6 mm) divisions, though some are marked in 1/32 inch (0.79 mm) increments for at least part of their total length. They are generally used for measurements that are recorded for descriptive purposes, but not for more precise measurements. For example, a ruler might be used to identify two groups of specimens: one group with four inch nominal gage lengths, and another group with six inch nominal gage lengths.

6.4.2.7 Special hole diameter measuring devices

Although, as noted above in Section 6.4.2.4, some calipers are designed for inside diameter measurement, special instruments are also available for such measurements. These include telescoping gages, small hole gages, and calibrated pins. Telescoping gages are "T" shaped devices with two spring loaded plungers forming the top of the "T." The measuring faces at the plunger ends are curved and they self-center against the inside walls of the hole. Once in position they are locked by turning a knurled screw on the stem of the "T," and the instrument is withdrawn from the hole. Hole diameter is then determined by measuring the distance between the locked plunger faces using a standard micrometer. The disadvantages of telescoping gages are (1) a set of several gages must be used to cover a range of hole sizes and (2) because of their size, gages are not available for holes smaller than about 5/16 inch (8 mm) diameter.

Small hole gages are similar to telescoping gages except that an adjustable split ball is used instead of plungers. The split ball is placed in the hole and is enlarged by turning the barrel of the device until the ball just contacts the hole walls. The instrument is then removed from the hole and measured with a standard micrometer in the same manner as the telescoping gage. These gages must also be used in sets to cover a wide range of hole sizes but, unlike the telescoping gages, holes down to about 1/8 inch diameter can be measured.

Sets of calibrated pins of known diameter may also be used to measure hole diameters. Pins of various sizes are inserted into the hole until a close, but not tight, fit is obtained. The hole diameter is then taken as the pin diameter. Pins are available in virtually any size, and are generally graduated in 0.0005 inch increments. Very extensive sets are needed to cover a range of nominal hole sizes.

Of the devices available, calipers or small hole gages are most useful and economical for measuring hole diameters in composite test specimens.

6.4.2.8 Calibration of dimensional measurement devices

In order to maintain the stated accuracy of mechanical measuring devices such as micrometers and calipers, they must be periodically calibrated. In general, there are no detailed calibration procedures available in high level (ASTM, ANSI, etc.) U.S. standards. Typically these instruments are calibrated using gage blocks, and specific procedures are contained in company internal specifications. Some ISO documents address aspects of this subject, and the reader is referred to standards under the jurisdiction of ISO Technical Committee 3 on Limits and Fits, as well as to ISO 10012-1 (Reference 6.4.2.8).

6.4.3 Load measurement devices

6.4.3.1 Introduction

The ability to accurately and repeatably measure load (force) is critical to the testing and characterization of composite materials. This section will discuss the various types of instrumentation used to make load measurements, and provide guidelines to insure the accuracy of those measurements. Load meas-

urement device classification and verification is discussed in ASTM E4 “Standard Practices for Force Verification of Testing Machines” (Reference 6.4.3.1(a)), ASTM E74 “Standard Practice of Calibration of Force-Measuring Instruments for Verifying the Force Indication of Testing Machines” (Reference 6.4.3.1(b)), and ASTM E467 “Standard Practice for Verification of Constant Amplitude Dynamic Loads on Displacements in an Axial Load Fatigue Testing System” (Reference 6.4.3.1(c)). Calibration of load devices is also discussed in ISO 5893 “Rubber and plastics test equipment – Tensile, flexural and compressive types (constant rate of traverse) – Description” (Reference 6.4.3.1(d)).

Note: Force in the case of testing machines is defined as pound-force, or Newton where one pound force is the force required to provide a one pound mass an acceleration of 32.1740 ft/sec^2 (9.80665 m/sec^2), and a Newton is the force required to provide a one kilogram mass an acceleration of 1m/sec. This force is used to determine the load applied on test specimens. Load is commonly used interchangeably with force in mechanical testing specifications and in MIL-HDBK-17.

6.4.3.2 Load cells

The most common type of force measurement device in the mechanical properties testing laboratory is the strain-gage instrumented load cell. These devices consist of an elastic member that deflects in a uniform, consistent, and repeatable manner under the application of load. The elastic member in the load cell is instrumented with strain gages so as to measure the deflection. The output of the strain gage circuit can easily be read by a variety of recording devices and data acquisition systems. The strain gages in the load cell form a complete bridge, carefully balanced, so that the load cell can be calibrated using a reference excitation voltage, and thereafter the output of the bridge circuit will be dependent only upon the external conditioning circuitry. The bridge is, therefore, guaranteed to be in a balanced condition (at thermal equilibrium) when no load is applied. Load cells with internal signal conditioning circuitry should be avoided when circumstances may require the heating and/or cooling of the load cell. An important factor in the design or choice of a load cell is the ability of the load cell to reject spurious inputs generated by improper but inevitable misuse, such as off-axis loading and heating/cooling of the load cell during a test. The ability of a load cell to reject off-axis loads and thermal drift is dependent on the design of the elastic member and the placement of the strain gages upon that member. (See Section 6.4.2.4 on strain gages). A well designed load cell can have a repeatability of 0.01% (of the full-scale output of the load cell), and a thermal stability of 0.001% (full scale) per degree F.

6.4.3.2.1 Design and specification considerations

Load cells should be chosen to provide the greatest degree of accuracy consistent with the required data. An indicated load accurate to within 0.1% of the actual load at critical points in the test (modulus chord points, failure load) will guarantee high quality test results. A variety of load cell configurations is available:

1. Bending beam load cells are constructed as a simple cantilever beam, with strain gages attached to measure deflection. The beam may be instrumented with a single strain gage (quarter bridge), two gages (half bridge), or four gages (full bridge). When two gages are used, they are connected in such a manner that the strains in the gages are summed, effectively doubling the sensitivity of the circuit. When four gages are used, they may be arranged so as to quadruple the sensitivity, or to compensate for the nonlinearity of the strain gradient in the beam. Bending beam load cells are used when cost is a factor, as when destruction of the load cell is a possibility. High accuracy is possible with this type of load cell when it is used correctly. The “S” beam load cell is a special form of the bending beam load cell which permits “in-line” loading to be used with an inexpensive load cell design. Bending beam load cells can reject torsional loading of the beam, and thermal effects, but at higher strains some designs become markedly nonlinear while still producing repeatable results.

2. Shear beam load cells, in their simplest form, utilize the uniform shear condition in the web of an I-beam shaped member as the surface of measurement. Precision load cells commonly utilize eight or twelve mechanical elements of the shear beam type arranged in a radially symmetric pattern,

which combined with a well designed bridge circuit utilizing four of the shear beams, allows the load cell to reject off-axis loads.

3. Ring load cells, essentially so-called proving rings, consist of an elastic member of a ring shape, which when loaded at diametrically opposite points deforms elliptically. This type of load cell can be of high accuracy, but does a poor job of rejecting off axis loads.

6.4.3.3 Other load measuring systems

The following is a brief summary of other types of load measuring devices sometimes used. These systems are generally for highly specialized uses, or are based on older technology, and are not preferred for obtaining MIL-HDBK-17 data.

LVDT Devices -- A load cell which uses an LVDT (linear variable differential transformer, see 6.7.2.4.4) as a strain measuring device may occasionally be seen. This type of load cell may be as accurate as a bonded strain gage type cell, but is somewhat less rugged.

Solid state load transducers -- Special purpose load cells utilizing piezoelectric or piezoresistive semiconductor strain measuring elements (see "Strain Gage Technology" (Reference 6.4.3.3) are available for measuring load during impact, when the strain change rate might exceed the ability of a bonded foil strain gage load cell to accurately indicate load. Semiconductor strain gages are extremely sensitive to temperature changes, and will yield rapid zero shifts with changing temperature. Therefore, they must be used only at thermal equilibrium.

Bourdon tubes, etc. -- There are older test machines in everyday use which rely on Bourdon tubes and other ingenious mechanisms for indicating load. A Bourdon tube is a sealed tube, formed in a spiral, semi-circle or helical shape and filled with fluid. When pressurized, the fluid causes the tube to move in a reproducible manner, mechanically acting on a readout device or indicating needle. The indicating dials on these machines should be relied on only as relative indicators of load level. In all cases, these machines should be retro-fitted with electronic load cells and indicators which can be calibrated more readily, and to greater degrees of accuracy.

Calibrated weights -- Creep testing machines are commonly of the unequal arm dead-weight loading type. The weights used on these machines are commonly cast iron and should be calibrated to Class 6 of ASTM E 617 (note this is the least precise class given in ASTM E 617 and has a tolerance of 0.01%). The measurement of the length of the arms and the condition of the knife-edges should be verified per the machine manufacturers instructions.

Levers -- Test machines with an integral system of levers and knife edges for indicating load as on a beam balance or compound scale should be retrofitted with electronic load indicators to simplify calibration and data acquisition.

6.4.3.4 Instrumentation and calibration

Calibration (more properly, verification) of test machines and load cells requires a "CLASS A" load standard. Those standards are commonly high precision load cells or proving rings. The load standard must have an uncertainty not exceeding 0.25% of the load being measured. Therefore, the minimum load which it may be used to calibrate must be at least 400 times the uncertainty. ASTM E 4 allows the uncertainty of a load device used for testing to be up to 1% of full scale. ASTM E 74 contains a detailed explanation and analysis of the calibration of load measuring devices, and it should be studied closely by anyone responsible for calibrating these devices. Machines meeting the ISO requirements have a sliding scale of allowable error with a maximum of ±1.0% at full scale for Grade A machines and ±2.0% of full scale for grade B machines. A load cell calibrated to ISO 5893, Grade A meets the load cell requirements of ASTM E 4, though there are additional requirements in ASTM E 4 for test machines that are not covered in the ISO standard. The ISO and ASTM standards both include other details such that they are not strictly interchangeable.

6.4.3.5 Precautions

Certain precautions should be observed to insure the accuracy of load cell readings.

a. In all cases, scrutinize the specifications of a commercial load cell, or carefully analyze the bridge circuit of a self manufactured load cell. Curve fitting of nonlinear output is possible, but care should be taken to insure that the fitting equation is correct, and that it is applied correctly.

b. The load cell should be calibrated at regular intervals to verify its performance per ASTM E 4 and E 74. The calibration device should be traceable to a national standard such as NIST, and its readout and accuracy should exceed that of the device being calibrated by a factor of 4 or more.

c. The load cell calibration should be reverified whenever unusual loading conditions occur, such as overloading, impacting, or bending (off-axis loading). Refer to the specification of the individual load cell for overload tolerances.

d. Load cells not specifically declared to be tolerant of temperature changes during testing should be assumed to be inaccurate at elevated or depressed temperatures. Therefore, care should be taken to isolate the load cell and its cabling, from temperature changes and/or gradients.

e. Care must be taken to insure that the applied load axis corresponds as nearly as possible to the indicated loading axis of the load cell. Off-axis loads should be avoided and may result in inaccurate readings, and may damage the load cell or other parts of the load train.

f. In general, the capacity of the load cell to be used for a given test should be determined such that the predicted failure load is between 15% and 85% of the capacity of the load cell. If expected loads are less than 15% of the load cell capacity, the user should insure that adequate calibration has been performed in the test range. Such a calibration may be outside the scope of a routine ASTM E 4 calibration, so special arrangements may need to be made. When the instrumentation used permits the “ranging” of the load cell, for instance where a 100,000 pound load cell might be used over a range of 10,000 pounds through amplification, the load cell must be separately calibrated under those circumstances as a 10,000 pound capacity load cell. Similar individual calibrations must be conducted for all “ranges” provided. Use of a load cell for testing when the expected data is greater than 85% of the load cell capacity is discouraged since an unexpected high load may exceed the capacity of the load cell.

6.4.4 Strain/displacement measurement devices

6.4.4.1 Introduction

The ability to accurately and repeatability measure deformation and displacement is critical to the testing and characterization of composite materials. This section will discuss the various types of instrumentation used to make strain measurements, and provide guidelines to help determine the appropriate methods for various test types, material forms, test conditions, and data requirements. Extensometer classification and verification is discussed in ASTM E 83 (Reference 6.4.1). The class of the extensometer is determined from the maximum expected error. Class A has the least expected error, followed by classes B-1, B-2, C, D, and E in that order. Calibration to class A is very difficult to achieve. Only those extensometers which can be classified as ASTM E 83 Class B-2 or better are acceptable for generating data to be included in MIL-HDBK-17.

6.4.4.2 LVDT (Linear Variable Differential Transformer) deflectometers

LVDT's are electromagnetic devices designed so that as a ferromagnetic core is displaced within a transformer (consisting of three windings), a linearly varying a.c. voltage and phase shift are produced, this signal is demodulated to produce a varying d.c. output. LVDT's are available in both linear and angular configurations. LVDT's are available in lengths to 10 feet (3 meters), their output linearity is about 0.1%, and their maximum resolution is 1 microinch (25 μm). The accuracy of a given LVDT is commonly limited to 0.01% of total travel. An LVDT may be used directly as a deflectometer with its core contacting the specimen; it can be used with a linkage; or it can be incorporated into a contacting extensometer High temperature LVDT's may be usable up to the Curie Temperature of the core material, but are generally used with extensions or linkages to avoid exposing them to hostile environments. LVDT's must be calibrated at the temperature to which they will be exposed in use.

6.4.4.3 Contacting extensometers

Contacting extensometers and compressometers are devices that are used to determine the relative displacements of two points on a specimen. The contact extensometer must be clamped to the specimen surface in such a way that the contact points cannot slip, and that the extensometer does not affect the test. Extensometers are relatively complex devices which rely on integral strain gages or LVDT's to convert the relative displacements of their attachment points into linearly related outputs. Extensometers are available in a range of fixed gage lengths from 0.500 to 2.00 in. (12 - 50 mm), their output linearity is 0.1%, and they can resolve displacement to 1 microinch (25 μm). This resolution does not imply accuracy or calibration. A well-made contact extensometer is accurate to 0.01% of full scale, and can measure strain up to 1.00 (100%). Repeatability of contacting extensometers is dependent on their maintaining a constant initial gage length, therefore, when a zero stop is provided it should always be used when attaching the extensometer to a specimen.

Contact extensometers are available which can be used at liquid nitrogen temperatures, others can safely be exposed to temperatures of 500°F (260°C) for extended periods of time. Extensions and linkages are available which allow remote use of extensometers on specimens exposed to temperatures up to 3000°F (1600°C). ASTM E 83 requires that extensometers be calibrated at the temperature at which they will be used. Extensometer calibration should be verified whenever the extensometer is subjected to deflection exceeding the normal range, been exposed to a hostile environment, received rough handling, and whenever the knife edges or points are replaced.

6.4.4.3.1 Contacting extensometers, applications

Extensometers are chosen in preference to bondable strain gages when one or more of the following conditions exist:

1. The price of individual bonded strain gages exceeds the cost of a comparable extensometer.
2. The construction of a laminate will induce a non-uniform strain field under a bonded strain gage.
3. Strains will exceed the practical limit of bonded strain gages (0.03 or 3%).
4. The net deformation of a complex structure or assembly is required (for example a bonded or bolted joint).
5. When specimen conditioning or preconditioning will not allow proper bonding of strain gages.

Extensometers are not recommended when the following circumstances apply:

1. Extensometers fitted with points or knife edges may cause premature failures in notch sensitive materials.
2. Extensometers of large inertial mass respond unpredictably to rapid changes in strain.
3. Catastrophic failure of a specimen while an extensometer is attached will result in damage to the extensometer requiring repair and recalibration or replacement.

6.4.4.4 Bondable resistance strain gages

Strain gages are structures of precisely etched metal foil or wire (usually on a polyimide film substrate) which are permanently bonded to a specimen surface so that the strain field of that surface is immediately transmitted to the gage. In use, the strain gage forms part of a Wheatstone Bridge circuit, which allows strain to be accurately measured as a function of the change in resistance of the grid. Strain gages are made from alloys (Constantan, Karma Alloy) which show relatively small changes in strain sensitivity (ratio of change in resistance to change in length) when they are deformed beyond their proportional limits (Reference 6.4.4.4).

Strain gages have inherently infinite resolution (limited by the accuracy of the gage factor calibration); their ability to indicate small changes in strain accurately is limited only by the instrumentation used.

Strain gages are versatile:

1. Strain gages can be applied directly to a specimen, or can be used to construct extensometers or beam bending deflectometers.
2. Several strain gages can be applied to a single specimen in different orientations to measure simultaneous multiaxial properties.
3. Several strain gages can be applied to a single specimen in various places in similar orientations to identify stress concentrations.

6.4.4.4.1 Strain gage selection

Strain gages are available in a wide range of styles. The selection of the proper strain gage is critical if accurate and repeatable results are to be obtained. Polymeric matrix composites are relatively poor thermal conductors, therefore, 350Ω or higher resistance gages are usually chosen in preference to 120Ω gages, higher resistance gages operate at lower currents for a given strain and are less likely to produce errors due to self-heating (Reference 6.4.4.4.1(a)).

Since stresses in woven composites are transmitted by the interaction of relatively large repeating units, the gage must be large enough to integrate any strain gradient associated with the weave. The grid size chosen for a composite specimen will generally be larger than that for a similar metal specimen. Grid sizes of 0.125, 0.250 and 0.500 in. (3.17, 6.35, and 12.7 mm) are commonly used, with specimen size limiting the size of the gage which can be used. The installation of gages very close to specimen edges is to be avoided, as edge effects are difficult to predict. Finally, gages are made to function optimally over a limited range of temperatures, and it is important that the manufacturers' recommendations be heeded regarding maximum operating temperatures of different gage styles (Reference 6.4.4.4.1(b)).

6.4.4.4.2 Surface preparation and bonding of strain gages

Careful evaluation of surface preparation and bonding techniques for strain gages must be done if reliable data are to be obtained. Details of these techniques will be found in Section 6.2 and Reference 6.4.4.4.2. Extreme care should be used when abrading composites to minimize damage to the fibers of the surface laminae. It should be noted that the bonding of strain gages to thermoplastic materials is especially difficult.

6.4.4.4.3 Strain gage circuits

A strain gage or gages function as the variable element(s) in a resistance bridge; the Wheatstone bridge of four elements, shown in Figure 6.4.4.4.3, is the most usual. The diagram illustrates a 1/4 bridge, with a single active gage, 3-wire configuration (the 3-wire configuration removes the effects of lead wire resistance from the circuit). P+ and P- represent the excitation voltage for the bridge, S+ and S- represent the output signal. R1 and R3 are fixed resistors of identical value. When R2 and RG (the resistance of the strain gage) are identical, the bridge is said to be balanced, and no current flows between S+ and S-. A change in resistance of similar value and sign in *adjacent* elements (e.g., R2, R3) is a null input

to the bridge. A change in resistance of similar value and sign in *opposite* elements (for example, R1, R3) is summed in magnitude. These results are useful in strain measurement: in the first case a gage can be applied to a spare piece of specimen material, and if this second gage is positioned at R1 in the circuit (therefore adjacent to RG) and then exposed to the test conditions, it will compensate for the thermal responses of the specimen and the active gage. In the second case, referred to as a half bridge, a specimen has two active gages both placed within a constant strain field, the second gage is placed at R2 (opposite to RG), then the gage outputs will be summed, and dividing by 2 will give the average strain, with a 2-fold increase in resolution. Contact extensometers are often designed using four gages in a "Full-Bridge" configuration which makes good use of the bridge by effectively summing all elements (adjacent gages are positioned so as to be exposed to strain fields of equal value and opposite sign). In all cases where passive bridge elements exist they are referred to as "Bridge Completion" and are a necessary part of the instrumentation associated with strain gages.

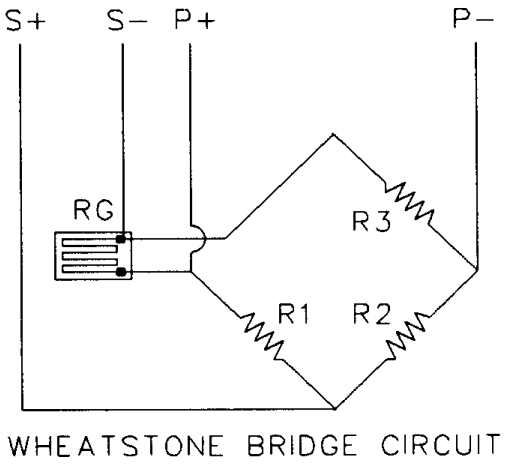


FIGURE 6.4.4.4.3 *Wheatstone bridge.*

6.4.4.4.4 Strain gage instrumentation

The instrumentation used with strain gages (and extensometers utilizing strain gages as their active elements) is usually of the constant voltage type. The bridge circuit is provided with a stabilized d.c. excitation voltage between 2 and 10 Volts, and the output is on the scale of microvolts. High gain instrumentation amplifiers with low drift and excellent stability are used to scale the outputs up to Volt levels.

The combination of excitation and amplification in a single instrument is called a *conditioner*. Conditioners are available with fixed or variable excitation voltages. A variable excitation conditioner can be used to achieve high resolutions at high excitation voltages (high signal to noise ratio), or extended strain ranges at low voltages. It is a good idea to avoid using excitation voltages greater than 10 Volts for 350Ω gages on polymer matrix composites, which do not dissipate heat efficiently, to avoid "self-heating" of the gage (Reference 6.4.4.4.4). Conditioners with fixed excitation voltages usually offer variable amplifier gains to scale outputs. There is less possibility of overheating the gage with a fixed voltage conditioner.

6.4.4.4.5 Strain gage instrumentation calibration

Strain conditioner linearity is verified by the use of strain simulation. With 350Ω taken as the balance point or zero, strain values can be simulated by using a high accuracy decade resistance box with ranges from 0.01Ω to 100Ω in place of the active gage, and using the following equation to simulate strain values:

$$\Omega = 0.0007\,\varepsilon_{sim} + 350 \qquad 6.4.4.4.5$$

where

Ω = decade resistance box setting to simulate target strain (ohms)
ε_{sim} = target strain to be simulated (microstrain)

When fixed excitation conditioners have been verified in this way and found acceptable, no further calibration is necessary before testing. The output of the conditioner is simply multiplied by $2/K$, where K is the *gage factor* reported by the gage manufacturer.

When conditioners offer variable excitation, *shunt calibration* is required.

6.4.4.4.5.1 Shunt calibration (for 1/4 bridge)

When a variable excitation conditioner is used, the excitation voltage is generally chosen to scale the conditioner output (span) to the expected maximum strain level expected in the test. This provides the maximum resolution over the range of the test. With an active gage in the circuit (usually an actual specimen with no load applied), the conditioner output is zeroed. A precision resistor is placed in the circuit parallel with a bridge resistor. The value of the resistor is chosen so that when it is wired parallel to the gage, the combined resistance is exactly that necessary to simulate a known strain, called the shunt value. The excitation voltage is then adjusted so that the conditioner readout shows a value equal to $2/K$ multiplied by the shunt value. After instrument scaling, the indicated strain will be correct at the magnitude of the calibration strain, but slightly in error at other strain levels. The corrected strain at any different strain level can be calculated from Reference 6.4.4.4.1(b) :

$$\varepsilon = 2\,\varepsilon_i / (2 + K(\varepsilon_s - \varepsilon_i)) \qquad 6.4.4.4.5.1$$

where

ε = corrected strain (microstrain)
ε_i = indicated strain (microstrain)
ε_s = shunt cal value (microstrain)
K = gage factor of strain gage

The topic of shunt calibration of Wheatstone bridges is treated simply here, but is actually a matter of great complexity, and it is recommended that the serious researcher carefully study Reference 6.4.4.4.5.1.

6.4.4.5 Other methods

A number of extensometric methods exist which see limited use in the determination of polymer matrix composite properties due either to unreliability or difficulty of use. However, under appropriate circumstances these techniques yield valuable data which could otherwise not be obtained, therefore, they are described here in limited detail.

6.4.4.5.1 Optical methods of extensometry

A number of methods of strain measurement based on optical phenomena exist: photoelasticity, Moiré interferometry, and laser extensometry. Photoelastic methods and Moiré may be used to verify the results of finite element calculations, and to investigate stress distributions on test specimens or struc-

tures. The application of these techniques to the design of test specimens and fixturing is an important stage in optimization of test geometry.

The non-contact nature of laser extensometry makes it particularly attractive in circumstances where strain gages would be unreliable - at high temperatures, on small radii, and on rough surfaces.

6.4.4.5.2 Capacitative extensometers

Contact extensometers are available which utilize the capacitance of an air gap between two probes fixed to the specimen surface to determine strain. These probes are accurate only for very small gage lengths, and cannot be used to record strain to failure as they are easily destroyed. They are used to determine modulus of materials at very high temperatures (>1000°F or 500°C). Capacitative extensometers can be difficult to calibrate and require complicated conditioning instrumentation. They cannot be calibrated better than ASTM E 83 Class B-2.

6.4.4.6 Special considerations for textile composites

The inhomogenity of textile composites requires that strains and displacements are measured over sufficient gage lengths to be representative of bulk (average) specimen response. Results of a study on composites made of 2D triaxial braids, 3D weaves, and stitched uniwoven laminates to determine the effect of strain gage size on strain measurements are given in Reference 6.4.4.6(a).

In general, strain gages should be longer than the unit cell length of the textile and gage width should be no less than half the length. For specific standards in selecting strain gages use Reference 6.4.4.6(b). The gage lengths of extensometers should also be larger than the unit cell size to obtain an average or macroscopic displacement. These recommendations for minimum gage length should apply for thermal loads as well as mechanical loads.

Although not addressed in References 6.4.4.6(a) and (b), several gages might be arranged end to end to avoid more costly special order gages for unit cells longer than .5 in. (12.7 mm).

6.4.5 Temperature measurement devices

6.4.5.1 Introduction

Many of the properties that characterize a composite lamina or laminate are temperature dependent. Thus, temperature is one of the variables that must be measured to fully characterize a material. Many tools and techniques exist to measure temperature, but not all will provide the desired results or function in the required environment for the duration of the test. Temperature measurement devices can be divided into two categories: contact and noncontact. Five types of contact temperature measuring devices are commonly encountered: thermocouples, resistive temperature devices (metallic RTDs and thermistors), bimetallic devices, liquid expansion devices, and change of state devices. A noncontact temperature measuring device commonly used is an infrared detector.

6.4.5.2 Thermocouples

A thermocouple consists essentially of two strips or wires made of different metal alloys and joined at one end. Referring to Figure 6.4.5.2., changes in the temperature T1 at that junction induce a change in electromotive force (emf) V_{ab} between the other ends. As temperature goes up, this output emf of the thermocouple rises, though not necessarily linearly. The open-end emf is a function of not only the closed-end temperature T1 (i.e., the temperature at the point of measurement), but also the temperature T2 at the open end. Only by holding T2 at a standard temperature can the measured emf be considered a direct function of the change in T1. The industrially accepted standard for T2 is 32°F (0°C); therefore, most tables and charts assume that T2 is at that level. In industrial instrumentation, the difference between actual temperature at T2 and 32°F (0°C) is usually corrected for electronically, within the instrumentation. This emf adjustment is referred to as the cold-junction, or CJ, correction. Temperature

changes in the wiring between the input and output ends do not affect the output voltage, provided that the wiring is of thermocouple alloy or a thermoelectric equivalent. For example, if a thermocouple is measuring temperature in a furnace and the instrument that shows the reading is some distance away, the wiring between the two could pass near another furnace and not be affected by its temperature, unless it becomes hot enough to melt the wire or permanently change its electrothermal behavior.

Thermocouples have advantages over other contact sensors in that they are simple, rugged, inexpensive, require no external power, are available in a wide variety of forms, and can be used over wide temperature ranges. Disadvantages of thermocouples are that they are nonlinear, produce very low voltages, and require an external temperature reference.

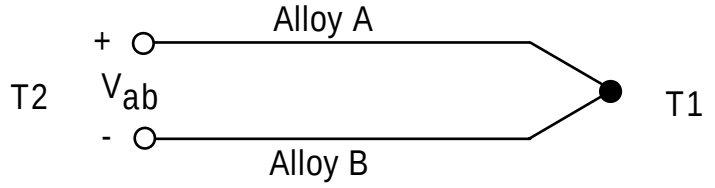


Figure 6.4.5.2 *Schematic of thermocouple junction.*

Thermocouples must be selected to meet the conditions of the application. Only general recommendations on size and type can be given. Some of the considerations involved are length of service, temperature, atmosphere, and desired response time. Smaller gauge sizes provide faster response at the expense of service life at the elevated temperatures. Larger gauge sizes provide longer service life at the expense of response time. As a rule, it is advisable to protect thermocouple elements with a suitable protecting tube or drilled well.

Thermocouples are available in different combination of metals or 'calibrations'. The four most common calibrations are J, K, T, and E. Each calibration has a different temperature range and environment, although the maximum temperature varies with the diameter of the wire used in the thermocouple.

Type J: [Iron (+) Constantan (–)]
Maximum recommended operating temperature is 1400°F (760°C).
Type K: [CHROMEL (+) ALUMEL (–)]
Maximum recommended operating temperature is 2300°F (1260°C).
Type T: [Copper (+) Constantan (–)]
Recommended operating temperature range is –328 to 662°F (–200 to 350°C).
Type E: [CHROMEL (+) Constantan (–)]
Maximum recommended operating temperature is 1652°F (900°C).

6.4.5.3 Metallic resistive temperature devices

Resistance temperature devices (RTDs) rely on the temperature dependence of a material's electrical resistance. They are usually made of a pure metal having a small but accurate positive temperature coefficient. A typical metallic RTD consists of a fine platinum wire wrapped around a mandrel and encased in a protective coating. Usually, the mandrel and coating are glass or ceramic. The resistance of the platinum wire rises more or less linearly with temperature. By measuring the resistance of the wire, its temperature can be determined. RTDs made of platinum wire are well characterized and linear from –434 to 1112°F (–259 to 600°C).

Although the response of an RTD is more stable and linear than that of a thermocouple, RTDs cannot be used over as broad a temperature range as thermocouples. The large thermal mass and poorer ther-

mal coupling combine to produce slow response to temperature changes. The RTD responds to mechanical, as well as thermal strains, making it sensitive to loads and vibration, in addition to temperature. Unlike the thermocouple, the RTD is not self-powered. Since a current must be passed through the device to provide a voltage that can be measured, the device is prone to self heating. This is particularly true if a large current is employed, a small RTD is employed, or if the RTD is not well coupled thermally.

6.4.5.4 Thermistors

Thermistors are generally composed of ceramic semiconductor materials that exhibit a large change in resistance with a change in temperature. There are both positive temperature coefficient (PTC) and negative temperature coefficient (NTC) devices on the market. A PTC thermistor is defined by an increase in resistance with an increase in temperature. A NTC thermistor is defined by a decrease in resistance with an increase in temperature. The majority of thermistors, however, are of type NTC.

Thermistors can generally be classified into two major groups depending upon the method by which electrodes are attached to the ceramic body. The first group consists of bead type thermistors and the second group consists of metallized surface contact thermistors. All of the bead type thermistors have platinum alloy leadwires which are sintered into the ceramic body. As a group, the sealed bead type thermistors are more stable than the metallized surface contact type. The bead types are generally smaller in size and have faster thermal time constant values. That is an advantage in many temperature measurement applications. However, the bead types have lower dissipation values that result in greater self-heating effects in most applications. The metallized surface contact type thermistors are easier to manufacture and, therefore, less expensive than the bead type thermistors. However, the metallized surface contact type thermistors are generally rated at 300°F (150°C) with the best continuous operating temperature stability at 221°F (105°C) or less.

Thermistors are extremely sensitive to temperature changes and can detect temperature changes that could not be observed using other devices. Although thermistors can be very accurate, their measurement range is small in comparison to thermocouples and RTDs. Since a current must be passed through the device to provide a voltage that can be measured, the device is prone to self heating. Thermistors are also somewhat more fragile than other temperature measurement devices.

6.4.5.5 Bimetallic devices

Bimetallic temperature indicators take advantage of the difference in the rate of thermal expansion of different metals. Strips of two dissimilar metals are bonded together. When heated, one side of the composite will expand relative to the other. The resulting bending is translated to a temperature reading via mechanical linkages. These devices are portable, and require no power. However, they are not as accurate as other temperature measurement devices, cannot be used to make point measurements, and do not generate data in a form that can be readily recorded. They can be used to acquire a qualitative record of the ambient temperature if a pen is attached to the indicating pointer, and traces a line on a moving chart.

6.4.5.6 Liquid expansion devices

Liquid expansion devices, typified by the liquid-column bulb thermometer, require no power, and are stable even after repeated thermal cycling. On the other hand, they do not generate data that can be easily recorded and they do not respond well to transient temperature changes. Since they must be immersed in the medium whose temperature is being measured, they cannot be used to make point measurements. Their primary use is measuring the temperature of the test environment.

6.4.5.7 Change-of-state devices

Change-of-state temperature sensors consist of various labels, pellets, crayons, lacquers, or liquid crystals whose appearance changes once a certain temperature is reached. The typical response time is measured in minutes, so they do not respond well to transient temperature changes. The accuracy is

lower than with other types of sensors, and the change in state is irreversible, except in the case of liquid crystal displays. Change-of-state sensors can provide a handy, qualitative confirmation that a material has, or has not, reached or exceeded some temperature.

6.4.5.8 Infrared detectors

Infrared (IR) detectors are noncontacting devices that measure the amount of radiation emitted by a surface. At temperatures above absolute zero, all matter radiates electromagnetic energy. The level and frequency of the radiated energy are proportional to temperature. In many engineering situations, much of the radiation is in the infrared region. If the radiating characteristics of the surface are known, its temperature can be inferred from the level of the infrared energy at a specific wavelength. The simplest IR detector design consists of a lens to focus the IR energy onto a detector, that converts the energy to electrical signals that are displayed in units of temperature after being adjusted for ambient temperature variations.

IR thermometers (IRT) come in a wide variety of configurations pertaining to optics, electronics, technology, size, and protective enclosures. The basic IRT design is comprised of a lens to collect the energy emitted from the target; a detector to convert the energy to an electrical signal; an emissivity adjustment to match the IRT calibration to the emissivity characteristics of the object being measured; and an ambient temperature compensation circuit to ensure that the temperature variations within the IRT due to ambient changes are not transferred to the final output.

Single-wavelength thermometry design measures the total energy emitted from a surface at a prescribed wavelength. These devices measure and evaluate the intensity, or brightness, of the intercepted thermal radiation. Intensity, or, more generally, radiance is measured in a narrow wavelength band of the thermal spectrum. Band selection is dictated by the temperature range and the type of material to be measured. The configuration can range from handheld probes with a simple remote meter to sophisticated portables with simultaneous viewing of target and temperature, plus memory and/or printout capabilities.

Dual- and multi-wavelength thermometry are used in applications where absolute accuracy is critical, and where the product is undergoing a physical or chemical change. Dual-wavelength thermometry involves measuring the spectral energy at two different wavelengths. The target temperature can be read directly from the instrument if the emissivity has the same value at both wavelengths. The advantage of ratio measuring is that temperature readings are greatly independent of emissivity fluctuations and/or sight path obscurations. The technique is generally used for temperatures above incandescence (1300°F (700°C)), but measurements down to 400°F (200°C) are possible.

Advantages of infrared detectors are that they are non-contacting, can be used to measure very high temperatures, and can be used to measure temperatures in hostile environments, provided visual access can be obtained. One disadvantage is that the surface emissivity at the temperature of interest must be known (this information is not always known). In addition, the device will average all of the temperatures in its field of view. If a target does not completely fill the field, the temperature of its background will contribute to the reading. If the target is not a perfect emitter, it will reflect infrared energy from other sources that can be detected by the device.

6.4.5.9 Calibration of temperature measurement devices

The effectiveness of any temperature measuring equipment is dependent on its accuracy and its repeatability. As with other measuring equipment, temperature devices must be calibrated and periodically verified to maintain confidence that their indicated output is within a certain known tolerance to the true value. Calibration and verification of temperature devices is simple in concept and involves merely exposing the device of interest and a reference device to the same temperature. Any deviation of outputs then can be corrected, in the case of calibration, or noted as in or out of tolerance, in the case of verification. For the purposes of this document, calibration and verification will be considered together and will

both be referred to as “calibration”. General information on temperature measurement can be found in References 6.4.5.9(a) and (b).

Temperature measurement devices are nearly always attached to a readout or control instrument of some type, which must also be calibrated. Often the instrument can be included with the probe and the assembly calibrated as a system. This is preferred because all components of the system are considered together, which leads to greater accuracy and can save considerable time. The user should refer to the specific instrument operations manual for its calibration requirements and procedures. Additionally, good information for temperature device calibration can be found in the following ASTM specifications:

- ASTM E220 Calibration of Thermocouples by Comparison Techniques (Reference 6.4.5.9(c)).
- ASTM E77-92 Standard Test Method for Inspection and Verification of Thermometers (Reference 6.4.5.9(d)).
- ASTM E1502 Use of Freezing Point Cells for Reference Temperatures (Reference 6.4.5.9(e)).

Note that though these standards are generally oriented toward a particular type of sensor, many of the practices can also be applied to other sensor types, particularly when applied in concert with the instructions provided in the owner’s manual for a given temperature sensor or system.

The general calibration procedure for probes involves physically placing them in a known temperature environment together with a reference standard that should be traceable to National Institute of Standards and Technology (NIST) standards. The critical components of a probe calibration setup are shown in Figure 6.4.5.9, and include:

- The “calibrator” (the device used to generate a known temperature).
- The reference standard probe - usually of the same type as the probe being calibrated.
- A readout device (typically a high resolution digital multimeter of 5½ digit resolution or an indicator which provides scaling and cold junction compensation for the probe).
- An ice point reference (used to locate the open end of a thermocouple - T_2 - at the proper reference temperature as discussed in Section 6.4.5.2). This is required when calibrating thermocouple type probes only, and provides the cold junction reference for thermocouple probes not otherwise compensated for the cold junction.

The Calibrator

Calibrators are the heating or cooling sources used to provide the thermal environment into which the instrument to be calibrated is placed. Calibrators must have outstanding temperature control capability, be extremely thermally stable, and free from temperature gradients. Circulating air furnaces are usually not sufficiently stable and exhibit relatively high thermal gradients. The calibrators most often used are specifically designed for probe calibration and are one of three types: block calibrators, circulating liquid baths, and fluidized powder baths.

The block calibrator consists of an electrically powered unit that uniformly heats or cools a solid block of material (often copper) into which the probes are inserted. Block calibrators are clean and easy to maintain, but change temperature relatively slowly. It is also important that the probe fit snugly into the block, so thermal “wells” of many different sizes are often required when calibrating a variety of probes. Typical temperature ranges of block calibrators are -40°F to 1200°F (-40°C to 648°C).

The circulating bath simply circulates a temperature-controlled fluid in a bath into which the probe is inserted. This type of calibrator is the least expensive of the three, but has a relatively limited temperature range, typically -5 to 266°F (-20 to 130°C). More exotic and expensive baths can extend this range to -250 to 1170°F (-160 to 630°C).

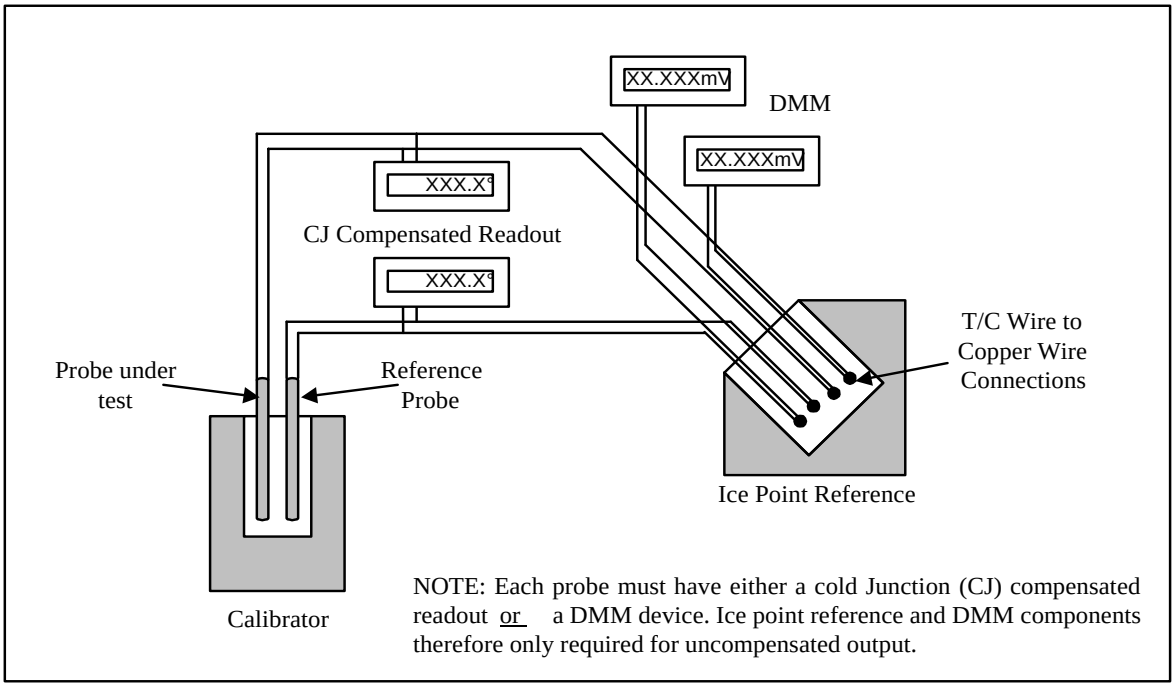


FIGURE 6.4.5.9 *Typical calibration setup.*

Fluidized powder bath calibrators use a gas, usually low-pressure air or nitrogen, to fluidize dry particles of powder – typically aluminum oxide. These baths have excellent heat transfer characteristics and are clean and easier to maintain than a circulating bath. They also have a significantly higher temperature range, though they are generally not capable of cryogenic temperatures. Common temperature ranges are from 122°F to 1112°F (50°C to 600°C). Extended range powder baths are available from -100 to 1800°F (-70 to 980°C).

The Reference Standard Probe

Reference standard probes are simply temperature probes that are calibrated and traceable to NIST. Obviously the calibration tolerance of the reference probe must be taken into account in the final tolerance of the probe being calibrated. When calibrating a thermocouple probe without an attached readout instrument, it is important that the reference standard be of the same thermocouple type as the probe being calibrated. This insures that both probes behave identically at the T_2 ice point reference. When a system calibration is being performed, this is not essential because the instrument connected to the probe will provide independent cold junction compensation. Similarly, if the reference standard is not a standalone probe, but is a calibrated system consisting of a reference probe and a readout device that performs cold junction compensation, the probes need not be of the same type.

All temperature measurement devices have limited temperature ranges over which their response is well behaved. It is therefore essential to verify that the reference standard probe is well behaved and calibrated over the full range of its use.

The Readout Device

Depending on the type of reference standard used, and the probe being calibrated, the readout device can vary considerably. If using reference standards or calibrating temperature probes without a readout device that performs scaling and/or cold junction compensation, the recommended readout device is a 5½ digit digital multimeter (DMM). This precision instrument allows the thermocouple output to be read

to within 0.001 millivolt, which is the precision to which the NIST thermocouple reference tables are published.

If a self-scaling output device is used, its precision (and verified accuracy) must be sufficient to provide at least four times the accuracy desired from the calibration.

The Ice Point Reference

The ice point reference is used to bring the T_2 junction to its standard value of 32°F (0°C) during calibration of the probe. Again, this is only necessary if either the calibration standard or the probe being calibrated does not have another form of cold junction compensation. Ice point reference chambers are often simply a well-controlled and monitored ice bath. Electronic ice points are also available which greatly simplify the setup. It is important to note that the wiring from the T_2 end of the thermocouple (which is at 32°F (0°C) in the ice point reference) to the readout device should be exclusively copper. This ensures that the emf response of the assembly is as-assumed by the thermocouple reference tables.

6.4.6 Data acquisition systems

This section is reserved for future use.

6.5 TESTING ENVIRONMENTS

This section is reserved for future use.

6.5.1 Introduction

This section is reserved for future use.

6.5.2 Laboratory ambient test environment

This section is reserved for future use.

6.5.3 Non-ambient testing environment

6.5.3.1 Introduction

Composite materials can be affected by exposure to non-laboratory ambient environmental conditions and so must be tested to determine those effects. Below laboratory ambient conditions as well as above laboratory ambient conditions must be included in the test matrix to determine each effects. Guidelines for the above and below laboratory ambient test conditions are included below. Many different regimes of testing may be appropriate depending on the usage of the materials. Normal environmental conditions for terrestrial applications would be from as cold as -67°F (-55°C) and up to 350°F (180°C). Conditions in space would widen the band of performance interest from -250°F to 450°F (-160°C to 230°C). Cryogenic conditions (less than -250°F (-160°C)) may be of interest for storage tank usage. Special conditions may dictate the usage of composite materials up to and beyond the short duration limit of 600°F (315°C) around leading edges or engine components. The user must determine what the limits for their particular application may be to allow for proper non-laboratory ambient testing to be completed on the materials used in the application.

The purpose of this section is to give the user some guidance in the testing of materials under other than standard laboratory conditions. Both below and above room temperature test conditions are discussed below. Further guidance related to non-laboratory ambient testing can be received from SACMA SRM 11R-94, Recommended Method for Environmental Conditioning of Composite Laminates.

6.5.3.2 Subambient testing

Testing performed at below laboratory ambient test temperatures can present unique challenges. Special fixturing or lubrication may be needed to ensure that properties measured are material behavior related and not due to freezing or sticking of sliding surfaces. Materials can become more brittle and change their failure modes. Special instrumentation may be necessary to record material properties at the colder temperatures. Adhesives used for tabbing or strain gaging should be types that retain their elongation at the cold temperatures.

Test temperatures as cold as -67°F (-55°C) are common and are discussed here. The test setup in a test chamber must be precooled until stabilized at test temperature. Fixturing should be allowed to stabilize prior to testing. Cooling medium may be liquid nitrogen (LN_2), liquid carbon dioxide (LCO_2), or a refrigerated chamber. Temperature measurements are commonly made with J, K or T type thermocouples (T/C's). See Section 6.4.5 for more information on temperature measurement. A dummy test specimen should be used to determine soak times prior to actual testing. The dummy specimen should be fabricated using the same material and ply orientation as the test specimen. To determine the soak time, a T/C should be inserted into a hole drilled at the centerline of the dummy specimen. Record the time it takes to reach the desired test temperature. This time should be used when testing to regulate when the test specimens are at the appropriate test temperature . Cool down rates should be controlled to minimize thermal shock and possibility of damage and/or microcracking.

Freezing of test fixtures can be a cause of anomalous test results. Fixture clearances must be checked to ensure free sliding surfaces exist. Proper lubricants or no lubricants should be used at the cold temperatures to prevent any fixture related effects on the test results.

A thermocouple (T/C) should be placed in contact with the surface of the test specimen at the time of test. A typical soak time of 5-10 minutes, or the time determined from actual experimentation, should be used, after reaching test temperature. Appropriate safety equipment should always be worn to prevent cold burns. Care must be taken if using LN_2 or LCO_2 when cooling the chamber to ensure that room oxygen is not depleted.

6.5.3.3 Above ambient testing

Testing performed at above ambient temperatures must be done with consideration for the temperature and moisture content of the test sample. Special fixturing may be needed to accommodate the high temperatures. The possibility for adhesive failures and drying of test specimens should be evaluated before proceeding with a test program. Special lubricants may be required to prevent fixturing from sticking or binding. Instrumentation made especially for the required temperatures must be used to ensure valid data is recorded. Strain gages, extensometers, and adhesives with the correct temperature rating must be identified and used. Special strain gage foils or backing materials may be required to withstand the elevated temperatures during testing. Instrumentation may require additional calibration at test temperatures.

The above ambient test temperatures, to 350°F (180°C), are discussed here. The test setup in a test chamber must be heated until stabilized at test temperature. Fixturing should be allowed to stabilize prior to testing. Heating of the test fixture with specimen or only the specimen is usually accomplished with an electrically heated chamber. Temperature measurements are commonly made using J, K, or T type thermocouples (T/C's). A dummy test specimen should be used to determine soak times prior to actual testing. The dummy specimen should be fabricated using the same material and ply orientation as the test specimen. To determine the soak time, a T/C should be inserted into a hole drilled at the centerline of the dummy specimen. Record the time it takes to reach the desired test temperature. This time should be used when testing to regulate when the test specimens are at the appropriate test temperature. Heat up rates should be controlled to minimize thermal shock and possibility of damage and/or microcracking.

Excessive heat up rates may cause charring or melting of test specimens or adhesives. An appropriate lubricant, such as molybdenum disulfide, should be used on sliding surfaces to ensure freedom of movement of test fixtures.

A T/C should be placed in contact with the surface of the test specimen prior to testing. A standard soak time would be 5-10 minutes, after reaching test temperature, if the test condition is dry. A standard soak time would be 2 minutes, after reaching test temperature, if the test condition is wet, to prevent too much dryout of the test specimen.

If moisture content is a testing variable, then the dryout of the test specimen, unless humidity is controlled during test, should be evaluated by weighing a traveler before and after a specimen soak time and test. See Section 6.3 for moisture conditioning guidelines. Appropriate safety equipment should always be worn to prevent burns.

For moderate test conditions, i.e., less than 200°F (93°C), a humidity controlled test chamber is optional for short duration tests. When testing above 200°F (93°C), then a precise humidity control is impractical and specimen dryout is a concern, especially for fatigue testing. Soak times prior to test should be kept short (<3 min.) to minimize the dryout.

Testing performed at temperatures above 350°F (180°C) must use special strain gages and strain gage adhesives, extensometry, and fixturing designed for the elevated temperatures. Special high temperature capable tab materials and tab adhesives will need to be utilized to prevent tab failures. Usage of these materials may be inappropriate at other temperatures.

Thermocouples are the most common transducer for measuring temperatures. Various T/C types may be used but J, K, and T are the most common. Some special conditions may dictate the use of resistance temperature detectors (RTD's). See Section 6.4.5 for more information on temperature measurement.

6.6 THERMAL/PHYSICAL PROPERTY TESTS

The physical analysis methods for laminae and laminates provide information on the integrity of the fabricated composite. Thermal analysis methods are used to determine the glass transition and crystalline melt temperatures, coefficient of thermal expansion, and residual heat of reaction. Additional analytical methods discussed in the following sections are used to determine fiber volume, void volume, density, dimensional stability, and moisture weight gain.

6.6.1 Introduction

The thermal analytical techniques described in Chapter 4, Section 4.5.2 may also be used to evaluate composite materials. Information obtained from thermal analysis includes the glass transition temperature, crystalline melt temperature, expansion/contraction properties, thermal stability, and extent of cure for thermosets.

6.6.2 Extent of cure

Characterization of extent of cure of composite materials has become increasingly important as controlled staging of complex or thick parts has been implemented as part of advanced processing schemes. Debulking and staging of stiffeners or other structural details can be used to facilitate assembly of large complex parts, with the ultimate goal of allowing out-of-autoclave processing. Debulking and staging are also a critically important aspect of the fabrication of thick parts to prevent resin migration and fiber waviness.

Several different thermal analysis techniques are commonly used for extent of cure measurements in fiber reinforced organic matrix composites. There include differential scanning calorimetry (DSC) or dy-

namic thermal analysis (DTA) to measure the extent of the residual curing exotherm and dynamic mechanical analysis (DMA) or thermomechanical analysis (TMA) to measure the glass transition temperature. Measurement of Tg is discussed in some detail in Section 6.6.3 below.

6.6.3 Glass transition temperature

6.6.3.1 Overview

The glass transition of a polymer matrix composite is a temperature-induced change in the matrix material from the glassy to the rubbery state during heating, or from a rubber to a glass during cooling. A change in matrix stiffness of two to three orders of magnitude occurs during the glass transition, due to the onset or freezing out of long range molecular mobility of the polymer chains. The temperature at which the glass transition occurs is a function of the molecular architecture and crosslink density of the polymer chains, but it is also dependent on the heating or cooling rate used in the measurement, and on test frequency if a dynamic mechanical technique is employed. In addition to the change in stiffness, the glass transition is marked by a change in the heat capacity and the coefficient of thermal expansion of the material, and so has at least some characteristics of a second order thermodynamic transition (see Reference 6.6.3.1).

The glass transition is frequently characterized by a glass transition temperature (Tg), but since the transition often occurs over a broad temperature range, the use of a single temperature to characterize it may give rise to some confusion. The experimental technique used to obtain the T_g must be described in detail, especially temperature scanning rate and frequency used. The method by which T_g is calculated from the data must also be clearly stated. Reported T_g may reflect onset of the glass transition or midpoint temperature depending on the data reduction method.

Upon exposure to high humidity environments, polymer matrices will absorb environmental moisture and be plasticized by it. One effect of this plasticization is the depression of T_g, frequently by a significant amount. A highly crosslinked resin (one based for instance on a tetrafunctional epoxide such as TGMDA) may have a high initial T_g, but it may be depressed more strongly than that in a less highly crosslinked system. Measurement of the T_g in a composite material plasticized by absorbed moisture poses some difficult experimental challenges. Heating the test specimen as required by the measurement will drive off at least some of the absorbed moisture, thereby affecting the measured properties.

Due to the decrease in matrix stiffness that occurs at the glass transition and to the low strength of these polymer matrices in the rubbery state, the matrix can no longer function effectively to transfer load to the fibers or suppress fiber buckling above the glass transition. T_g is, therefore, frequently used to define the upper use temperature of a composite material, although the time-dependent properties of the material such as creep compliance may be more sensitive to temperature within the glass transition range than are the quasi-static mechanical properties. A safety margin of 50F° (28C°) between the T_g and the material operational limit (MOL) has been proposed for epoxy matrix composites (see Section 2.2.8). This approach is useful for initially estimating the MOL, or for verifying a previously chosen MOL. However, since glass transition frequently occurs over a temperature range, and the measured value of T_g is highly dependent on method, supplemental mechanical property tests should be considered, particularly for new material systems (see Section 2.2.8).

6.6.3.2 T_g Measurements

Several different methods have been used to characterize the glass transition in polymeric materials, and most of these are also applicable to fiber reinforced materials.

6.6.3.2.1 Differential scanning calorimetry (DSC)

Since the heat capacity of a composite material changes at the glass transition, differential scanning calorimetry (DSC) may be used to determine T_g. The glass transition is detected as a shift in the heat flow versus temperature curve (see Figure 6.6.3.2.1). Many calorimeters are supplied with software

which may be used to calculated T_g. T_g of neat resin specimens is relatively easy to detect with DSC, but in composite specimens the resin content in the specimen is small, and the more highly crosslinked the resin, the smaller the change in heat capacity. It is, therefore, sometimes difficult to detect T_g in highly crosslinked cured composites (see Reference 6.6.3.2.1).

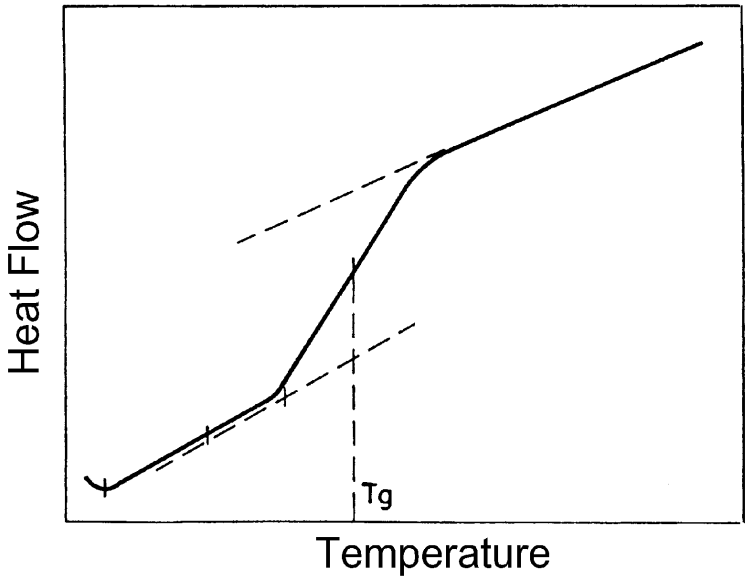


FIGURE 6.6.3.2.1 *Differential scanning calorimetry (DSC).*

6.6.3.2.2 Thermomechanical analysis (TMA)

Thermomechanical techniques such as expansion, flexure, or penetration thermomechanical analysis (TMA) may also be used to determine T_g. In expansion TMA, the coefficient of thermal expansion α is measured as a function of temperature. As noted above, α undergoes a change during the glass transition, and T_g is determined by the point of intersection of lines fit to the thermal expansion data above and below the glass transition range. Figure 6.6.3.2.2 illustrates the specimen geometries and data reduction methods used for various TMA techniques.

In flexural TMA, a rectangular specimen is loaded in bending and the dimensional change is measured as a function of temperature. A curve fitting technique as illustrated in Figure 6.6.3.2.2 is used to calculate T_g. Flexural TMA measurement of T_g is similar to heat distortion temperature (HDT) measurement, since in both cases the specimens are loaded in flexure. An HDT specimen may be a full-size flexural test specimen, and is loaded in three-point bending or as a cantilever beam. Displacement is measured as a function of temperature, and the HDT is the temperature at which the displacement reaches some predetermined value. Use of a full-size specimen minimizes moisture loss during the HDT test, but flexural TMA and HDT measurement share the disadvantage that values of T_g or HDT obtained will be sensitive to the modulus of the reinforcing fibers in the composite sample and they will give different results depending on the nature of the fiber.

As shown in Figure 6.6.3.2.2, penetration mode TMA measures the hardness of the material. One disadvantage of this technique is that if the probe is touching a reinforcing fiber, an accurate measurement of the T_g of the matrix will not be obtained.

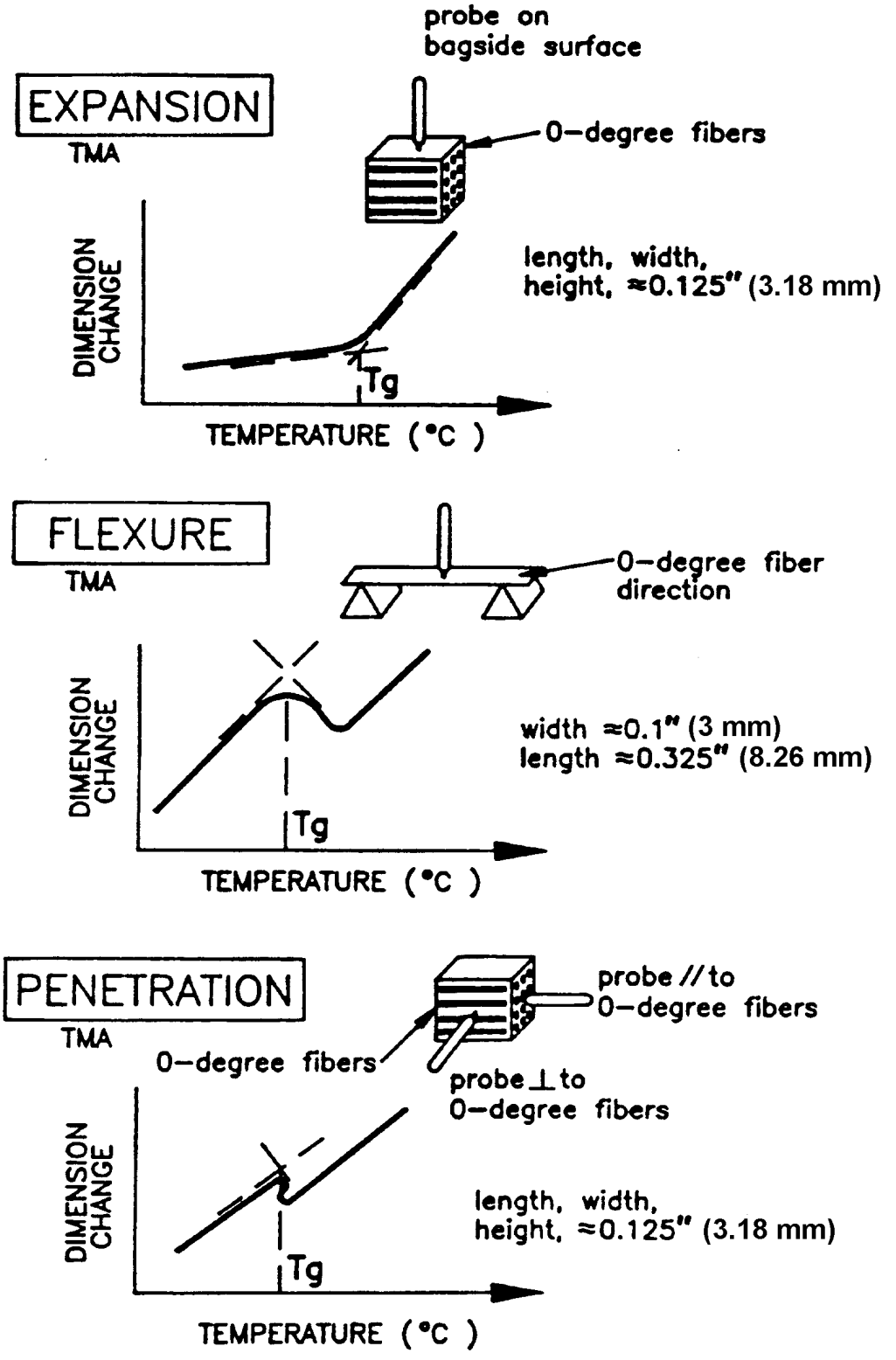


FIGURE 6.6.3.2.2 *Thermomechanical Analysis (TMA) - typical dimensions shown.*

6.6.3.2.3 Dynamic mechanical analysis (DMA)

Dynamic mechanical analysis (DMA) is the most common and preferred method of characterizing the glass transition of organic matrix composites. There are several types of DMA which have been used with composites, including torsion pendulum analysis (TPA) and other resonant techniques, and forced oscillation measurements in tension, torsion, and shear. These forced measurements are made using a number of DMA instruments, manufactured by DuPont, Perkin Elmer, Polymer Laboratory, Rheometrics, TA Instruments, and others.

All these DMA techniques produce curves of dynamic storage and loss modulus and loss tangent ($\tan \delta$) or log decrement (Λ) as a function of temperature (see Figure 6.6.3.2.3(a)). Tan δ and Λ are proportional to the ratio of the loss modulus (E'' or G'') to the storage modulus (E' or G'). They reflect the amount of energy dissipated during each cycle of loading, and go through a peak value during the glass transition. T_g may be determined from DMA data in several different ways, and this may be a source of differences in reported values for T_g. As shown in Figure 6.6.3.2.3(a), T_g may be determined as the temperature at the onset or the midpoint of the transition based on the storage modulus curve, at the maximum in $\tan \delta$, or at the maximum in loss modulus. Clearly the method used for calculating T_g could produce markedly different values for the same set of DMA data. The temperature scanning rate and frequency employed will also affect the results, as discussed above.

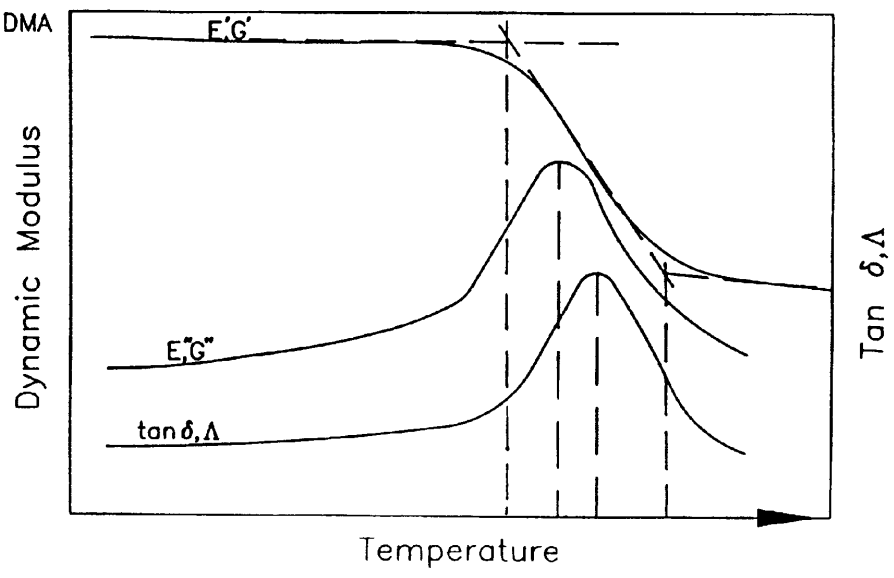


FIGURE 6.6.3.2.3(a) *Dynamic mechanical analysis (DMA).*

An ASTM standard (D 4065) is available for DMA of plastics, covering both forced and resonant techniques (Reference 6.6.3.2.3(a)). The test techniques described in this standard practice are the same as those used for fiber reinforced plastics. In addition, a newly released SACMA method (SRM 18R-94) recommends the use of DMA for the measurement of T_g in oriented fiber-resin composites (Reference 6.6.3.2.3(b)). SACMA SRM 18R-94 specifies a forced oscillation measurement at 1 Hz, a heating rate of 5C° (9F°) per minute, and calculation of an onset T_g from the dynamic storage modulus curve. If a consistent material operational limit (MOL) is to be calculated from T_g, standards for these experimental vari-

ables should be specified along with a temperature safety margin. Otherwise the measured T_g may be shifted by increasing or decreasing heating rate or frequency.

As discussed above, measurement of T_g in a wet composite material is made more difficult by the drying which occurs as the specimen is heated. Techniques which seek to prevent this drying by sealing the specimen in some way may be helpful in slowing the weight loss, but it cannot be prevented completely. If the specimen is sufficiently thick, the drying will occur primarily at the outside surface, resulting in a broadened or even bimodal glass transition (see Figure 6.6.3.2.3(b)). The lower temperature region will reflect the T_g of the interior of the specimen which is still wet, and the higher temperature region will reflect the T_g of the dried material. The loss tangent or log decrement curve will be broadened, or will exhibit two peaks or a peak and a shoulder, with the relative peak heights indicating the amounts of wet and dried material present in the specimen. In measuring T_g of a wet specimen, the lower temperature part of the transition may be the region of interest, suggesting that calculation of an onset T_g would be the appropriate and conservative approach.

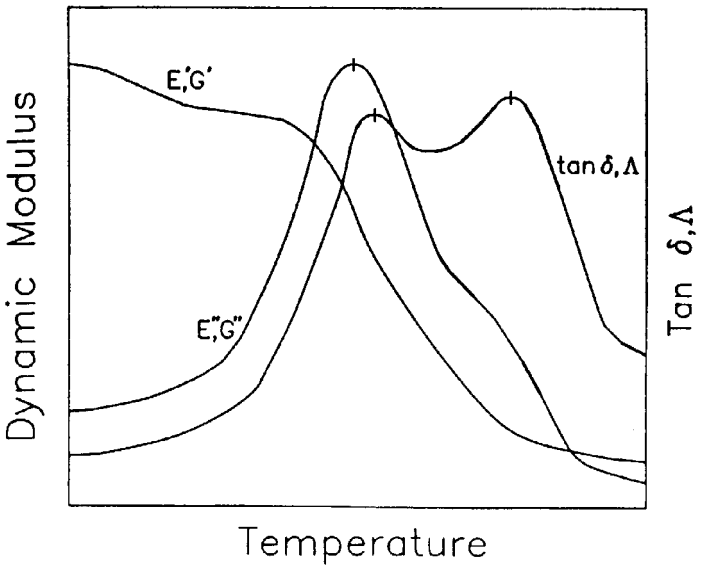


FIGURE 6.6.3.2.3(b) *DMA of a wet composite material.*

6.6.3.3 Glass transition test methods for MIL-HDBK-17 data submittal

Data generated by DMA as described above are currently being accepted by MIL-HDBK-17 for consideration for inclusion in Volume 2. In addition to the specific apparatus used for the measurement, the heating rate and frequency must be included, and the method used to calculate T_g from the data must be specified. If a resonant method such as torsion pendulum is used, the frequency in the glassy region should be included with the data.

6.6.3.4 Crystalline melt temperature

The crystalline melt temperature (T_m) of semi-crystalline thermoplastic composites can be obtained from DSC or DTA experiments. In addition, an estimate of the degree of crystallinity can be made. This becomes an important parameter since the properties of semi-crystalline thermoplastic composites may be dependent upon the degree of crystallinity of the matrix resin. The heating required for processing prepregs into composite structures may have an affect on the degree of crystallinity as well as the crystal structure.

6.6.4 Density

6.6.4.1 Overview

The density of composite materials is useful both directly, as for estimation of bulk weight or for thermal or dynamic analysis, as well as indirectly, as in derivation of quantities based on other measurements, like thermal conductivity (with specific heat and diffusivity) and void volume (with fiber and resin density). The application will determine the optimum test method, each of which have different levels of precision and bias (see Section 2.2.4) as well as different levels of ease of use. The last application mentioned, void volume determination of composites, probably has the most demanding density determination requirements. In order to accurately determine void volume to within 0.5% or better the density of the composite and the constituents must be known to within about 0.005 g/cm^3 (1.8x10^{-4} lb/in^3) or better.

Density can be measured directly or calculated from separate volume and mass measurements. From these two approaches the focus will be on the three main density test methods in current use, which are: 1) Archimedes volume determination by liquid (most often water) displacement, as standardized by ASTM D 792 (Reference 6.6.4.1(a)); 2) direct measurement of density by observation of the level at which the test material is suspended in a density gradient column, as standardized by ASTM D 1505 (Reference 6.6.4.1(b)); and 3) measurement of test specimen volume by pressure changes of a known amount of an inert ideal gas (helium pycnometry), as standardized by ASTM D 4892 (Reference 6.6.4.1(c)) for a non-composite material and modified by MIL-HDBK-17 for use with composites as described in Section 6.6.4.4.1.[1]

While all three test methods provide generally accurate density values, the helium pycnometry method has not been demonstrated to be sufficiently accurate for use in determining void volume (see evaluation results in Section 6.6.4.5), although this may change in time with future modifications to procedure and instrumentation. For typical applications in composites, the Archimedes method, as described in ASTM D 792 and modified below, is preferred for its low cost, relative simplicity, and high accuracy (when properly performed). While also accurate, the density-gradient technique is less desirable due to the high labor cost and low through-put (since it may take several hours for one determination to stabilize in the column). Also, the long exposure of the specimen to the column fluid may not be desirable for subsequent procedures like matrix digestion. Each of the test methods is described in detail in the sections below.

Liquids are used almost exclusively in displacement techniques. However, there are advantages to using a gas medium in place of liquid to determine specimen volume. One advantage is minimization of errors associated with liquid surface tension. The gas displacement approach is often referred to as helium pycnometry. When helium pycnometry is used, the test specimen volume is determined by measuring pressure changes of a confined amount of gas. Helium pycnometry is not yet a standardized test method for measuring the volume and density of composites, yet it has been demonstrated to be a viable technique (References 6.6.4.1(e) and (f)). As no test standard or guidelines exist for this method as applied to composites, a test procedure has been developed within the MIL-HDBK-17 Testing Working Group. The procedure has been included as Section 6.6.4.4.1. This procedure will remain in effect until such time as a standard method for composites is adopted by ASTM or another standard-certifying organization.

6.6.4.2 ASTM D 792, Standard Test Method for Density and Specific Gravity (Relative Density) of Plastics by Displacement

Density of composites is frequently measured by means of ASTM D 792 which is under the jurisdiction of ASTM Committee D-20 on Plastics. This standard actually describes a means of measuring specimen volume and combines that value with a weight measurement to calculate density. Obtaining an accurate volume measurement is the key to a reliable composite density value.

[1]For a quick and convenient, but less accurate density determination method, the reader is referred to the micrometer technique as specified in ASTM D2734, Test Method C (Reference 6.6.4.1(d)). This method obtains specimen volume by simple dimensional measurement and is only appropriate for precision work in limited specific cases.

The method is based on the weight of a specimen in air compared to its weight while completely immersed in a liquid of known density, most often water. When using water as the medium it must be degassed and either deionized or distilled for high accuracy work. A close eye must be kept for nucleated gas bubbles which are most likely to appear on rough surfaces such as machined edges. Also, the machined surfaces are usually more porous and may not wet completely. Close scrutiny of these surfaces is recommended to verify that no visible microbubbles are present within surface cavities. If microbubbles are present, switch to a higher wetting liquid or add a surfactant (for example, add four drops of Cole-Palmer 8790 Micro-Lab Cleaning Solution per 200 ml of water) for optimum results.

In general, the bigger the specimen the better. As specimen size and weight get closer to zero, the measurement limits on volume and weight begin to affect the density value. The recommended minimum test specimen is 1 gram (for carbon/epoxy this is about 0.037 in^3 (0.6 cm^3), for glass/epoxy it is about (0.024 in^3 0.4 cm^3). A balance accuracy of 0.0001 g ($2.2x10^{-7}$ lb) is required for precision work on a specimen of this size.

The D 792 test method states room temperature testing should be conducted after conditioning the specimens for at least 40 hours at the standard laboratory atmosphere of 73.4 ±3.6°F (23 ±2°C) with 50 ±5% relative humidity. If the test is being performed to referee a disagreement, the tolerances are ±1.8F° (±1C°), and ±2% relative humidity. For cases where immersion liquids other than water are being used, the temperature tolerance is ±0.9F° (±0.5C°). For an improvement in precision, the ASTM D-30 committee recommends bringing the material to oven-dry equilibrium to determine the initial weight (References 6.6.4.2(a) and (b)).

Two techniques are offered: Test Method A, employing water as the immersion liquid; and Test Method B using a liquid other than water, such as kerosene. Test Method B is often used when the specimens are either lighter than water, or when water will cause the specimens to undergo physical changes such as swelling.

Advantages and Limitations of ASTM Test Method D 792

The main advantages to choosing this method are practicality and precision (when carefully performed), and it is by far the most frequently used method. Equipment needed is simple and inexpensive, with the exception of a quality analytical balance. With careful technique, accuracy within ±0.005 g/cm^3 ($±1.8x10^{-4}$ lb/in^3) is typically obtained. The apparatus includes a balance, a bridge for spanning the balance pan, a wire or filament, beaker, tweezers, thermometer, water or other liquid, and perhaps some sinking weights if the specimen is less dense than the liquid.

After a time investment of about a day for practicing the technique so that reproducible results can be obtained, the tests can begin. Four to six specimens per hour can typically be tested using D 792, however, there is a certain amount of tedium associated with this technique and actual through-put will likely be lower.

As with all the density methods mentioned here, it is good practice to take care in specimen preparation, especially edge quality. Care must be exercised in cutting specimens to avoid density changes. Other issues to be mindful of are specimen size, surface wetting (some experimenters add trace amounts of surfactants to the water), liquid absorption during measurement, and water temperature (vacuum degassing will cool the water, slightly changing its density). D 792 relies on a liquid medium, and problems with entrapped and/or clinging gas bubbles are a concern. High wetting, degassed liquids are required for optimum results.

6.6.4.3 ASTM D 1505, Standard Test Method for Density of Plastics by the Density-Gradient Technique

ASTM D 1505 is significantly different from D 792 in that specimen density is measured directly; no calculations are used. It determines density by floating the test specimen in a glass column containing a liquid mixture of known varied density. Composite specimens work well with this technique as long as the liquid medium selected is inert to the specimens. The test is useful for tracking materials that undergo

physical changes over time, checking uniformity, and identifying materials. It has also been reported to be more precise, and likely, more accurate than the D 792 method (Reference 6.6.4.3).

The method uses two approaches: an incremental varied-density liquid column (Test Method A), and two continuous varied-density liquid columns, Test Method B, progressively less dense liquid, and Test Method C, progressively more dense liquid. When the column is filled correctly the gradient remains remarkably stable and linear. The densities of the starting liquids are first closely approximated using a calibrated volumetric canister (liquid pycnometer). Calibrated sink floats are used to determine the linear variation of liquid density with the height of the column. By noting the level at which the specimen floats, the density of the specimen can be matched to the known density of the column liquid at that height. The accuracy and precision of this test are set by the sink floats along with a highly linear density variation of the column liquid vs. height.

Advantages and Limitations of the ASTM Test Method D 1505

Equipment for this method can be purchased commercially for a few thousand dollars. This includes an assortment of column fluids and sink floats. Alternatively, the apparatus can be assembled from standard lab glassware and components for minimal cost, but expect to spend time on machining and assembly before any testing can be done.

The procedure for filling the column to make a linear gradient is best described as “artful.” Expect to invest several days to a week learning this portion of the procedure. The column sensitivity is under the control of the experimenter. A skilled experimenter can make columns that are in the range of $0.001 g/cm^3/cm$ (9.2×10^{-5} $lb/in^3/in$). Column sensitivity can be adjusted up or down to match the test need. Sensitivity is set by the density difference of the starting liquids.

Once the column is ready, specimens should be carefully introduced at the top with tweezers. To avoid gas bubbles clinging to the specimen it is helpful to set aside a small amount of the liquid mixture to pre-wet the specimens. As with D 792, if bubbles are present they can often be seen through the glass and transparent liquid, although there is not much that can be done to rectify this in the case of D 1505: once a specimen is immersed it is extremely difficult to retrieve without destroying the gradient.

The D 1505 test itself is rather time-consuming. The column must be filled slowly and carefully to preserve the gradient and typically takes several hours. Once the specimens are immersed it takes time for them to settle to their equilibrated height in the column. If many specimens need to be measured, one column will not be able to handle them all, so several gradient columns will have to be set up, broken down, and refilled.

As with all the density methods mentioned here, care must be exercised in cutting specimens to avoid density changes. The concerns with using a liquid medium are the same as those mentioned for D 792 in the last paragraph of the above Section 6.6.4.2.

6.6.4.4 Use of helium pycnometry to determine density of composites

Technological advances in electronics and automation have made it possible to obtain accurate and reliable volume determinations of composites (as well as fibers and matrix resins) using a gas in place of liquids. Helium pycnometry is a method for measuring the volume of solids of all types, including powders and open and closed cell materials. This flexibility is made possible because the medium used is an inert ideal gas, usually helium, which will penetrate the tiniest pores. High purity helium is the most commonly recommended gas because it is completely inert. High purity nitrogen is a good alternative to helium.

Helium pycnometry is not a new technology. Pycnometers, using both liquids and gases, have been used in ASTM test standards for many years. However, it was not until fairly recently that helium pycnometers became sophisticated enough to be used for high accuracy volume determinations. ASTM adopted standard D 4892 (Reference 6.6.4.4) in 1989, which uses a helium pycnometer to determine the volume/density of a non-composite material to three decimal places.

Interest in using helium pycnometry to measure composite density stems from its potential to be both as accurate and precise as D 1505 while at the same time having a higher through-put and better ease of use than D 792. Another distinct advantage of using a gas medium is that it guarantees reproducible penetration into surface pores. With a liquid medium the experimenter has no way of knowing what percentage of the surface porosity is left unfilled.

In helium pycnometry the volume of solid objects is measured by employing Boyle's Law, which states that the decrease in volume of a confined gas results in a proportionate increase in pressure. Both helium and diatomic nitrogen are used because they behave as ideal gases at room temperature. A helium pycnometer makes use of this by making two precise pressure measurements on two known calibrated volumes. These are the cell volume and the cell volume plus a smaller expansion volume usually called the "added volume." The first pressure is measured with all gas confined in the main measurement cell where the test material is located. After this pressure is determined a valve is opened connecting the main and expansion cells together. This results in a second, lower pressure which is recorded. Using the ideal gas law the volume of the test material in the main cell is determined using the equation below in Section 6.6.4.4.1.

Advantages and Limitations of the Helium Pycnometry Procedure

A distinct advantage of using a gas medium is that it guarantees reproducible penetration into surface pores. With a liquid medium the experimenter has no way of knowing what percentage of the surface porosity is left unfilled.

The accuracy of this method begins to drop off when the ratio of the sample volume to test cell volume (V_s/V_c) is low. Experiments have shown that if this ratio is approximately 30% or higher the pycnometer will be near its optimal performance (References 6.6.4.1(e) and (f)). This is not to say that useful data can not be obtained when the ratio is below 30%. The precision and bias remain quite useful to low ratios below 1% (References 6.6.4.1(e) and (f)). The ratio needed for a particular test is determined by the accuracy needed; for example, if one significant digit is sought, then there would be no restriction on the V_s/V_c ratio. However, if three digits are needed the V_s/V_c ratio becomes important and should be 0.3 or higher.

The gas pressure is sensitive to temperature. Tests were run to detect temperature fluctuation in the measurement cell. It was found that no matter what cell volume was used, the internal temperature of the measurement cell was extremely stable, despite the constant influx and expansion of fresh helium (Reference 6.6.4.1(f)).

The shape of the test specimen can cause problems during insertion into the measurement cell. The cells of off-the-shelf pycnometers sold today are usually cylindrical. If the test specimen is restricted to some rectangular shape it may be difficult to get more than 30% of the cell volume filled due to geometric incompatibility. Commercially available pycnometers are like this because, although capable of working with any solid object, they are designed for their most popular application, which is powders. This problem can be remedied by cutting the composite specimen to match the cell geometry, for example, stacked disk-shaped specimens.

A quality helium pycnometer, such as the one used in this work, is a sophisticated piece of analytical equipment with a base cost in the $10,000 range. Much like a lab analytical balance, once the equipment is purchased very modest costs are associated with the test thereafter. Expect to spend several days getting familiar with the equipment and procedures.

Pycnometry has an advantage in that much less labor is involved, as the equipment is automated. Aside from sample preparation and conditioning, the experimenter needs to change specimens at the end of each run. Once running, the pycnometer gathers data at a rate of 25 to 30 measurements per hour. An important aspect of automation is that it significantly reduces variations introduced by operator-to-operator skill and "artfulness" which enter into the test results.

As with all the density methods mentioned here, care must be exercised in cutting specimens to avoid density changes.

6.6.4.4.1 Helium pycnometry test procedure for determining composite density

Helium pycnometry is not a formally recognized method for measuring composite density. As no test standard or guidelines exist for this method, a procedural guideline is included here. This procedure will remain in effect until such time as a standard test method is adopted.

Background

Volume and density of solid objects is measured by employing Boyle's Law, which states that the decrease in volume of a confined gas results in a proportionate increase in pressure. Both helium and diatomic nitrogen behave as ideal gases at room temperature. As noted above, the pycnometer makes use of this by making two precise pressure measurements on two known volumes. These are the cell volume and the cell volume plus a smaller expansion volume usually called the "added volume." The working equation used to calculate the test specimen volume is

$$V_s = V_c + \frac{V_a}{1-(P_1 / P_2)} \qquad 6.6.4.4.1$$

where

V_s = specimen volume

V_c = previously calibrated empty specimen cell volume

V_a = previously calibrated added volume

P_1 = pressure when all gas is confined to the main cell

P_2 = second pressure when the gas fills both cells

Before running the pycnometer, V_a and V_c are calibrated by using a volume calibration standard. Assumptions made are that the cell temperature is constant, the two cell volumes are constant, and the moles of gas present is constant. The specimen density is obtained by supplying the pycnometer with a weight measurement of the test material from an analytical balance.

Testing has shown that when the V_s/V_c ratio is approximately 30% or higher the pycnometer will operate near optimal performance. This is not to say that reliable data can not be obtained when the ratio is below 30%, but rather, some loss in performance begins to occur.

It is important to note that the gas medium guarantees filling the tiniest of surface pores, which other liquid immersion methods may or may not do, due to surface tension. This should be kept in mind when comparisons are made. When surface pores are not filled the density data is shifted to some lower value. Therefore, liquid immersion methods may return density data that is biased slightly to the low side when compared to data from the helium pycnometry method. This density shift may or may not be noticeable, depending on such factors as the amount of surface porosity and the wetout between the liquid and the composite.

Apparatus

Helium pycnometer (the pycnometers specified in ASTM D 4892, Footnote 4 are acceptable for use here); volume calibration standard; high purity helium or nitrogen gas cylinder with regulator; analytical balance; desiccator; disposable plastic gloves or tweezers (for specimen handling).

Specimen Preparation

Heat and pressure from the specimen cutting process can locally alter the specimen density. Composite specimens should be sanded with fine grit paper and wiped clean of any remaining loose dust. Specimen shape is irrelevant to the test, but consideration of specimen geometry is required in order to ensure enough material gets into the measurement cell. The recommended shape for a cylindrical cell is

a circular specimen with a diameter nominally 0.080 inches (2.0 mm) less than the cell diameter. Specimen diameter can be larger, but not so large that there is a risk of jamming against the cell walls. The disks should be stacked to fill as much of the cell as possible. If significantly lower precision is acceptable, there is no need to consider what percentage of the cell volume is filled (unless the specimen volume is extremely small). Useful data will result even when V_s/V_c is near 1% (References 6.6.4.1(e) and (f)).

Procedure

1. Precondition the test specimens according to ASTM D 618 "Conditioning Plastics and Electrical Insulating Materials for Testing" (Reference 6.6.4.4.1(a)) or, for improved accuracy, bring the specimen to oven-dry equilibrium as recommended by References 6.6.4.2(a) and described by 6.6.4.2(b). Store the test specimens in a desiccator at 73.4°F (23°C) until they are ready to test.

2. In general, follow the manufacturer's instructions to determine the composite density. A list of, and comments on, the test steps is given here.

- The helium (or nitrogen) source should be connected to the pycnometer gas input via a gas-tight pressure fitting.

- The printer and computer (if so equipped) should be connected to the data output interface via an appropriate cable.

- After the pycnometer power is turned on, let it warm up to its equilibrium operating temperature, which is typically 3.6 to 5.4F° (2 to 3C°) above ambient temperature.

- If the pycnometer has not been calibrated, run the calibration procedure specified in the user's manual. From time to time the pycnometer should be recalibrated, especially if the ambient temperature has changed by an appreciable amount or if it fluctuates. The pycnometer is only as good as the calibration standards used to calibrate it. Be sure the standards used meet proper specification. If the accuracy of the standards is in doubt they can be spot-checked using a test such as D 792. Certified standards can be obtained through the National Institute of Standards and Technology (Reference 6.6.4.4.1(b)).

- Once calibrated, the pycnometer is ready to run. Remove the sealed cap, opening the main cell to the ambient air. Place the pre-weighed test specimen inside the cell. The cap is then replaced and the run started. Before measurement begins the pycnometer typically purges itself for several minutes with helium. This serves two purposes; it ensures that only helium is in the cell and it carries off residual moisture from the specimen surface. At this point the actual measurement runs begin.

- If the pycnometer is automated it will rerun the same specimen a pre-selected number of times and, when completed, will print a summary of the run, including the average volume and density with their associated standard deviations. Automated machines will download the raw data and the report to a personal computer if desired. Once the download is complete the pycnometer is ready to repeat the measurement cycle.

- Change out the specimens, re-seal the measurement cell and begin a new run. Recalibration is not necessary for continuous use if the ambient temperature is stable.

6.6.4.5 Summary of helium pycnometry experimental results

A high quality helium pycnometer (Quantachrome Ultrapycnometer 1000) was tested to determine its viability as a tool for measuring the volume/density of composites. Conclusions reached as a result of this testing are as follows (refer to References 6.6.4.1(e) and (f) for all conclusions):

- The D 792 method was more accurate with a maximum deviation of 0.003 g/cm^3 (1.1x10^{-4} lb/in^3) from the certified value for the specimen volumes used. More typically the data varied within 0.001 g/cm^3 (3.6x10^{-5} lb/in^3) of the NIST standard.

- Above 30% fill of the measurement cell the pycnometer data deviated by a maximum of 0.003 g/cm^3 (1.1x10^{-4} lb/in^3). More typically the data varied within 0.002 g/cm^3 (7.2x10^{-5} lb/in^3) of the standard. Below 30% fill the maximum deviation was 0.015 g/cm^3 (5.4x10^{-4} lb/in^3) and a fall off in accuracy with decreasing V_s/V_c was evident.

- The standard deviations of the two techniques are comparable and tight with the pycnometer data showing slightly tighter or equal values for all data points. The typical standard deviation for the D 792 method was 0.001 g/cm^3 (3.6x10^{-5} lb/in^3) while the standard deviations for the helium pycnometer typically ranged from 0.0008 to 0.0002 g/cm^3 (2.9x10^{-6} to 7x10^{-7} lb/in^3). The maximum standard deviation recorded by the pycnometer was 0.003 g/cm^3 (1.1x10^{-4} lb/in^3). The largest standard deviation of the D 792 method was 0.004 g/cm^3 (1.4x10^{-4} lb/in^3) recorded in two instances.

- The pycnometer data have a persistent tendency to read slightly below the actual density value as if a constant offset phenomena was occurring. The reason for the low offset is not known at this time.

One difference between gas and liquid mediums is that gases are much more susceptible to temperature variations. The periodic influx of cool helium, and the further cooling of the helium due to expansion was initially a concern as this situation could cause erroneous low pressures to be recorded. Testing showed that the thermal environment in the sample cell is extremely stable, with the helium temperature worst case recovery time of 9 sec. The worst case maximum deviation value reached was -3.8F° (-2.1C°), which occurred within 50 milliseconds after expansion. From this data it is concluded that the gas expansion is not an issue and that the helium is quickly reheated by the thermal mass of the sample cell. Further, 200 repetitions of the gas expansion event showed no change in the temperature recovery curve or any drop in the cell temperature over time, indicating that the sample cell has ample heat capacity to maintain a stable temperature environment for an indefinite period.

6.6.4.6 Density test methods for MIL-HDBK-17 data submittal

Data produced by the following test methods (Table 6.6.4.6) are currently being accepted by MIL-HDBK-17 for consideration for inclusion in Volume 2.

TABLE 6.6.4.6 *Composite density test methods for MIL-HDBK-17 data submittal.*

Property	Symbol	Fully Approved, Interim, and Screening Data	Screening Data Only
Density	ρ	D 792, D 1505, 6.4.4.4.1*	D 2734C

*When this method is used to generate data for subsequent determination of composite void volume, the test specimen must occupy at least 30% of the test cell volume.

6.6.5 Cured ply thickness

NOTE: Throughout this discussion the term "cured" refers to a fully processed state. For thermosetting materials it means chemically cured. For thermoplastic materials it denotes a fully consolidated condition.

6.6.5.1 Overview

The thickness of a composite part is an important property from the standpoint of weight and dimensional compliance (fit) in hardware applications. Part thickness is governed by the number of plies in the lay-up, the amount of matrix resin present (resin content), the amount of reinforcing fiber (fiber volume), and the amount of porosity (void volume). In the case of resin transfer molding (RTM), the tool dimensions dictate thickness (by controlling resin content). If it is assumed that the amounts of resin, fiber, and porosity do not vary from one ply to another within the structure, then the thickness per ply times the number of plies is representative of the part thickness. In practice, the proportions of resin, fiber, and porosity may vary somewhat from ply to ply. The magnitude of this variation is largely a function of processing parameters. For example, surface bleeding during cure may produce lower resin content in the outer plies compared to interior plies, depending on the mobility of the resin through the part thickness. However, the average cured ply thickness multiplied by the number of plies generally provides a reasonable estimate of part thickness.

Since test panel laminates are typically processed in a manner that simulates a production part process, panel cured ply thickness may also be used to estimate part thicknesses. In addition, cured ply thickness of a test panel may be used in the calculation of fiber volume and subsequent normalization of mechanical test data (see Section 2.4.2 on normalization).

Determination of cured ply thickness generally involves measuring the thickness of a laminate (panel or part) in a number of locations, averaging these thickness values, and dividing by the number of plies in the lay-up. Laminate thickness can be measured by direct means (using devices such as micrometers) or indirectly (using ultrasonic instruments). Sections 6.6.5.2 and 6.6.5.3 below briefly discuss the use of direct and indirect means of measuring laminate thickness. Section 6.6.5.4 discusses SRM 10R-94, which is the only current standard for measuring cured ply thickness.

6.6.5.2 Thickness measurement using direct means

Deep throat micrometers are typically used to directly measure thickness at various locations over the laminate surface. While this is a fairly straight forward procedure, there are several issues to consider.

First is the matter of panel or part size and shape. If the laminate to be measured has large length and width dimensions, the micrometer may not reach far enough into the interior. This problem may be overcome by substituting a dial indicator or similar device suspended from a rigid framework, but accuracy is usually sacrificed. Also, if the laminate has curvature, the micrometer throat may interfere to the extent that the anvils will not reach the laminate surfaces. Laminate surface texture is another significant issue. The reader is referred to Section 6.4.2 for a detailed discussion of this topic. If the size and shape of the laminate do not present a problem, ball-faced micrometers offer an accurate, low cost means of direct thickness measurement.

6.6.5.3 Thickness measurement using indirect means

Pulse-echo type ultrasonic equipment can be used to measure laminate thickness. This technique makes use of the fact that sound can be directed through a laminate, reflected from the opposite surface, and its traverse time measured. If the sound velocity through the laminate material is established by testing specimens of known thickness, then the unknown laminate thickness can be calculated. ASTM E797-90 (Reference 6.6.5.3(a)) describes this practice, but does not include detailed information or any specifics relative to measurement of composite laminates.

One advantage of using an ultrasonic method is that access to only one surface is required. This is important for measuring skin thickness on closed structures, or for measuring large laminates where micrometer measurement is not possible. However, the disadvantages are considerable. First, the equipment can be expensive relative to other options. Second, calibrations must be run on specimens of known thickness. This must be done for each specific material to be tested since sound velocity may be different for each. A further complication is that velocity is also affected by the ratio of fiber to matrix resin

in the laminate and, in fact, SACMA method SRM 24R-94 (Reference 6.6.5.3(b)) takes advantage of this very fact to estimate resin content of prepreg. Third, surface texture is a concern as discussed earlier in Section 6.6.5.2.

Because of the significant disadvantages, this measurement method is not recommended where direct measurement using a micrometer or similar device is possible.

6.6.5.4 SRM 10R-94, SACMA Recommended Method for Fiber Volume, Percent Resin Volume and Calculated Average Cured Ply Thickness of Plied Laminates

The cured ply thickness portion of this method (Reference 6.6.5.4) specifies that thickness readings be taken in at least 10 locations over the surface of the laminate using a ball-faced micrometer. It is recommended that no readings be taken closer than one inch (25 mm) from any edge. The average laminate thickness is calculated and divided by the number of plies to obtain an average cured ply thickness. The method recommends that a laminate be subdivided for the calculation of fiber volume if the variation in laminate thickness exceeds 0.008 inch (0.2 mm). This indirectly suggests that a single cured ply thickness should not be calculated under such conditions.

6.6.5.5 Cured ply thickness test methods for MIL-HDBK-17 data submittal

Methods which meet the requirements of SRM 10R-94 are acceptable when submitting data to MIL-HDBK-17 for consideration for inclusion in Volume 2. In addition, cured ply thickness may be calculated using measured thicknesses of test specimens obtained from a panel provided there are at least 10 specimens distributed over the entire area of the panel (so as to be equivalent to SRM 10R-94).

6.6.6 Fiber volume (V_f) fraction

6.6.6.1 Introduction

The fiber volume (expressed as a fraction or percent) of cured polymer-matrix composites is commonly obtained by matrix digestion, ignition loss, areal weight, and image analysis methods. These methods generally apply to laminates fabricated from most material forms and processes, but the areal weight method cannot be used for filament wound material or other forms that do not consist of discrete individual plies. Each method has its benefits and drawbacks. Other less common methods will not be discussed.

6.6.6.2 Matrix digestion

The method of matrix digestion is covered under ASTM Test Method D 3171 “Fiber Content of Resin-Matrix Composites by Matrix Digestion” (Reference 6.6.6.2). The technique is based on digestion of the matrix by a suitable liquid which does not attack the reinforcing fibers. Depending on the resin, three different procedures are used: Procedure A, concentrated nitric acid; Procedure B, aqueous mixture of sulfuric acid and hydrogen peroxide; and Procedure C, a mixture of ethylene glycol and potassium hydroxide. For example, epoxies generally respond well to all three procedures. Although toughened systems respond better to procedure B, some fiber types are attacked more by B than by A. BMI's, polyimides, and thermoplastics usually respond well to procedure B. Aramid fibers are attacked by both A and B, and therefore, procedure C works best on aramid fiber composites.

Possible causes of error:

If the fiber is significantly attacked by the digestion fluid, results will be erroneous. It is recommended to validate the process by testing a control sample of only fiber to determine mass change of fiber during test.

Some toughened resin systems have additives such as elastomers or thermoplastics. If these additives are not dissolved by the digestion fluid, they may cling to the fiber causing erroneous results.

Incomplete digestion of the resin.

Sample size must be large enough to be representative and weighed accurately.

Accuracy is dependent on accuracy of density measurements.

6.6.6.3 Ignition loss

The method of ignition loss is described in standard test method ASTM D 2584 “Ignition Loss of Cured Reinforced Resins.” The technique determines ignition loss of cured polymer-matrix composites which can be considered to be the resin mass. A weighed specimen is heated until the resin matrix is oxidized and converted to volatile materials. After removing any remaining ash, the residue (reinforcing fiber) is weighed and the percent loss is calculated. Fiber density and composite density (to three significant figures) are required in order to calculate fiber volume.

Possible causes of error:

If the fiber gains or loses weight under the conditions of the test, the results will be erroneous. (For this reason, this procedure is not appropriate for aramid fibers and requires special temperature controls for carbon fibers.)

Fillers, if present, must be oxidized with resin.

Incomplete decomposition of resin (and fillers, if present) during the test.

Any volatiles such as water, residual solvent, etc., will cause error unless they are small enough to be ignored.

If the sample is heated too rapidly, mechanical loss of noncombustible residue (fiber) can occur, causing erroneous results.

6.6.6.4 Areal weight/thickness

Sections 6.6.6.2 and 6.6.6.3 discussed methods for determining fiber volume by destructively separating the fibers from the matrix through chemical or thermal means and measuring the mass of the fibers in a sample. As discussed in Section 2.4.3 on data normalization, there is a relationship between laminate (or specimen) thickness and fiber volume for given values of fiber areal weight and fiber density. This fact provides the basis for another method of fiber volume determination, which is not destructive.

In general the method involves measuring the laminate or specimen thickness and calculating the fiber volume using this measured thickness, the number of plies in the laminate, and previously determined values of fiber areal weight and fiber density. Equation 6.6.6.4(a) or an equivalent form is used.

$$V_f = \frac{FAW \times n}{t \times \rho_f} \times k \qquad 6.6.6.4(a)$$

where

V_f = fiber volume fraction
FAW = fiber areal weight (mass per area per ply)
n = number of plies in the laminate
t = measured laminate (or specimen) thickness
ρ_f = density of the reinforcing fiber
k = units conversion factor (if required)

The calculated fiber volume is the reinforcing fiber's contribution to the total volume. Although void content does affect the laminate thickness (and hence fiber volume), it is not a factor in the calculation since it contributes to total volume in the same way as the resin or any other non-reinforcement component. Since the calculation requires the number of plies, the method is applicable only to material forms with distinct plies for which fiber areal weight can be determined.

This general procedure is documented in SRM 10R-94 (Reference 6.6.6.4(a)), which references other SACMA Recommended Methods for determination of fiber areal weight and fiber density. The specimen defined by this method is a laminate panel, but the concept could be extended to individual test specimens or to application parts. The method notes that the fiber areal weight and fiber density used in the fiber volume calculation must be representative of the sample (panel, specimen, or part) under evaluation. This is an important point. Although resin content may typically be the major factor affecting fiber volume, fiber areal weight and fiber density variations can also have a significant effect on the accuracy of this method. "Typical" or "data sheet" values for these parameters should not be used in the calculation. SRM 10R-94 recommends that, as a minimum, fiber areal weight of the individual prepreg roll and lot average fiber density be used. If careful thickness measurements are taken (see Section 6.6.5) and appropriate fiber areal weight and fiber density values are used, this method can be quite accurate, and potentially more reliable than destructive methods that depend heavily on laboratory technique.

For certain types of specimens the accuracy of thickness measurements may not be sufficient to yield accurate fiber volumes. In particular, very thin specimens present the problem of obtaining three significant figure precision. In addition, thickness cannot be reliably measured for specimens with irregular surfaces. As an alternate to measuring specimen thickness directly, it may be calculated using equation 6.6.6.4(b):

$$t_c = \frac{M}{A \times \rho_c} \qquad 6.6.6.4(b)$$

where

t_c = calculated laminate (or specimen) average thickness
M = mass of the specimen
A = surface area of the specimen
ρ_c = measured density of the composite specimen

Combining equations 6.6.6.4(a) and 6.6.6.4(b) yields the following expression for fiber volume fraction in terms of laminate specimen mass, density, and area:

$$V_f = \frac{FAW \times A \times \rho_c \times n}{M \times \rho_f} \times k \qquad 6.6.6.4(c)$$

When mass is in grams, FAW in g/m^2, densities in g/cm^3, and area in in.^{2}, then k = 1/1550.

For this variation of the method, the specimen must be extracted from the laminate or part such that its edges are cut cleanly without any fraying. Ideally the specimen should be rectangular to permit area calculation from specimen length and width. If possible, the specimen should be cut at least one inch from any laminate (part) edge to minimize edge effects caused by fiber wash. The specimen mass is obtained by weighing on a balance with adequate precision, and density is measured using archemedian or other suitable methods (see Section 6.6.4 of this volume). More detail may be found in Reference 6.6.6.4(b).

Possible causes of error:

- Inaccuracies in measured thickness caused by use of inappropriate measuring equipment, particularly on textured surfaces (see Section 6.4.2).

- Inaccuracies in calculated surface area (for the area variation of this method) caused by edge damage induced during specimen cutting or by inappropriate measuring equipment or technique, especially for irregularly shaped specimens.

- Wide variation in measured thickness across the specimen (in which case the calculated average fiber volume is not representative of all areas of the specimen).

- Significant fiber wash (spreading) during cure (which causes an effective reduction in fiber areal weight in the specimen compared to the value measured for the prepreg).

- Use of fiber areal weight and/or fiber density values that are not representative of the fiber in the specimen.

6.6.6.5 Determination of fiber volume using image analysis

6.6.6.5.1 Background

The method of image analysis offers a technique of measuring fiber volume that eliminates the creation of waste chemicals while providing information about void volume laminate orientation and through thickness fiber distribution. The basic assumption for this technique is that the evaluation of the two dimensional distribution of fibers through a random cross section is representative of the volumetric fiber distribution. This assumption is valid for fibers of constant cross section, such as is found in tape laminates, but is not valid for woven laminates. This technique works well for carbon fibers in a polymer matrix, and for other fiber/matrix combinations where adequate contrast can be achieved. It does not, for example, work as well for glass fibers, since the low contrast between the glass fibers and surrounding matrix makes accurate measurements very difficult. There are no industry standard test methods for this type of evaluation. Therefore, this section will describe the procedure in general terms. Computer software for image analysis is commercially available.

6.6.6.5.2 Apparatus

This technique requires the use of metallographic specimen preparation equipment, a reflected light microscope with a magnification of at least 400 times which has the capability of porting the image to a digital camera, a computer with image acquisition card and image analysis software. While automated image acquisition systems are available, this analysis can also be performed by manual specimen translation and focusing. The use of software macros can reduce the time required to process a fiber volume measurement. Macros allow the user to automate repetitive software instructions.

6.6.6.5.3 Specimen preparation

A small section of laminate is prepared using standard metallographic techniques. A typical specimen is ¾ inch (2 centimeters) on a side. The cross section is taken across the thickness of the laminate. The orientation of the cross section with respect to the laminate is dependent on the orientation of the fibers in the laminate being evaluated. The image analysis technique can be used to evaluate fiber volume in laminates with ply orientations from 0 to approximately 60°. At angles above 60 degrees the fiber edge definition becomes distorted by the subsurface fibers. If a laminate with 0/90 lay-up is evaluated, the laminate can be sectioned at 45° to enable evaluation of the fiber volume in all plies. If fiber volume is to be measured on a 0/±60 laminate, the cross section can be oriented at 0° so that all plies can be evaluated.

The surface of the polished specimen should display a clear delineation between the fibers and the matrix. The fiber volume measurement should be made at as high a magnification as possible. This to

some extent depends on the fibers being examined, but for most fibers a magnification of at least 400 to 1000 times should be used. This should get 30 to 100 fibers in the field of view. As these are areal measurements being performed, the partial fibers can be included in the analysis. This would not be true if the individual fiber area were being determined. A typical image is shown in Figure 6.6.6.5.3.

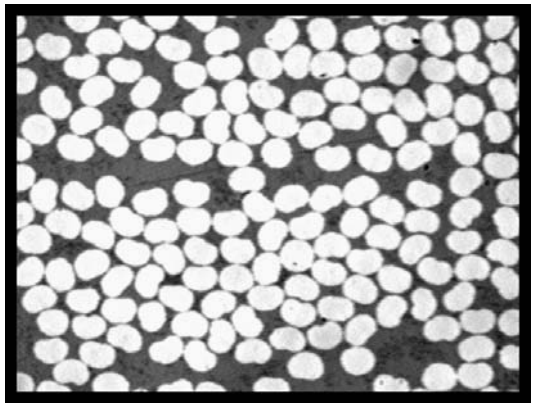


FIGURE 6.6.6.5.3 *Typical grayscale image (M55J fibers).*

6.6.6.5.4 Image analysis

The objective of the fiber volume image analysis technique is to discriminate between the fibers and matrix. The image is acquired as a gray scale, threshold intensity is used to select the delineation point between the fibers and matrix. The threshold level can be determined by evaluating a histogram of the image as shown in Figure 6.6.6.5.4(a). Typically once the threshold is selected for a given cross section it does not have to be altered as additional images are acquired for that specimen. It is good practice to display the histogram while acquiring images to confirm the veracity of the threshold level. The threshold level is used to convert the grayscale image to a binary image (Figure 6.6.6.5.4(b)) where the fibers will be either black or white and the matrix will be the opposite (white or black). The computer then counts the number of black and white pixels, and the ratio of the fiber pixels to the total number of pixels in the image is the fiber volume.

While automated image systems can be programmed to analyze the entire cross section, this may require as many as 1000 images. Accurate results can be obtained from manually operated systems using from 20 to 50 samples. Testing has shown that where the fibers/resin are evenly distributed, the mean fiber volume converges to a constant value in as few as 20 samples. The manual sampling should be distributed throughout the cross section.

The typical steps in the analysis of a single frame are:

1. Position the specimen. (Manually/automatically move the stage on which the specimen rests.)
2. Focus the microscope. (Manual focusing requires the use of a real time monitor on older computers. Newer computers have acquisition rates fast enough to preclude the use of a monitor.)
3. Acquire an image. (The image used to measure the fiber volume may be a single frame or an average of multiple frames. Integrating several images can offset low luminescence of an image. Examination of the histogram will indicate if the image is suitable for evaluation.)
4. Identify pixels corresponding to fibers. (The histogram should be checked to insure that the correct threshold value has been selected.)
5. Create a binary image. (The pixels with values greater than the threshold value will be black and the pixels with values less than the threshold value will be white.)

6. Count the number of white and black pixels. (Typically it will be necessary to obtain a count of only one of the colors. The total number of pixels in the frame remains constant so the number of fiber pixels for a given image is all that needs to be recorded.)

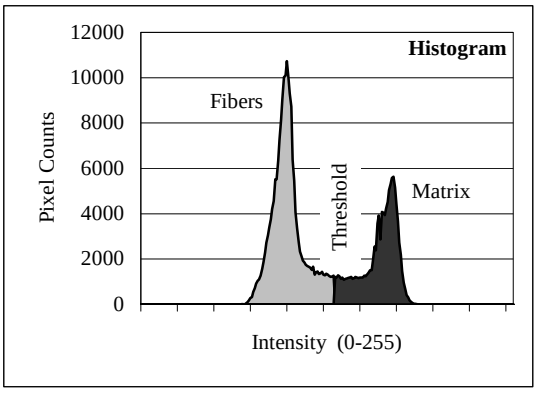


FIGURE 6.6.6.5.4(a) *Typical histogram of gray scale fiber and matrix distributuion.*

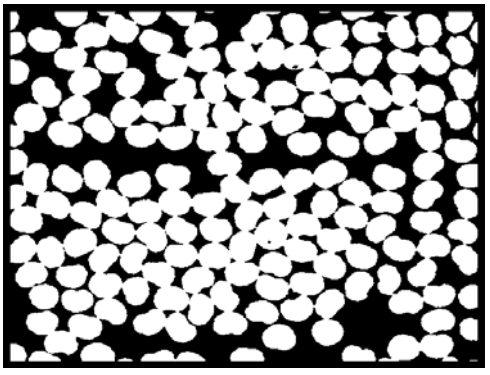


FIGURE 6.6.6.5.4(b) *Binary fiber image.*

The time required to process a fiber volume measurement can be accelerated by the use of macros within the image analysis program. After the microscope has been focused (step 2), a macro can be initiated via a single keystroke which initiates the acquisition sequence (steps 3 through 6). The histogram can be displayed so that the operator can verify the adequacy of the threshold value selected. Some imaging software programs will have an automatic threshold operation.

6.6.6.5.5 Sources of error

- Out of focus or dirty optics can distort the image which in turn will give inaccurate results.
- Poor metallographic preparation techniques of the cross-section surface makes accurate thresholding difficult.
- Insufficient magnification will result in poor definition of the fibers.

- Poor microscope lighting or incorrect use of blank field image will distort the intensity distribution. The filament in the incandescent bulb used to illuminate the surface may not be uniform. This would result in a non-uniform distribution of light on the specimen surface and yield a distorted histogram. This can be corrected by creating a blank field image that is subtracted from the acquired image. The blank field can be taken either with the microscope slightly out of focus or in focus on a clean area of the polished mounting material surface. Figures 6.6.6.5.5(a) and (b) illustrate this correction.

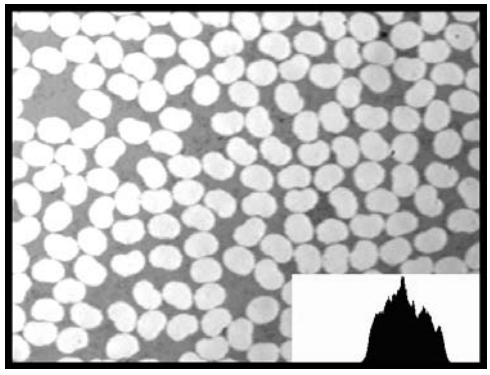


FIGURE 6.6.6.5.5(a) *Gray scale image and histogram of cross-section with a variation in illumination.*

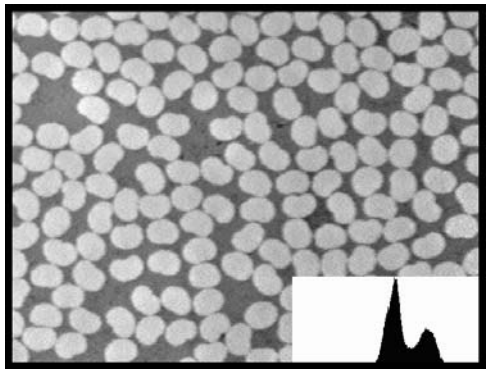


FIGURE 6.6.6.5.5(b) *Gray scale image with the variation in illumination compensated using a blank field.*

6.6.7 Void volume (V_v) fraction

6.6.7.1 Introduction

Increasing void volume (expressed as a fraction or percent) of a composite material may adversely affect its mechanical properties. The void volume of cured polymer-matrix composites may be obtained by digestive and image analysis assessment. Digestive evaluation uses constituent content and density data to calculate the volumetric void content. Image analysis assessment is obtained by micrographic methods.

6.6.7.2 Digestive evaluation

The most common test method to determine void content is described in ASTM D 2734 “Test Method for Void Content of Reinforced Plastics” (Reference 6.6.7.2). Void content by volume is calculated using

resin and fiber weight percents in the laminate (see Section 6.6.6), along with the densities of the laminate (see Section 6.6.4), fiber (see Section 3.3.2) and resin (see Section 4.5.5). (Fiber and resin densities are commonly obtained from the material supplier.)

This procedure is sensitive to variations in densities and constituent weight percents. Therefore, it is important to use fiber and resin densities that are representative of those constituent densities within the sample tested, and are accurate to three significant figures. Occasionally, negative values of void content may be calculated. Test accuracy is on the order of ± 0.5%. Therefore, calculated values between -0.5% and 0% are typically considered to be zero. Larger negative values should be investigated for possible errors in technique or procedure. Note that the location and size of the sample should be representative of the material and large enough to minimize experimental error.

Possible causes of error:

Volume measurement may be inaccurate if the sample is not cut with precision.

Laminate density may be inaccurate if the sample is not dried prior to density determination.
Void volumes may not be accurate if density values are determined to less than three significant figures.

6.6.7.3 Determination of void volume using image analysis

6.6.7.3.1 Background

The image analysis technique described in Section 6.6.6.5 can also be used to determine void volume percent. This technique assumes that porosity is essentially the same throughout the laminate and that a random cross-section, therefore, functions as an accurate representative. This assumption is not correct if significant linear (along fiber length) porosity is present. A typical gray scale image is shown in Figure 6.6.7.3.1(a).

The void volume measurement involves using a histogram with three peaks instead of two as shown in Figure 6.6.7.3.1(b). The first peak representing the fibers, the second is the matrix (resin), and the third are the voids. Three colors are then used to represent the areas of the histogram (white, gray, and black, or other user-selected colors). Area percent measurement then proceeds as described in Section 6.6.6.5, with the area of each color measured against the total measured area. The assumption here is that there is adequate contrast between the three areas.

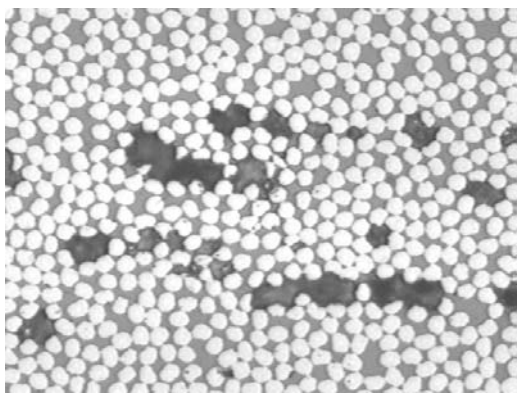


FIGURE 6.6.7.3.1(a) *Typical gray scale image of laminate with voids taken at 400X.*

The apparatus and specimen preparation are identical to that described in Section 6.6.6.5. A lower magnification, 100 to 200 times, can be used when only void volume is measured or when the void distribution is not uniform. The image analysis is identical to that described in Section 6.6.6.5.

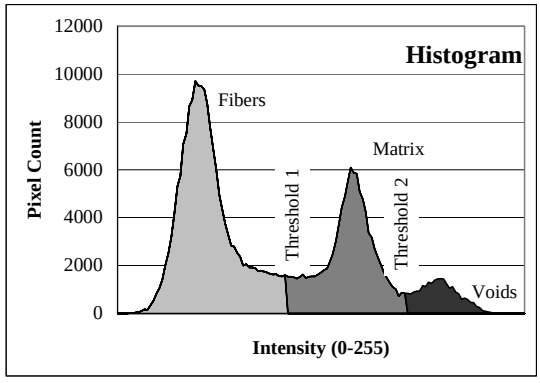


FIGURE 6.6.7.3.1(b) *Histogram of intensity distribution of image shown in Figure 6.6.7.3.1(a).*

6.6.7.3.2 Sources of Error

- Non-uniform void distribution can lead to significant errors in void volume measurement. Figures 6.6.7.3.2(a) and 6.6.7.3.2(c) show the area around the voids shown in Figure 6.6.7.3.1(a). Figures 6.6.7.3.2(b) and 6.6.7.3.2(d) reflect the histograms for Figures 6.6.7.3.2(a) and 6.6.7.3.2(c), respectively. The void distribution is not uniform across the cross section. Consequently the measured void volume decreases as the magnification is reduced. Using a single image acquired at 400, 200 and 100 times magnification the void volumes were found to be 7.71%, 2.17%, and 0.78%, respectively. As the 100X image encompasses almost the entire thickness of the laminate, the void volume measured from this image is the most accurate. For thicker laminates 100 times magnification would only be a fraction of the laminate thickness. The accuracy of the fiber volume measurement also decreases with decreasing magnification. It is more accurate to perform void volume measurements at lower magnifications than those used for fiber volume measurements.

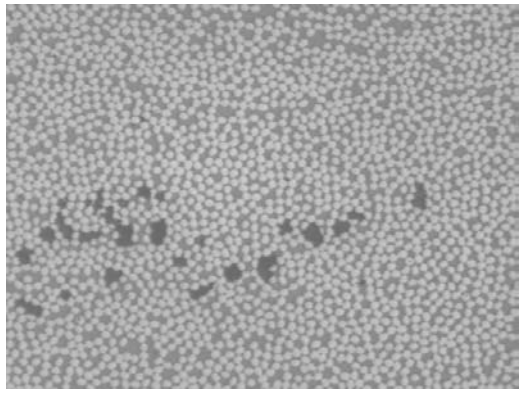


FIGURE 6.6.7.3.2(a) *Typical gray scale image of laminate with voids taken at 200X.*

- Matrix modifications in a discrete phase (thermoplastic toughening particles) could be confused with voids.

- The lower the magnification used, the lower the effective pixel size.

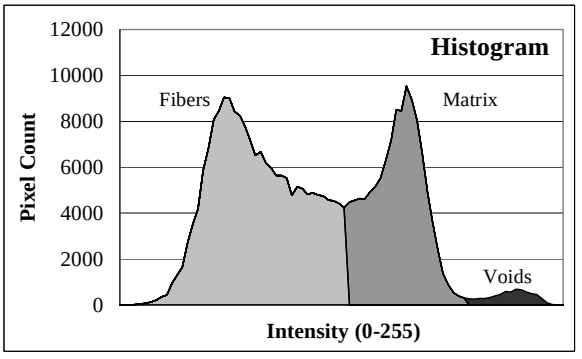


FIGURE 6.6.7.3.2(b) *Histogram of intensity distribution of image shown in Figure 6.4.7.3.2(a).*

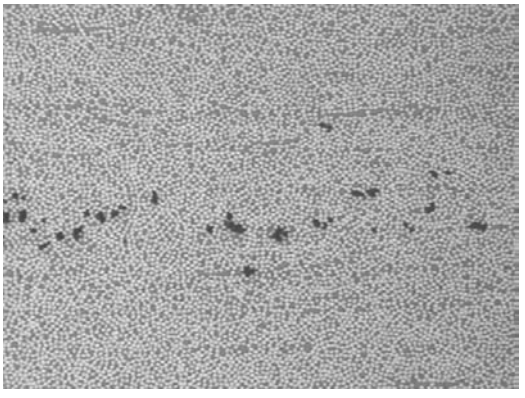


FIGURE 6.6.7.3.2(c) *Typical gray scale image of laminate with voids taken at 100X.*

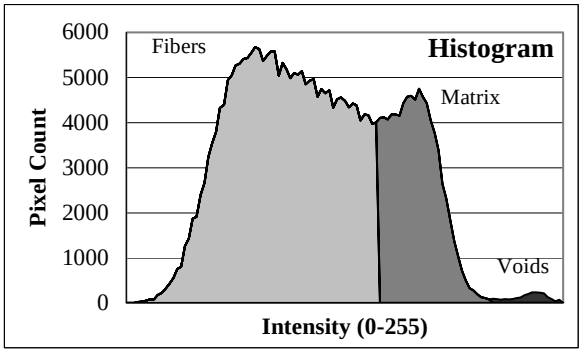


FIGURE 6.6.7.3.2(d) *Histogram of intensity distribution of image shown in Fig. 6.4.7.3.2(c).*

6.6.8 Moisture/diffusivity

Through-thickness moisture/fluid diffusivity: D_3 or D_z
Moisture/fluid equilibrium content: M_m
In-plane moisture/fluid diffusivity: D_1, D_2; D_x, D_y

Many polymeric materials absorb moisture, though in varying amounts and at varying speeds. Moisture is most widely encountered in humid air but is also obviously seen in water (and salt-water) immersion. Other types of fluid exposure, such as hydraulic fluid or jet fuel or even (as in biomedical applications) body fluids, are also encountered during service life in some applications and are considered moisture for the purposes of this discussion.[1]

The most commonly adopted moisture diffusion model, and one that many materials have been demonstrated to follow reasonably well, is Fickian diffusion. A one-dimensional example of this model (with a moisture-concentration independent diffusivity) is shown below; it is a direct analog to the more commonly studied thermal diffusion equation:

$$\frac{dc}{dt} = D\left(\frac{d^2c}{dz^2}\right) \qquad 6.6.8(a)$$

where:
c = moisture concentration (g/mm^3)
t = time (s)
D = moisture diffusivity (mm^2/s)
z = coordinate direction of diffusion (mm)

The only standard test method for moisture diffusivity (ASTM Test Method D 5229, discussed in the next section) assumes that the test material behaves as a single-phase Fickian material. The procedure in this test method for calculation of moisture diffusivity will not be accurate for materials that behave otherwise. The prime example of non-Fickian behavior is material containing linked microcracks that allow a direct path for moisture movement. To determine if a material is Fickian, conduct the test, examine the behavior, and calculate the diffusivity, then compare predicted behavior to the test result. If the test behavior does not follow guidelines in the ASTM standard, or the test/analysis correlation is poor, the diffusion may not follow the single-phase Fickian model. However, by procedures not contained in the standard, the data acquired during testing can be used to calculate diffusivity for multi-phase Fickian behavior, if such a model better fits the test results. Also, moisture equilibrium content can be determined by these tests even for materials that do not follow single-phase Fickian behavior.

Other methods of determining moisture content are available but not widely used due to expense, lack of standardization, and/or other limitations, and are not discussed further. One of the newer such methods is nuclear magnetic resonance (NMR) as used in magnetic resonance imaging machines, which, for electrically non-conductive materials (this excludes carbon reinforcement), can non-destructively determine the moisture concentration spacial distribution.

The two primary moisture-related properties of a polymeric composite material are the through-thickness *moisture diffusivity constant,* D_3 or D_Z, (speed of moisture diffusion) and *moisture equilibrium content,* M_m. M_m is the total absorbed moisture as a percentage of overall material weight, determined at equilibrium. For a given material, moisture diffusivity is actually a constant only for a given environment and direction of diffusion, as it normally varies very strongly as a function of temperature. Equilibrium

[1] There are several possible definitions of the term “moisture.” It has been used to refer to the vapor of a fluid, or its condensate, or even the bulk fluid itself in large quantities. It has been restricted to water itself, in one or more of these forms, or applied to other fluids. While the term “fluid” may arguably be a more precise term for what we are covering in this section, the term “moisture” is retained for general use, largely due to the historical emphasis and use of the term in discussing these problems. For example, the symbol “M” used to describe absorbed fluid equilibrium content is taken from the word “moisture.” And so it is convenient for the purposes of this discussion to extend the definition of moisture to all absorbed fluids.

moisture content, on the other hand, does not vary strongly with temperature, but does vary, in the case of humid air, with relative humidity level. (Important background material on the use and application of these properties is located in Volume 1, Sections 2.2.6-8 and 6.3.)

Moisture diffusion of polymeric composites is generally not isotropic. It is not unusual for in-plane moisture diffusivities (D_1, D_2; D_x, D_y) to be an order of magnitude higher than D_3 or D_z. General diffusion models consider moisture diffusivity, like thermal diffusivity and thermal conductivity, to be a second-order tensor quantity that mathematically varies (transforms) as a function of direction in accordance with tensor transformation rules. While it may be tempting to ignore the surface area of edges when they constitute a small proportion of the overall surface area, moisture diffusion through the edges can be significant in a test specimen of limited size. With an understanding of this, the usual procedure is to limit diffusion through the edges so that its effects can be ignored. In-plane moisture diffusion is rarely precisely quantified. No standard test methods for determination of in-plane moisture diffusion currently exist.

Provided the moisture exposure has not cracked or chemically altered the material, the desorption behavior is the reverse of the absorption behavior. In fact, well-behaved desorption is one of the qualitative indications of Fickian behavior.

6.6.8.1 Standard test methods
1) ASTM D 5229/D 5229M
2) SACMA RM 11R

The only standard test method that rigorously covers determination of the two primary properties is ASTM D 5229/D 5229M (Reference 6.6.8.1(a)). Another test method that covers determination of moisture equilibrium content only is SACMA RM 11R (Reference 6.6.8.1(b)). SACMA RM 11R is based on similar methods used in the ASTM standard, covers determination of properties with somewhat less rigor, and is limited to a single fixed environment of 85% RH humid air. Both of these test methods are gravimetric experiments. A piece of the material is initially weighed and then periodically weighed during exposure to the fluid environment in question. From these data the through-thickness moisture diffusivity (ASTM only) and equilibrium moisture content (both ASTM and SACMA) can be determined.

Both of these test methods, especially ASTM D 5229, cover the subject quite well within the test method documentation. Anyone interested in better understanding this subject should start by reading the test methods, as well as the related sections of this handbook referenced above. The primary reference for these standards is Reference 6.6.8.1(c). Beyond this, further reading would include the other references specified within ASTM D 5229.

Testing issues worth noting that are not currently covered by the test methods themselves include:

1. ASTM D 5229 specifies stringent requirements for a single test specimen that is used to determine, in the same experiment on the same specimen, both moisture diffusivity and moisture equilibrium content. However, it is substantially more practical to test two different test specimen geometries to obtain these properties: a thin specimen from which maximum moisture content can relatively quickly be determined, and a thick specimen from which an extremely stable initial linear slope of the moisture mass gain versus square-root of time plot can be found. The two-specimen approach provides a much more accurate and cost-effective result than that literally described in the 1992 initial release of this standard. ASTM D 5229 is expected to be revised as soon as practical to include this concept.

2. While the ASTM test method uses a diffusivity-based guideline to assess satisfaction of equilibrium (e.g., the maximum allowable change in mass between the last two weighings, which determines when effective equilibrium has been attained, is lower for higher-diffusivity specimens), the SACMA test method uses a fixed reference time period of 24 hours. The approach used by the ASTM test method essentially adjusts the test parameters to result in a fixed maximum error, while the SACMA test method fixes the test parameters and results in variable error.

It is also important to note that there is wide variation in the moisture response of polymers. There are classes of polymers, and their composites, that essentially do not absorb moisture to any appreciable extent due to their molecular structure. These test methods may not produce meaningful results on such materials. One such polymer is polybutadiene.

There are other polymers that, upon cursory examination, *appear* to have no appreciable moisture weight change response, but actually have a low diffusion constant. However, given sufficient exposure time, these polymers will eventually absorb a meaningful amount of moisture. Some high-performance thermoplastics and thermosets fall in this category.

At the other end of the scale are the polymers that absorb (and desorb) moisture so quickly that extreme care must be taken with the gravimetric measurements and with the calibration and control of the environmental chamber, in order to avoid large measurement errors. The most convenient solution for reducing the test sensitivity of these materials is to simply increase the thickness of the test specimen. Polyetherimide is one such material.

While not strictly within the scope of this section, it is worthy to note that most current polymeric matrix sandwich core materials absorb, and are affected by, moisture. When honeycomb core, in particular, is evaluated by itself for moisture absorption response or is mechanically tested as bare core under conditions of controlled moisture content, the surface area is so large relative to the thickness of the cell walls (often as small as 0.003 inch) that the material can absorb/desorb very quickly. Bare honeycomb core is, therefore, very sensitive to experimental procedure for environmental testing or conditioning.

6.6.8.2 Moisture diffusion property test methods for MIL-HDBK-17 data submittal

Data produced by the test methods in Table 6.6.8.2 are currently being accepted by MIL-HDBK-17 for consideration for inclusion in Volume 2.

Table 6.6.8.2

Property	Symbols	Fully Approved, Interim, and Screening Data	Screening Data Only
In-Plane Moisture Diffusivity	D_1, D_2 (lamina) D_x, D_y (laminate)	---	---
Through-Thickness Moisture Diffusivity	D_3 (lamina) D_z (laminate)	D 5229	---
Equilibrium Moisture Content	M_m	D 5229 SRM 11R (85% humid air only)	---

6.6.9 Dimensional stability (Thermal and Moisture)

Dimensional changes in composite materials are typically a function of temperature and/or moisture. Changes in length or volume of a sample can be detected by mechanical, optical or electrical transducer and recorded as a function of temperature or time. Several techniques for measuring linear expansion, such as dial gauges, micrometers, telescopes, linear variable differential transformers, interferometers, and X-ray diffraction patterns, have been used.

6.6.9.1 Dimensional stability (thermal)

α, α_{11}, α_{22}

6.6.9.1.1 Introduction

It is well known that most materials change dimensions with a change in temperature. In fact, most materials expand as the temperature is increased. By definition, isotropic materials, which typically include bulk metals, polymers and ceramics, expand equally in all directions. The reinforcing fibers used to reinforce these bulk materials may or may not be isotropic. For example, inorganic fibers such as glass, boron, and other ceramics are isotropic, while organic fibers such as carbon, aramid (e.g., DuPont's Kevlar), polyethylene (e.g., Allied's Spectra) and others are not.

Even if an isotropic fiber is used in combination with an isotropic matrix, the resulting composite will not be isotropic. The oriented fibers, which are presumably stiffer than the matrix, produce a composite stiffness higher in the direction of orientation than in the transverse direction. Correspondingly, the thermal expansion of the isotropic reinforcing fiber will typically be different than that of the matrix. In the fiber direction the fiber and matrix expand in parallel, while in the transverse direction they expand in series. Thus, in the axial direction the composite thermal expansion is strongly controlled by the thermal deformation of the (stiff) fiber. In the transverse direction the thermal expansion is in proportion to the relative amounts of fiber and matrix in that direction, and their respective thermal expansions. That is, even for isotropic fibers in an isotropic matrix, the composite thermal expansion is anisotropic, and is governed in a complex manner by both the mechanical and thermal properties of each constituent, the fiber orientation, and the relative amounts of fiber and matrix present.

For anisotropic fibers the thermal anisotropy of the resulting composite is even more complex, although no less predictable or measurable. Of special utility is that anisotropic fibers such as carbon, aramid, and polyethylene have negative coefficients of thermal expansion in the fiber axial direction, and relatively high positive coefficients of thermal expansion in the transverse (diametrical) direction. Since the fiber axial stiffness tends to be much higher than that of the matrix, the resulting composite is likely to have a negative coefficient of axial thermal expansion (although the transverse expansion will be positive). In fact, by combining a fiber of the appropriate axial stiffness and negative thermal expansion with a matrix of given stiffness and (positive) thermal expansion, a composite having a zero axial thermal expansion can be achieved (although again a positive transverse expansion will be obtained). Thus, the thermal expansion properties of a composite can be tailored to the specific application just as mechanical properties are.

Thermal stability is customarily defined in terms of a Coefficient of Thermal Expansion (CTE) expressed symbolically as α. Typical units are 10^{-6}/K (microstrain per degree Kelvin). Since CTE is calculated as the slope of the expansion versus temperature curve, linear expansion over the temperature range of interest is assumed. However, depending on the material and the temperature range, expansion may not be linear. When expansion over the temperature range of interest is not linear, it is common practice to calculate a separate CTE for each subrange that approximates linearity. Thus, in general, CTE is temperature dependent, and there is not a single value of CTE for a given material.

The classes of polymers used as matrix materials, e.g., epoxies, bismaleimides, polyimides, and high temperature thermoplastics, have higher values of CTE compared to metals and ceramics. At temperatures above their glass transitions, they have larger values of CTE than below their transitions, a behavior which may be used to determine glass transition temperature (see Section 6.6.3). Materials having several polymer constituents may have multiple glass transitions, making the expansion versus temperature curve more complex.

6.6.9.1.2 Existing test methods

There are four ASTM standards governing the experimental determination of the thermal expansion of unreinforced (neat) polymers and their composites. ASTM Standard D 696 (Reference 6.6.9.1.2(a)) is the

simplest of these standards, being applicable only over the relatively narrow temperature range of -20°F to 90°F (-30°C to 30°C). The reason for this narrow range is that the standard is intended primarily for testing (commodity) plastics, which by their nature have a limited range of use temperatures. The apparatus itself, a vitreous silica dilatometer (often termed a fused silica or a quartz tube dilatometer, although not in this ASTM standard), can be used over a much wider range of temperature. In fact, ASTM Standard E 228 (Reference 6.6.9.1.2(b)) utilizes a similar apparatus and specifies a use temperature range of -290°F to 1650°F (-180°C to 900°C), although some minor cautions are given for applications over 900°F (500°C). This standard is intended for a broader range of test materials, including metals, plastics, ceramics, refractories, composites and others.

ASTM E 831 (Reference 6.6.9.1.2(c)) utilizes thermomechanical analysis (TMA) to measure thermal expansion. The principle of operation of TMA is not unlike that of a vitreous silica dilatometer, and thus the applicable temperature range is comparable. ASTM E 831 indicates a range of applicability from -180°F to 1100°F (-120°C to 600°C), and suggests that this range may be extended depending upon the specific instrumentation and calibration materials used.

Per ASTM D 696, the vitreous silica dilatometer is limited to the measurement of thermal expansion coefficients greater than 1 microstrain/K. ASTM E 831 suggests a lower limit of 5 microstrain/K for TMA, this lower resolution being due to the smaller specimen used in a TMA apparatus. In either case, this level of resolution is adequate for most bulk materials, certainly for most metals and polymers, although marginal for some ceramics. Obviously it is not adequate for those composites designed to have a coefficient of thermal expansion near zero, as described above.

ASTM Standard E 289 (Reference 6.6.9.1.2(d)) utilizes interferometry, which permits the measurement of coefficients of thermal expansion as low as 0.01 microstrain/K. ASTM E 289 indicates an applicable temperature range of -240°F to 1300°F (-150°C to 700°C), again with the suggestion that this range may be extended depending on the instrumentation and calibration materials used. Interferometry does require much more operator skill and care, and more complex equipment, than dilatometry.

In addition to the ASTM standard methods, which utilize a dilatometer or interferometer, bonded foil strain gages can also be used to determine CTE. Typically, the measured thermal expansion of the test material is compared to the expansion of a reference material of accurately known CTE in the same chamber. Although not an ASTM or other standard method, Reference 6.6.9.1.2(e) presents complete details of using strain gages to measure thermal expansions. The applicable temperature range depends upon the material being measured, and the type of strain gage utilized. Per Reference 6.6.9.1.2(e), maximum accuracy gages can be used within a temperature range of -50°F to 150°F (-45°C to 65°C), although this range can be extended somewhat by using alternate types of gages. However, an equally important limitation is the stiffness of the test material at temperature relative to the corresponding stiffness of the gage material. A stiff gage can locally reinforce the test material, leading to erroneously low CTE results.

6.6.9.1.3 Test specimens

Thermal expansion specimens, whether neat polymer or composite, are typically cylindrical in form, and as long in the direction of measurement as the available material and test apparatus permit. The longer the specimen, the greater the length change, and thus the more precise the measurement of CTE for a given equipment resolution. While the specimen cross-sectional shape is somewhat immaterial, specimens are typically circular, square or rectangular. Some minor axial compressive force is usually applied to the specimen during the test to keep the indexing apparatus in contact with the ends of the specimen. This can induce column buckling in materials that have low stiffness at elevated temperatures, e.g., some unreinforced polymers. In fact, even specimen sag under the force of gravity can induce erroneous deformations in such materials. Thus the specimen cross-sectional shape is kept as compact as practical, e.g., square or circular.

Per the general guidelines of ASTM D 696, specimens tested using a dilatometer are typically 2 to 5 in. (50 to 130 mm) long and 0.25 to 0.50 in. (6 to 13 mm) in lateral dimensions. Per ASTM E 831, TMA specimens should be between 0.08 and 0.40 in. (2 to 10 mm) in length, with lateral dimensions not exceeding 0.39 in. (10 mm), although other lengths are permitted if noted in the report. The TMA apparatus configuration itself typically limits the specimen size. For interferometry, ASTM E 289 states that the optimal length is between 0.4 and 0.8 in. (10 to 20 mm) and the lateral dimensions between 0.2 and 0.5 in. (5 to 12 mm).

These small sizes are recommended mainly because of the geometry of a Fizeau interferometer and the advisability to have minimal internal temperature gradients in reference and test samples. The Michelson approach is much more versatile and is not limited to any sample size or shape except via the coherence length of the laser itself. This in turn is dependent on the frequency stability of the particular laser employed. A typical application is to place reflecting (e.g., mirror) surfaces on, at or near the ends of a sample separated by distances of anything from near zero to about 200m. In most cases it is advisable not to use sample ends. This is because many materials, especially composites, laminates or sandwich structures, have changing stress states near edges or ends and these regions will exhibit different CTE values than internal or bulk regions.

Work is ongoing to modify ASTM D289 accordingly.

6.6.9.1.4 Test apparatus and instrumentation

The three general types of apparatus in use, dilatometers, thermomechanical analyzers and interferometers, are fully described in the corresponding ASTM standards, as already introduced in Section 6.4.9.1.2.

6.6.9.1.5 CTE test methods for MIL-HDBK-17 data submittal

Data produced by the following test methods (Table 6.6.9.1.5) are currently being accepted by MIL-HDBK-17 for consideration for inclusion in Volume 2:

TABLE 6.6.9.1.5 *Coefficient of thermal expansion test methods for MIL-HDBK-17 data submittal.*

Material Type	Property (symbol)	Fully Approved, Interim, and Screening Data	Screening Data Only
Polymer Matrix (unreinforced)	α_m	ASTM E 228 ASTM E 831 ASTM E 289*	ASTM D 696
High Fiber Axial Expansion Composites	α_{11}, α_{22}	ASTM E 228 ASTM E 831 ASTM E 289**	ASTM D 696 (α_{22} only)
Low Fiber Axial Expansion Composites	α_{11} α_{22}	ASTM E 289 ASTM E 228 ASTM E 831 ASTM E 289*	ASTM D 696 ASTM D 696

* This level of resolution is not required.
** This level of resolution is not required for α_{22}

6.6.9.2 Dimensional stability (moisture)

$\beta, \beta_{11}, \beta_{22}$

6.6.9.2.1 Introduction

Dimensional stability due to moisture absorption is customarily defined in terms of a Coefficient of Moisture Expansion (CME), which is expressed symbolically as β (the second letter in the Greek alphabet, following α, which is typically used to symbolically express the Coefficient of Thermal Expansion, CTE, the analogous quantity for thermal dimensional stability). Composites can have different CME's in different directions, whereas unreinforced (neat) polymers typically expand equally in all directions.

Coefficients of moisture expansion of unreinforced polymers are conveniently expressed in units of 10^{-3}/wt%M, whereas CME values for fiber reinforced polymers are expressed in units of 10^{-6}/wt%M or ppm/ΔM. (Coefficients of thermal expansion are typically expressed as 10^{-6}/°C (microstrain/°C). The strains induced due to temperature changes and moisture changes are proportional to $\alpha\ \Delta T$ and $\beta\ \Delta M$, respectively. The effect on dimensional stability of moisture expansion can be considerably greater than the effect of thermal expansion.

The absorption of moisture in polymers and polymer-matrix composites usually causes volumetric expansion (swelling). Most natural fibers, and aromatic polyamide fibers, such as aramid (Reference 6.6.9.2.1(a)), absorb moisture. There are indications that many carbon fibers such as AS4, IM6 and IM7 (References 6.6.9.2.1(b) and (c)) also absorb moisture. In addition, many man-made fibers appear to have negative coefficients of moisture expansion. Polyethylene fibers (e.g., Allied's Spectra) may also absorb moisture but they remain relatively stable dimensionally, particularly in the fiber axial direction, because of the rigid molecular structure. In general, it is the polymer matrix that dictates the amount of swelling due to moisture absorption. For a unidirectional carbon fiber polymer matrix composite, CME values are typically 50-60 parts per million per percent weight change (ppm/ΔM) in the (carbon) fiber direction and 3000-8000 ppm/ΔM in the transverse (cross fiber) direction, and also in the through-thickness direction. For laminates such as quasi-isotropic lay-ups, the in-plane CME values are typically in the 200-500 ppm/ΔM range.

No ASTM or other standard for moisture dimensional stability testing presently exists. A detailed presentation of test procedures, types of apparatuses used, and typical experimental results for both unreinforced (neat) polymers and their composites, is included in Reference 6.6.9.2.1(d). This information is summarized in Reference 6.6.9.2.1(e). Coefficient of Moisture Expansion (CME) measurements for solid laminates are also described in (Reference 6.6.9.2.1(f)), and for composite sandwich panels in (References 6.6.9.2.1(g) and (h)). ASTM C 481 (Reference 6.6.9.2.1(i)), ASTM D 5229 (Reference 6.6.9.2.1(j)), and ASTM E 104 (Reference 6.6.9.2.1(k)) provide assistance with general test techniques, including establishing relevant humidity conditions. The significance of maintaining a uniform moisture content through the laminate thickness when measuring moisture-induced strains is discussed in Reference 6.6.9.2.1(*l*).

The various classes of polymers used as matrix materials, e.g., epoxies, polycyanates, bismaleimides, polyimides, and high temperature thermoplastics (PEEK, PEKK, PPS, PAS, polyamide-imide, etc.) exhibit various magnitudes of coefficient of moisture expansion. However, it is equally important to recognize that there can also be a significant variation of moisture expansion coefficient from one polymer to another within a given class of polymers.

As discussed in Section 6.6.8, the various polymer matrices absorb moisture at significantly different rates (moisture diffusivity), and contain widely differing amounts of moisture at saturation. That is, it is important to recognize that the total influence of moisture on dimensional stability is the product of CME and weight percent moisture absorption; and the rate at which this influence occurs is dependent on the moisture diffusivity. This in turn varies significantly with temperature, stress level and damage state, such as

microcracking. Sudden changes in temperature (thermal spiking) or stress (mechano-absorptive effect) have major effects on properties such as moisture absorption, creep rates, and mechanical stiffness.

6.6.9.2.2 Specimen preparation

Moisture expansion specimens, whether neat polymer or composite, are typically very thin, to minimize the time required to absorb a significant fraction of their moisture equilibrium levels. The moisture diffusion coefficient can be determined from the initial part of a weight change versus time curve, but the coefficient of moisture expansion requires knowledge of the equilibrium or total weight change. A specimen thickness on the order of 0.050 in. (1.27 mm) is typical. The other dimensions of the specimen are made large enough (e.g., 3 – 10 inches long by 1 – 3 inches wide) so that both weight change and dimensional changes can be measured accurately. This specimen geometry, with its large areas on two opposing surfaces combined with minimal edge area, also exhibits the secondary advantage of permitting a one-dimensional diffusion assumption when reducing the experimental data.

Moisture expansion specimens can be fabricated to the thickness required, or machined from thicker material. Surface grinding usually works well for this purpose.

6.6.9.2.3 Test apparatus and instrumentation

Two quantities must be measured to obtain the coefficient of moisture expansion, viz., the total dimensional change and a total weight change. The CME, analogous to the CTE, is a thermodynamic property so a reproducible and equilibrium value of both these quantities must be established. This in turn requires a means for extrapolating partial strain and weight changes to infinite time, where no strain or mass gradients exist in the sample.

One measurement technique is to use two identical specimens in the same environmental chamber, one specimen being weighed on an analytical balance while the length of the other is monitored by some type of dilatometer. An analytical balance weighing to the nearest 0.1 milligram and a dilatometer (or interferometer) measuring microinches are generally suitable. It may be desirable to keep both the analytical balance and the dilatometer electronics outside of the environmental chamber. For example, the weight gain specimen can be suspended inside the chamber by a thin wire attached to the analytical balance. A quartz tube dilatometer can extend into the chamber, with the electronic components remaining on the outside. It is also possible to measure weight and length simultaneously on the same sample. To avoid erroneous readings, it is important that no moisture be allowed to condense on either the specimen or the suspension wire. Small heating elements can be located in these critical locations as required to keep the local temperature slightly higher than the surroundings, thus avoiding condensation in these critical locations.

The specimens should be dried thoroughly, following the general guidelines of Section 6.3 of Volume 1, so that subsequent measurements are referenced to the zero moisture state. Since the rate of moisture absorption increases rapidly with increasing temperature, to reduce the testing time it is customary to perform the test in a heated chamber, while maintaining a prescribed level of relative humidity. This may be as high as 98 %RH (staying sufficiently below 100 %RH so that moisture condensation on the specimen or support wire does not become an overwhelming problem). The acceptable chamber temperature is dictated to some extent by that which is allowable for the type of material being tested. However, since the rate of moisture diffusion is a strong function of temperature, and most polymers swell significantly with absorbed moisture, large moisture (and, therefore, strain) gradients can be induced through the thickness of the specimen, even though it is relatively thin. These strain gradients can induce surface microcracking, leading to erroneous dimensional change measurements (and nonrepresentative moisture weight gains also). For example, for a carbon/epoxy composite, as a general guideline conditioning chamber temperatures do not exceed about 170°F (77°C), with about 150°F (66°C) being a safer upper limit.

Although the use of elevated temperature accelerates the moisture absorption test, there is no assurance that the measured CME is independent of temperature. Thus, a measurement made at one tem-

perature may not be representative of the CME at other temperatures. This can only be determined by testing at multiple temperatures.

For a given test, conducted at a prescribed temperature, the plot of moisture expansion versus moisture content may not be linear, although it usually is relatively so. Since CME is the slope of this plotted curve, it will then not be a constant value.

6.6.9.2.4 CME test methods for MIL-HDBK-17 data submittal

At the present time there is no ASTM or other standard for coefficient of moisture expansion testing. Data submitted for consideration must include a detailed description of the test method.

6.6.10 Thermal conductivity

6.6.10.1 Introduction

The thermal conductivity for polymer-matrix composites is a required thermal response property applicable to all heat flow conditions. Measurement methods are available for either steady state or transient heat flow conditions. The steady state methods are described in this section.

On reaching a steady state, the thermal conductivity λ of a specimen in the thickness direction is determined from the Fourier relation:

$$\lambda = Q/\left(A \bullet \Delta T/L\right) \qquad 6.6.10.1$$

where

Q = heat flow rate in the metered section
A = metered section area normal to heat flow
ΔT = temperature difference across the specimen
L = specimen thickness

The units of the parameters of Eq (6.4.10.1) are:

Q - W
A - m^2
ΔT - K
L - m
λ - W/m•K

Transient methods actually are determinations of the thermal diffusivity, from which the thermal conductivity may be derived, and are described in Section 6.6.12.

6.6.10.2 Available methods

Several ASTM test methods are available for steady state thermal transmission characterizations. They can be categorized as one of two types: as an absolute (or primary) method of measurement in which no heat flux reference standards are required except to confirm accuracy or to establish traceability to recognized standards (C177); or, as a comparative (or secondary) method in which the results are directly dependent on heat flux reference standards (E1225, C518). The methods are briefly described below.

The choice of the measurement method for polymer-matrix laminates often depends upon the measurement direction. Out-of-plane measurements can be performed with the C177 method, but occasionally the E1225 comparative method is used also. In-plane measurements performed on thin laminates require that the specimen diameter be built up by stacking several laminates together. The C177 method is

usually preferred but results on specimens using the E1225 method are occasionally reported. The flash diffusivity method (E1461) is also a viable choice for laminates in either orientation (see Section 6.6.12). Advantages include shorter test times and smaller specimen sizes.

6.6.10.2.1 ASTM C177-97

ASTM C177-97, known as the guarded hot-plate method (Reference 6.6.10.2.1(a)), is an absolute determination method which covers the measurement of heat flux and associated test conditions for flat-slab specimens when their surfaces are in contact with solid, parallel boundaries held at constant temperatures. This test method is good for low thermal conductivity materials and is applicable to a wide variety of specimens and a wide range of environmental conditions.

Figure 6.6.10.2.1 shows the main components of the idealized system: two isothermal cold surface units and a guarded-hot-plate. The guarded-hot-plate is composed of the metered area centerpiece and a concentric guard ring. Some apparatus have a coplanar secondary guard. Sandwiched between these three units is the material to be measured. Figure 6.6.10.2.1 depicts the double-sided mode of measurement, i.e., the specimen is actually composed of two pieces. The measurement in this case produces a result that is an average of the two pieces and, therefore, it is important that the two pieces be as identical as possible. For guidance in the use of the single-sided mode of operation, in which the specimen consists of one piece placed on one side of the hot-surface assembly, see Reference 6.6.10.2.1(b), describing ASTM Practice C1044.

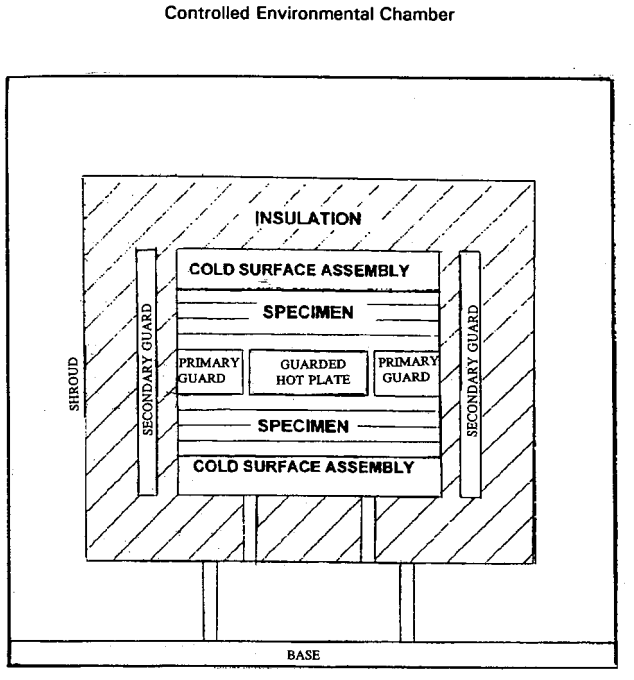


FIGURE 6.6.10.2.1 *General arrangement of the mechanical components of the components of the guarded-hot-plate apparatus (Reference 6.6.10.2.1(a)).*

The arrangement of Figure 6.6.10.2.1 demands that precautions be exercised concerning heat flux losses and proper use of the thermal guard ring, and concerning the accurate measurement of temperature differences and the temperature sensor separation. The guarded-hot-plate provides the power for the measurement and defines the actual test volume, that is, that portion of the specimen that is actually being measured. The function of the primary guard ring is to reduce lateral heat flow within the appara-

tus. The proper (idealized) conditions are illustrated in Figure 6.6.10.2.1 by the configuration of the isothermal surfaces and lines of constant heat flux within the specimen.

Steps must be taken to ensure that the heat flows uniformly into the specimen. Under vacuum conditions the slightest space between plate and specimen is an infinite thermal resistance except for radiative heat transfer. Good thermal contact between the hot and cold assemblies and the specimen surfaces is promoted by applying a reproducible constant clamping force to the guarded-hot-plate apparatus, such as the pressure produced by using constant force springs. Another potential solution is to mount a compressible thin sheet of conductive soft material or fibrous pad between the plates and specimen to improve the uniformity of the thermal contact.

Compliance with this test method requires the establishment of steady-state conditions and the measurement of the unidirectional heat flow Q in the metered section, the metered section area A, the temperature of the hot and cold surfaces, T_h and T_c (in Equation (6.6.10.1) $\Delta T = T_h - T_c$), the thickness of the specimen L, and any other parameters which may affect the heat flux in the metered region.

6.6.10.2.2 ASTM E1225-99

ASTM E1225-99, or the guarded longitudinal heat flow technique (Reference 6.6.10.2.2), is a comparative test method. Hence, reference materials or transfer standards with known thermal conductivities must be used. This test method is for materials with effective conductivities in the approximate range 0.2 $< \lambda <$ 200 W/m•K over the approximate temperature range between 90 and 1300 K. It can be used outside these ranges with decreased accuracy.

The general features of the technique are shown in Figure 6.6.10.2.2. A test specimen is inserted under load between two similar specimens of a material of known thermal properties (meter bars). A temperature gradient is established in the test stack by maintaining the top at an elevated temperature and seating the bottom on a heat sink. Heat losses are minimized by use of a longitudinal guard heater having approximately the same temperature gradient. At steady state equilibrium, the thermal conductivity is derived in terms of the measured temperature gradients in the respective specimens and the thermal conductivity of the reference materials.

The thermal conductance (ratio of thermal conductivity to length) of the reference material should match the thermal conductance of the specimen as closely as possible to ensure similarity in temperature gradients and better accuracy. When the meter bars and the specimen are right circular cylinders of equal diameter the technique is described as the cut-bar method. When the cross-sectional dimensions are larger than the thickness it is described as the flat slab comparative method. Essentially any shape can be used as long as the meter bars and specimen have the same conduction areas.

This test method requires uniform heat transfer at the meter bar to specimen interfaces, which is normally attained by use of an applied axial load in conjunction with a conducting medium at the interfaces. The stack is surrounded by an insulator and enclosed in a guard shell. At steady state, the temperature gradients along the sections are calculated from the measured temperatures along the two meter bars and the specimen. The value of the thermal conductivity in the specimen, λ_s, can then be determined using Equation (6.6.10.2.2) where Z_i = position of a thermocouple as measured from the upper end of the column (see Figure 6.6.10.2.2), T_i = the temperature at position Z_i, λ_m^1 = the thermal conductivity of the top meter bar, and λ_m^2 = the thermal conductivity of the bottom meter bar.

$$\lambda_s = [(Z_4 - Z_3)\,\lambda_m^1\,(T_2 - T_1)]/[(T_4 - T_3)\,2\,(Z_2 - Z_1)] + [\lambda_m^2\,(T_6 - T_5)]/[2\,(Z_6 - Z_5)] \qquad 6.6.10.2.2$$

This result is a highly idealized situation since it assumes no heat exchange between the column and insulation and uniform heat transfer at each meter bar-to-specimen interface. The errors caused by these assumptions are discussed in Reference 6.6.10.2.2.

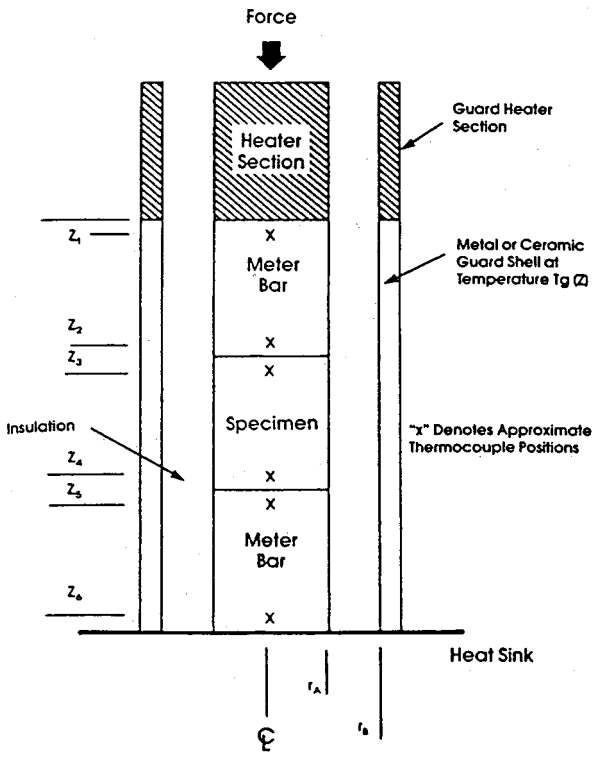


FIGURE 6.6.10.2.2 *Schematic of a comparative-guarded-longitudinal heat flow system showing possible locations of temperature sensors.*

6.6.10.2.3 ASTM C518-98

ASTM C518, Reference 6.6.10.2.3(a), describes the measurement of the steady state thermal transmission through flat slab specimens using a heat flow meter apparatus. This is a comparative, or secondary, method of measurement since specimens of known thermal transmission properties are required to calibrate the apparatus. The test applies to low conductivity materials. To meet the requirements of this test the thermal resistance of the test specimen should be greater than 0.10 m^2•K/W in the direction of the heat flow and edge heat losses should be controlled using edge insulation and/or a guard heater.

The important features of the heat flow meter apparatus are two isothermal plate assemblies, one or more heat flux transducers, and equipment to measure temperature and the output of the heat flux transducers. Either one or two specimens are used. Three common experimental configurations are depicted in Figure 6.6.10.2.3. Equipment to control the environmental conditions is employed when needed.

A heat flux transducer is a device that produces a voltage output which is a function of the heat flux passing through it. The various types of heat flux transducers are described in Test Method C1046, Reference 6.6.10.2.3(b). The gradient type, commonly used in Test C518-98, consists of a core across which the voltage is measured, normally with a thermopile. Appropriate calibration of the heat flux transducer(s) with calibration standards, and accurate measurement of the plate temperatures and plate separation are required. The procedures are detailed in C518-98.

The experimental procedure is to establish a steady state unidirectional heat flow through the test specimen(s), held between the two isothermal parallel plates, a hot plate and a cold plate. The heat flow

rate, Q, is obtained from the measured voltage output over the heat flux transducer. Equation (6.6.10.1) is then applied to calculate the thermal conductivity through the determination of Q, the separation between the hot and cold plates, L, the cross-sectional area, A, and the temperature difference across the specimen, ΔT.

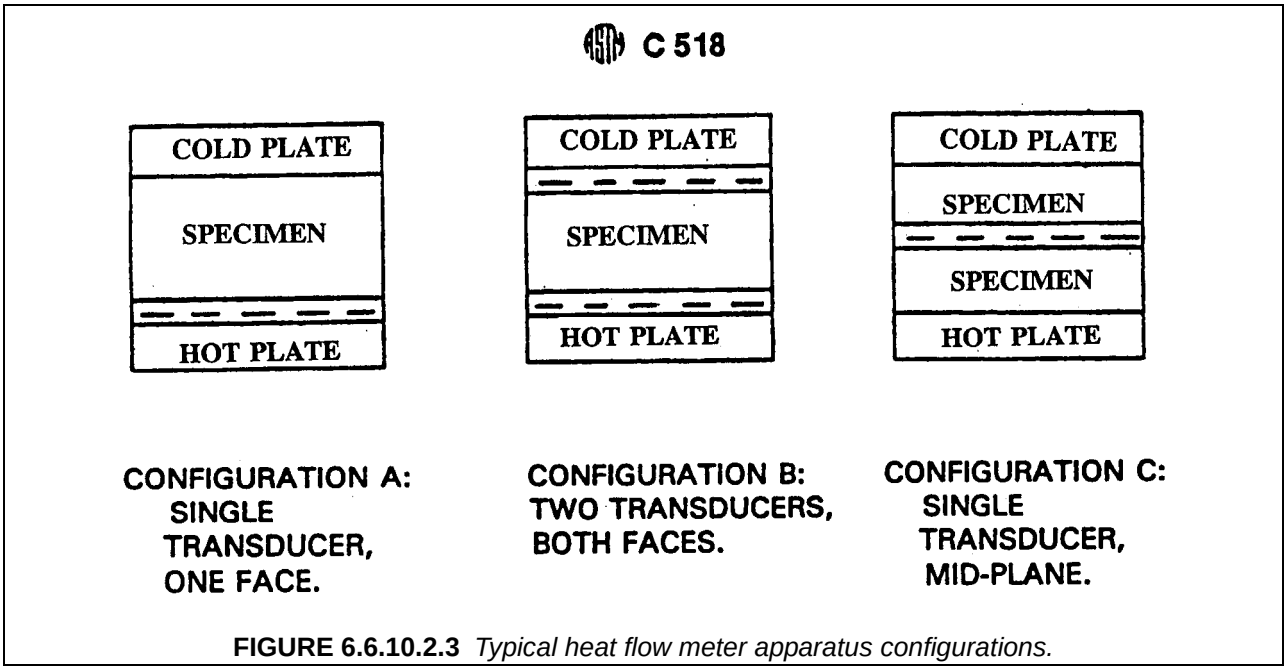


FIGURE 6.6.10.2.3 *Typical heat flow meter apparatus configurations.*

The C518-98 method has been utilized at ambient conditions of 10 to 40°C with specimen thickness up to approximately 25 cm and with plate temperatures from -195°C to 540°C at 2.5 cm thickness. Test Method E1530-93, Reference 6.6.10.2.3(c), is similar in concept to C518-98 but is modified to accommodate smaller test specimens having a higher thermal conductance. This method is relevant to specimens having a thickness less than 1.2 cm with a thermal conductivity in the range $0.1 < \lambda < 5$ W/m•K.

6.6.10.2.4 Fourier thermal conductivity test method for flat plates

One additional procedure applicable to thermal conductive panel materials will be given here. The method is not an ASTM standard. This test method is specifically tailored to determine the thermal conductivity of material whose thickness is much less than its lateral dimensions and which exhibits a thermal conductivity of at least 30 W/m•K. The upper limit can be as high as 1500 W/m•K depending on sensor location and geometry. Although the method is an absolute one as written, it is strongly recommended that results be correlated to a known standard of equivalent size tested with the same experimental arrangement.

This method evaluates the steady state one dimensional heat transfer characteristics in terms of the differential temperature as a function of distance. This is accomplished by acquiring the necessary data off the face of the panel as heat flows from one end of the panel where heat is applied, to the other end where heat exits via a heat sink. This method is applicable to panels where the cross sectional area of heat flow remains constant. Thermal conductivity is calculated using Equation (6.6.10.1).

The goal of this method is to satisfy the need for non-destructive thermal conductivity measurements that determine the overall thermal performance of the component rather than assessing only localized values. Multiple simultaneous measurements allow data to be generated over a large area. This is par-

ticularly important for materials such as composites where the distribution of the conductive reinforcement may vary locally on the component. The non-destructive nature of this method is accomplished through the use of removable heaters and temperature sensors. Also, the use of a liquid immersion heat sink allows for various sized components to be immersed without any machining or specimen sizing. This is particularly important where expensive or end use hardware is being evaluated.

This method also lends itself to being useful in evaluating materials that exhibit anisotropic properties. The method, which employs unidirectional heat transfer, allows for easy measurement of directional properties without the need for complex data reduction.

The apparatus consists of a liquid immersion heat sink; liquid chiller (5°C to 30°C range); Kapton laminated thermal-foil heaters; platinum resistive temperature devices (RTD's); pressure sensitive film adhesive; coated fiberglass insulation (five cm thick and with a thermal conductivity less than 0.1 W/m•K); a well regulated dc power supply (e.g., not less than 60 V dc at a current of at least 2 A); instrumentation for accurately measuring the voltage and current; and signal conditioning for the temperature sensors. The use of a computer data acquisition system is optional but desirable.

The liquid immersion heat sink must be a closed loop system that is hooked to a chiller which will allow near infinite sinking of heat from the test specimen. Figure 6.6.10.2.4(a) shows a schematic view of the system. This system acts as a heat exchanging device in which the specimen is clamped. A suitable liquid such as an equal parts mixture of ethylene glycol and water should be used for the recirculating coolant.

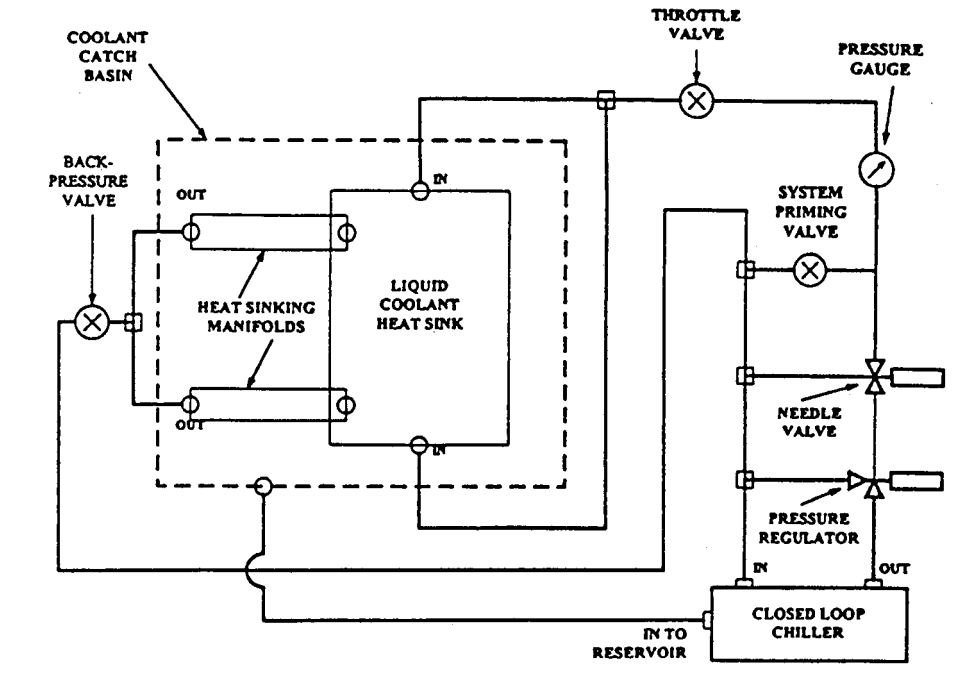


FIGURE 6.6.10.2.4(a) *Plumbing schematic of the MII liquid immersion heat sink.*

The heat source should employ foil heaters with densely spaced elements. These heaters should be able to tolerate at least 200°C. Aluminum backing may be necessary for anisotropic materials exhibiting poor heat spreading. This will insure uniform heat flow along the specimen surface.

The instrumentation should be capable of measuring voltage with a resolution of 1 mV; measuring current with a resolution of 1 mA; measuring temperatures with a resolution of 0.01°C; and the ability to transfer data to a data acquisition system if used.

The test specimen should be in a rectangular or square configuration with the sides 90E relative to each other. The cross sectional area in any place should not deviate more than 5% from the average cross sectional area. All surfaces should be clean from dirt and oils. It is recommended that the minimum width be at least one inch (25 mm), the minimum thickness at least 0.075 cm, and the minimum length in the direction of heat flow be at least ten cm. Larger surface dimensions will typically yield additional temperature resolution in the measurement. Smaller surface areas should be analyzed for adequate resolution prior to testing.

Thermal foil heaters and RTD's are attached to the surface via a thermally conductive pressure sensitive adhesive. In cases where the specimen thickness exceeds 0.50 cm heating end-on is required. This procedure is required in order to avoid temperature gradients through the thickness of the test specimen in the temperature measurement area. A typical arrangement is shown in Figure 6.6.10.2.4(b).

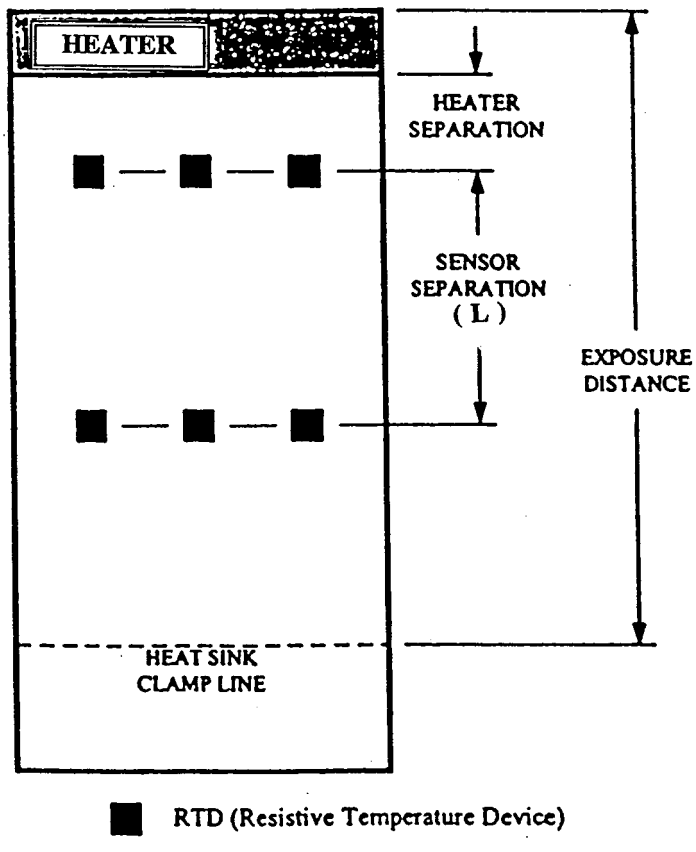


FIGURE 6.6.10.2.4(b) *Thermal conductivity specimen sensor location schematic.*

The heating location should be at the top edge of the panel with heaters mounted on both faces of the test specimen. The RTD's are mounted as two rows. The top row needs to be at a measured distance from the heater such that the RTD's are observing uniform heat flow. RTD's too close to the heater may be influenced by heat input effects. The bottom row needs to be a measured distance from the top row such that the temperature gradient during the test exceeds 1.50°C. Both rows of RTD's, as a set, should be placed approximately half way between the heat source and heat sink. It is important to ensure that there are no air bubbles or delaminations in the adhesive bond line.

All wiring should exit the specimen surface horizontally such that temperature gradients along the wire near the specimen are minimized. This will minimize thermal shorts.

The test procedure is to first measure all necessary dimensional data. This survey should include at least five measurements of thickness and width; measurement of RTD spacing relative to the heating elements; and reference data such as test distance, horizontal RTD spacing, and overall panel length.

Then immerse the test specimen into the liquid cooled heat sink and clamp between the heat sinking manifolds. At least one inch (25 mm) of panel material should be in contact with the liquid coolant heat sink to insure adequate heat sinking.

Adequately insulate the test specimen's exposure distance with fiber insulation. This step is required to insure an adiabatic system at equilibrium.

Allow the test stand to equilibrate at the heat sinking temperature. Record all initial RTD temperatures at the heat sinking temperature. These data will be used to normalize any zero offset of the sensor.

Energize the test specimen to a heat flux of approximately 100 kW/m^2. Other heat fluxes can be used based on the thermal conductivity of the test specimen and the test temperature that is targeted. The only requirement on determining heat flux is that there must be enough power for adequate resolution of the temperature gradients and that heat losses relative to total heat input be insignificant.

Record final temperatures and power parameters upon equilibrium at the heated state. Equilibrium should be defined as less than a degree change in temperature for every ten minutes of test time on all temperature sensors.

The record of test data should include all information describing the material; all dimensional data; initial and final temperatures taken at steady state conditions; voltage and current data; ambient and heat sink temperatures; and the average of all final RTD temperatures.

Thermal conductivity is calculated from Equation (6.6.10.1). The quantity Q is obtained as the product of the voltage and current of the heater. The cross-sectional area A is calculated as the thickness and width product. Separation of the two lines of RTD's in the heat flow direction, L, is measured directly. The temperature data are analyzed by first converting each data point value recorded by an individual RTD to a normalized value. The normalization is accomplished by taking the difference in temperature between the initial (steady state) measurement at the heat sink temperature and the final (steady state) measurement at the heated test temperature. The normalized values are averaged for each of the two rows of RTD's. Taking the temperature difference of the averaged normalized values of the two rows of RTD's yields the change in temperature ΔT to be used in Equation (6.6.10.1).

The final report should include the thermal conductivity in SI units (W/m•K), the average test temperature, and the ambient temperature.

Results must be correlated to a known "standard" of equivalent size tested with the same experimental arrangement, conditions, and power parameters. The "standard" should be a widely accepted material which has been well characterized and should have a thermal conductivity as close as possible to the test

specimen. (Typical “standards” are plates of aluminum or copper.) The conductivity result is normalized by using the correction factor (CF) given in the following equation.

CF = [(accepted value of “standard”)/(measured value of “standard”)]*[measured value of unknown]

Typical normalization of test results usually requires a correction of 3% to 6%, which accounts for heat losses in the wiring and through the insulation. Heat losses in excess of 10% should be considered questionable and a detailed analysis should be performed prior to acceptance of data.

6.6.10.3 Thermal conductivity test methods for MIL-HDBK-17 data

Data produced by the following test methods (Table 6.6.10.3) are currently being accepted by MIL-HDBK-17 for inclusion in Volume 2.

TABLE 6.6.10.3 *Thermal conductivity test methods for MIL-HDBK-17 data submittal.*

PROPERTY	SYMBOL	FULLY APPROVED, INTERIM AND SCREENING DATA
Thermal Conductivity	λ	C 177 E 1225 C 518 Fourier Test Method

6.6.11 Specific heat

6.6.11.1 Introduction

The definition of specific heat is the change in the internal energy of a material per degree temperature change per unit mass of material. In practice, the specific heat at constant pressure or enthalpy, C_p, is the measured quantity, with values reported in J/(kg•K) in SI units.

6.6.11.2 Available method

The standard test method for measuring the specific heat of polymer matrix composites is ASTM E1269-95, Reference 6.6.11.2, and is based on Differential Scanning Calorimetry (DSC). This test is generally applicable to thermally stable solids and has a normal operating range from -100 to 600 EC. The temperature range can be extended depending upon the instrumentation and specimen holders used.

6.6.11.2.1 ASTM E1269-95

A brief summary of the DSC test method is as follows. Empty aluminum pans are placed in the specimen and reference holders. An inert gas atmosphere, such as nitrogen or argon, is typically used as the blanketing atmosphere. An isothermal baseline is recorded at the lower temperature and the temperature is then increased by adding heat, Q (W), in a programmed manner over the range of interest. An isothermal baseline is recorded at the higher temperature, as indicated in the lower part of Figure

6.6.11.2.1, Reference 6.6.11.2.1. The procedure is then repeated with a known mass of specimen, M, in the specimen pan and a trace of energy absorbed against time is again recorded.

The data so produced at this point are theoretically sufficient to calculate the specific heat of the specimen, but in practice a calibration procedure is important and is discussed below. Heating rates in the range of 5-20°C/min are recommended with a rate of 10°C/min being commonly employed.

A quantitative measurement of energy imparted to a test specimen as a function of temperature must be obtained to determine specific heat. Thus, the instrument used for these measurements must be calibrated in both the heat flow and temperature modes.

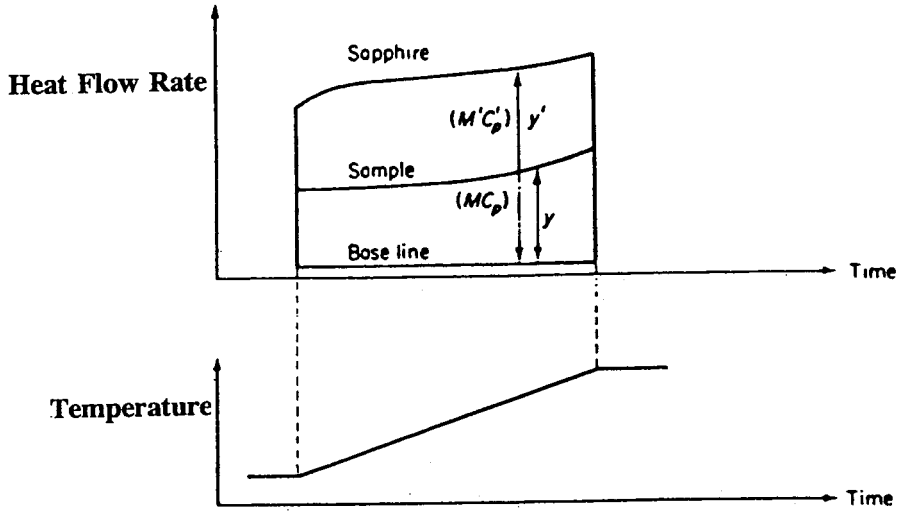


FIGURE 6.6.11.2.1 *Specific heat determination by ratio method.*

Since specific heat is not a quickly changing function of temperature, the instrument's temperature mode is ordinarily calibrated and checked only occasionally. Temperature calibration is achieved by observing the melting transition of reference materials. This calibration should be performed over the temperature range to be covered in the unknown specimen specific heat measurement. Materials suitable for use as DSC temperature calibration standards are listed in Table 6.6.11.2.1 (Reference 6.6.11.2).

The heat flow information is critical and calibration in this mode is achieved through the use of a standard material whose specific heat is well established. The calibration procedure is known as the Ratio Method. The recommended standard material is synthetic sapphire (α-aluminum oxide). Specific heat capacities for synthetic sapphire, C_p', are given in Reference 6.6.11.2.

The thermal scan procedure described above is now repeated with a known mass of sapphire, M', and a new trace is recorded. The two ordinate deflections at the same temperature, Figure 6.6.11.2.1, and the mass ratios are used to calculate the specimen specific heat, according to the formula

$$C_p = C_p' \bullet (y / y') \bullet (M' / M) \qquad 6.6.11.2.1$$

where

y = vertical displacement between the specimen holder and the specimen thermal curves at a given temperature

y' = vertical displacement between the specimen holder and the sapphire thermal curves at a given temperature

Notable features of the DSC method are comparatively short test times and milligram specimen sizes. Since such small quantities of specimen material are used, it is essential that specimens be homogeneous and representative. The latter condition may be difficult to achieve if specimens are removed from a polymer matrix composite panel of large size due to manufacturing variabilities from one area of the panel to another. The problem may be addressed by measuring a number of specimens taken from different panel locations and averaging the results.

TABLE 6.6.11.2.1 *Melting temperature of calibration material.*

Calibration Material	Melting Temperature	
	(°C)	(K)
Benzoic Acid	122.4	395.5
Indium	156.6	429.8
Tin	232.0	505.1
Lead	327.5	600.7
Zinc	419.6	692.7

Application of the DSC test method to polymer matrix composites may also encounter difficulties due to specimen mass loss from either moisture evolution or material decomposition, but this problem can be overcome by taking proper precautions.

6.6.11.3 Specific heat test methods for MIL-HDBK-17 data submittal

Data generated by DSC, Procedure E1269, are currently being accepted by MIL-HDBK-17 for consideration for inclusion in Volume 2.

PROPERTY	SYMBOL	FULLY APPROVED, INTERIM, AND SCREENING DATA
Specific Heat	C_p	E1269

6.6.12 Thermal diffusivity

6.6.12.1 Introduction

Thermal diffusivity is a thermal response property of a material derived from transient heat-flow conditions. If the density and specific heat are known, the thermal diffusivity, α, may be used to determine the thermal conductivity of a material from the relationship

$$\lambda = p \bullet c_p \bullet \alpha \qquad 6.6.12.1$$

where

λ = thermal conductivity
ρ = density
C_p = specific heat

The units of the parameters of Equation (6.4.12.1) are

λ = W/m•K
ρ = kg/m^3
C_p = J/kg≅K
α = m^2/s

6.6.12.2 Available test methods

A standard test method, the flash method, ASTM E1461-92, exists for the determination of thermal diffusivity of homogeneous opaque solid materials (Reference 6.6.12.2(a)). With special precautions, the method can also be used on some transparent and composite materials. Thermal diffusivity values ranging from 0.1 to 1000 mm^2/s have been measured by this technique and measurements can be made from about 100 to 2500 K, normally in a vacuum or inert gas environment. The flash method is the most common method reported in the literature for measurement of thermal diffusivity of polymer-matrix composites.

Test E1461-92 is a more detailed form of Test Method C714, Reference 6.6.12.2(b), but has applicability to much wider ranges of materials, applications, and temperatures with improved accuracy of measurement. The C714 method applies only to carbon and graphite.

6.6.12.2.1 ASTM E1461-92

This test method is considered an absolute method of measurement since no heat flux reference standards are required. The essential features of the apparatus used in the flash method are shown in Figure 6.6.12.2.1(a). These are the flash source, sample holder and environmental control chamber, temperature response detector, and data collection and analysis system. The flash source may be a laser, a flash lamp, or an electron beam. The usual specimen is a thin circular disc with a front surface area less than that of the flash beam. The initial temperature of the specimen is controlled by a furnace or cryostat. The detector can be a calibrated thermocouple attached to the rear face of the specimen or an infrared sensor or optical pyrometer focused on the rear face and filter protected from the flash beam.

To conduct the flash test, the source is pulsed on the front surface of the specimen and energy is absorbed by the specimen. The resulting rear face temperature rise is recorded. The measured temperature rise curve is examined to determine the base line temperature, the maximum temperature rise, ΔT_{max}, and the time of initiation of the thermal pulse.

Thermal diffusivity values are calculated from the specimen thickness L and the time required for the rear face temperature to reach a certain percentage of its maximum value. The equation is

$$\alpha = k_x \bullet L^2 / t_x \qquad 6.6.12.2.1(a)$$

where k_x is a constant corresponding to x% temperature rise and t_x is the time required for the temperature rise to reach x percent of ΔT_{max}. Values of k_x are given in Table 6.6.12.2.1(a), taken from Reference 6.6.12.2(a). The rear face temperature rise is typically 1 to 2 Kelvin.

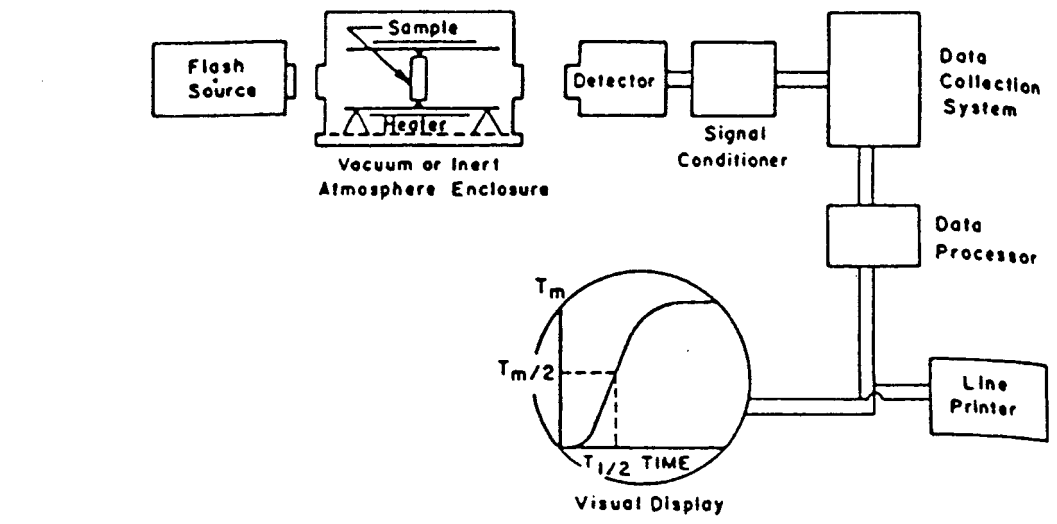


FIGURE 6.6.12.2.1(a) *Flash diffusivity apparatus (schematic).*

TABLE 6.6.12.2.1(a) *Value of the constant k_x for various percent rises.*

x(%)	k_x	x(%)	k_x
10	0.066108	60	0.162236
20	0.084251	66.67	0.181067
25	0.092725	70	0.191874
30	0.101213	75	0.210493
33.33	0.106976	80	0.233200
40	0.118960	90	0.303520
50	0.13879	…	…

Commonly, the half-rise time (one half the time to reach ΔT_{max}) is used in which case Equation (6.6.12.2.1(a)) becomes

$$\alpha = 0.13879 \bullet L^2/t \qquad 6.6.12.2.1(b)$$

The experimental results may be normalized in temperature rise and in half-rise time and compared to a theoretical model to check for effects of finite pulse time, radiation heat loss, or non-uniform heating. This is done by dividing the temperature rise by the maximum rise, thus non-dimensionalizing the ordinate. Times are divided by the half time to non-dimensionalize the abscissa. The values of normalized temperature versus time for the theoretical model are given in Table 6.6.12.2.1(b). Examples of the normalized plots are shown for an experiment that approximates the ideal case, Figure 6.6.12.2.1(b). Tests in which there is a finite pulse time effect and in which there are radiation heat losses are depicted in Figures 6.6.12.2.1(c) and (d), respectively. After examination of the temperature response data for the test specimen, any needed corrections are made following the procedures outlined in Reference 6.6.12.2(a).

TABLE 6.6.12.2.1(b) *Value of normalized temperature versus time for theoretical model.*

$\Delta T / \Delta T_{max}$	$t / t_{1/2}$	$\Delta T / \Delta T_{max}$	$t / t_{1/2}$
0	0	0.7555	1.5331
0.0117	0.2920	0.7787	1.6061
0.1248	0.5110	0.7997	1.6791
0.1814	0.5840	0.8187	1.7521
0.2409	0.6570	0.8359	1.8251
0.3006	0.7300	0.8515	1.8981
0.3587	0.8030	0.8656	1.9711
0.4140	0.8760	0.8900	2.1171
0.4660	0.9490	0.9099	2.2631
0.5000	1.0000	0.9262	2.4091
0.5587	1.0951	0.9454	2.6281
0.5995	1.1681	0.9669	2.9931
0.6369	1.2411	0.9865	3.6502
0.6709	1.3141	0.9950	4.3802
0.7019	1.3871	0.9982	5.1102
0.7300	1.4601	…	…

Typically, specimens are 0.2 to 0.7 in. (0.6 to 1.8 cm) in diameter, with thickness in the 0.06 to 0.16 in. (0.15 to 0.4 cm) range. Thinner specimens are desired at higher temperatures to minimize heat loss corrections. The optimum specimen thickness depends on the magnitude of the estimated diffusivity and should be chosen so that the time for the rear face to reach one-half of its maximum value falls within the 40 to 200 ms range. The duration of the energy flash should be less than 0.02 of the time for the rear face to reach one-half of its maximum value. If this condition is not met, the data must be corrected for the finite pulse time effect.

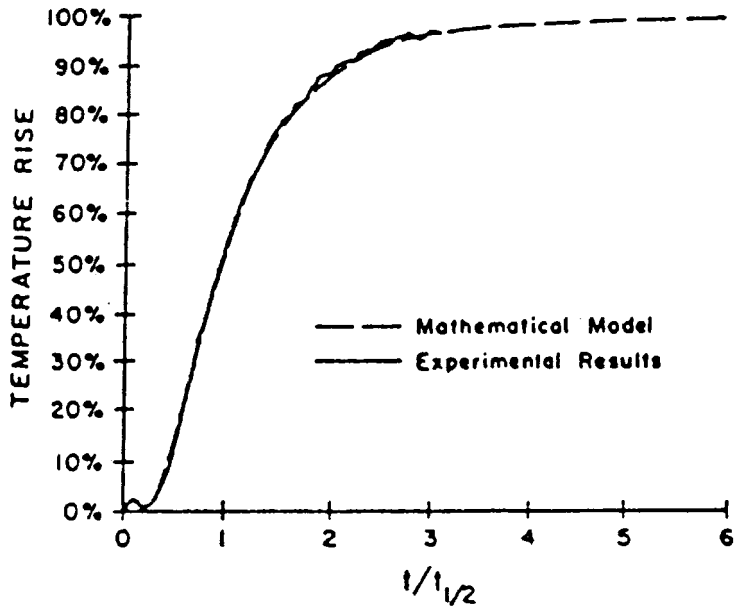


FIGURE 6.6.12.2.1(b) *Comparison of non-dimensional temperature response curve to mathematical model.*

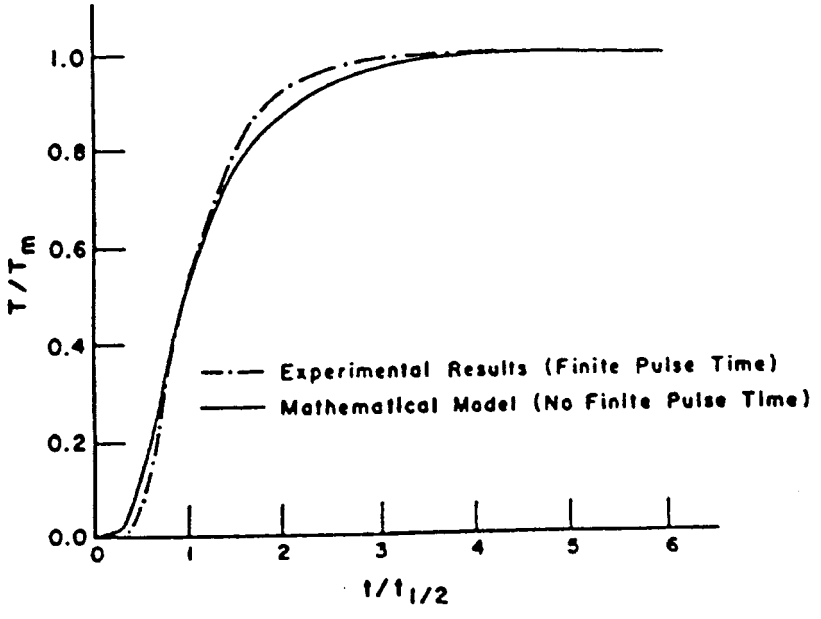


FIGURE 6.6.12.2.1(c) *Normalized rear face temperature rise: comparison of mathematical model (no finite pulse time effect) to experimental values with finite pulse time.*

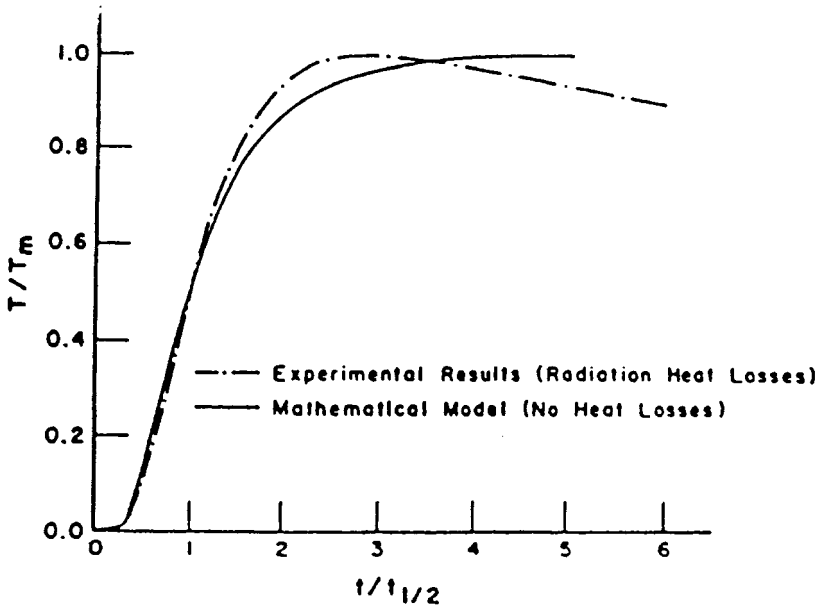


FIGURE 6.6.12.2.1(d) *Normalized rear face temperature rise: comparison of mathematical model (no heat loss) to experimental values radiation heat losses.*

Round robin test arrangements have shown that a measurement precision of ∀5% can be attained for the thermal diffusivity of a variety of materials. Two major sources of experimental uncertainty exist. One lies in the determination of L. This uncertainty is significant because test specimens are relatively thin and the thickness enters as a squared term. A second source is the response time of the detector and its associated amplifiers, which must be no more than 0.1 of the half time value. In general, optical instruments have an acceptable response time. Thermocouples tend to be slower and should be carefully checked for response time against a calibrated source or chopped beam.

Advantages of the flash method are the simple specimen geometry, small specimen size, rapidity of measurement, and ease of handling materials having a wide range of thermal diffusivity values with a single apparatus. Furthermore, the short measurement time reduces chances of contamination and change of specimen properties due to exposure to high temperatures. The flash method has been extended to two-dimensional heat flow so that large samples can be measured and the diffusivity in both the axial and radial directions can be measured.

Problems that can arise when applying the flash diffusivity method are: (1) partial transparency to the light beam exhibited by a specimen material, and (2) different magnitudes of heat transmission manifested by the components of a multi-phase specimen material, such as the reinforcement fiber and matrix of a composite. The first situation is commonly dealt with by coating the front surface of the specimen with a thin layer of a light absorbing material, e.g., graphite. If the second situation exists, the thermal pulse tends to move preferentially through the component phase having the higher thermal diffusivity, with the result that the temperature profile may be non-planar at the specimen rear surface and depart noticeably from the theoretical model. This effect is sometimes observed in practice for composites having a large fraction of high thermal conductivity fibers oriented along the heat flow direction. In this event, the flash method is not applicable.

6.6.12.2.2 ASTM C714-85

This test method covers the determination of the thermal diffusivity of carbon and graphite to ∀5 percent at temperatures up to 500EC. It requires a circular disk specimen of the order of one cm diameter and one half cm thick. The method has the sensitivity to analyze very low sulfur contents in graphite using small samples and, therefore, is relevant to nuclear reactors where sulfur, even in low concentrations, is a concern.

The method is summarized as follows. A high-intensity short-duration thermal pulse from a flash lamp is absorbed on the front surface of a specimen and the rear surface temperature change as a function of time is recorded. Thermal diffusivity is calculated from the specimen thickness and the time required for the temperature of the back surface to rise to one half of its maximum value, Equation (6.4.12.2.1(b)). The theoretical considerations and experimental caveats of Test E1461-92 apply directly to Test C714-85 and should be consulted at this point, Reference 6.6.12.2(a).

6.6.12.3 Thermal diffusivity test methods for MIL-HDBK-17 data submittal

Data generated by the flash method, Procedure E1461, are currently being accepted by MIL-HDBK-17 for consideration for inclusion in Volume 2.

PROPERTY	SYMBOL	FULLY APPROVED, INTERIM, AND SCREENING DATA
Thermal Diffusivity	α	E1461

6.6.13 Outgassing

Space-based optical payloads and components are exposed to a wide variety of particulate and molecular contamination sources. Many sources of this contamination are not within the scope of this handbook; but the molecular contamination created by outgassing of materials must be analyzed when choosing or specifying materials. Molecular contamination can lower the power output of the solar arrays, and can drastically decrease the throughput of optical components, particularly in the ultraviolet (UV). For example, the Hubble Space Telescope's WF/PC-1 pickoff mirror, returned by the first servicing mission, had developed a thick molecular contamination that had been photopolymerized by exposure to earth atmosphere. As a result, the reflectance at the UV wavelength 1216Å dropped from 0.72 to 0.005. While any molecular contaminant on a mirror tends to lower the UV reflectance, experiments and flight data have shown that the reflectance is especially degraded when the contaminated mirror is exposed to ultraviolet light of sufficient intensity (Reference 6.6.13(a)).

Contamination control engineering is a discipline under the continuous support of NASA. A good introduction is given in Reference 6.6.13(a). There are two ASTM test methods that measure the amount of molecular contamination likely to be produced by outgassing from the tested material. ASTM E 1559 is used to obtain data useful in modeling outgassing and contamination in a designed optical system. ASTM E595 is primarily a screening technique, and is used by NASA to construct tables that help material selectors sort out likely contaminators. (Reference 6.6.13(b)).

ASTM E 595 (1999), "Standard Test Method for Total Mass Loss and Collected Volatile Condensable Materials from Outgassing in a Vacuum Environment"

In this test, the material is first ground to a powder and brought to a standard moisture content. Then the material is placed in the apparatus. The sample is held at 257°F (125°C) in a vacuum lower than 7×10^{-3} Pa (5×10^{-5} torr) for 24 h., after which two parameters are measured: total mass loss (TML) and

collected volatile condensable materials (CVCM). CVCM is that mass which condenses on a plate held at 77°F (25°C). An additional parameter, the amount of water vapor regained (WVR), can also be obtained after completion of exposures and measurements required for TML and CVCM. This test method is primarily a screening technique for materials and is not valid for computing actual contamination on a system or component, because of differences in configuration and temperatures. In NASA RP 1124 (Reference 6.6.13(b)), a distinction is made by listing in Section C only those materials having TML and CVCM equal to or lower than a maximum 1.0 percent TML and a maximum 0.10 percent CVCM. Choosing, where possible, materials with TML and CVCM lower than these limits is always recommended and may be required, depending on the program.

ASTM E 1559-93, "Standard Test Method for Contamination Outgassing Characteristics of Spacecraft Materials".

This ASTM test method can be used for measuring the outgassing rate data that are necessary to develop kinetic expressions for use in models that predict the evolution of molecular contaminants and the migration and deposition of these contaminants on spacecraft surfaces. These mathematical models are described in Reference 6.6.13(a).

The measurements are made by placing the material sample in an effusion cell so that the outgassing flux leaving the cell orifice will impinge on three Quartz Crystal Microbalances (QCMs) which are arranged to view the orifice. A fourth QCM is optional. The effusion cell is held at a constant temperature in the high vacuum chamber and has a small orifice directed at the QCMs. The QCMs are controlled to selected temperatures. The total outgassing rate is determined from the collection rate on a cryocooled QCM. At the end of the isothermal test, the QCMs are heated in a controlled manner in order to determine the evaporation characteristics of the deposits.

While E595 makes one total measurement each of TML and CVCM, E1559 measures the mass loss of the sample over time, the mass condensed at several different temperature QCMs over time, and the mass evaporated from these QCMs over time as the temperature is raised. The mass loss and condensed material are measured indirectly, as a function of the resonant frequencies of the QCMs.

Test method A is the standard procedure using prescribed configurations and temperatures. An online database that includes this data for many materials (Reference 6.6.13(c)) is maintained by the Marshall Space Flight Center. Test Method B allows for the use of spacecraft system specific temperatures, configurations, and QCM collector surface finishes.

6.6.14 Absorptivity and emissivity

This section is reserved for future use.

6.6.15 Thermal cycling

This section is reserved for future use.

6.6.16 Microcracking

This section is reserved for future use.

6.6.16.1 Introduction

This section is reserved for future use.

6.6.16.2 Microcracking due to the manufacturing process

This section is reserved for future use.

6.6.16.3 Microcracking due thermal cycling

This section is reserved for future use.

6.6.16.4 Microcracking due to mechanical loading/cycling

This section is reserved for future use.

6.6.17 Thermal oxidative stability (TOS)

This section is reserved for future use.

6.6.18 Flammability and smoke generation

6.6.18.1 Introduction

A significant concern in any application of organic matrix based composites in occupied spaces is the possibility that an accidental (or deliberate) fire may impinge on the structure. This is potentially problematical for two reasons. First, heat weakens the polymer binder. Thermoplastic binders begin to creep and then to flow as the impinging flames raise their local temperature past the glass transition temperature. Thermoset binders degrade to a char or gasify or both. The functioning of the binder is thus diminished and the composite loses strength. If the structure is one in which the composite forms only a secondary or repair role, the consequences of a local, heat-induced composite failure are not likely to be serious; time is available to repair the damaged material. However, if the affected composite component is part of a primary critical structure, such as the wing of an aircraft, the structure may collapse.

The second aspect of the problem can greatly magnify the first. The binder may ignite and support the spread of flame on the composite surface and also release heat and generate potentially toxic smoke. Thus the localized, external fire may cause a larger structural fire involving the composite which now becomes the fuel for the growing fire. In confined or enclosed spaces such as ships and aircraft, the growing fire could lead to a flashover condition in which all combustible materials within the enclosure begin to burn. In open spaces such as bridges, a growing fire clearly increases the chance of structural collapse. Again the consequences are less threatening when the composite merely serves a secondary role as opposed to being a primary structure. For earthquake reinforcement, the problem is somewhat more complex. Fires accompany earthquakes but they tend to lag the initial shock. If a quake induced fire did destroy the composite reinforcement on a structure, the structure might readily survive the initial quake only to fall victim to an aftershock occurring after the fire.

Compared to many flammable materials, composites have a built-in advantage that helps resist the worst consequences (extensive fire involvement). This is a result of their (usually) inert fiber content of as high as 70% by weight in some cases. The fibers displace polymer resin, making less fuel available to the fire. When the outermost layers of a composite lose their resin due to heat induced gasification, they act as an insulating layer, slowing heat penetration into and evolution of gases from the depth of the composite (References 6.6.18.1(a)-(c)).

6.6.18.2 Fire growth test methods

For most applications of composites, fire growth potential should be the first issue addressed and overcome for habitable environments. Rather surprisingly, this issue has received relatively little attention, except for a limited number of compartment fire growth studies. Much of the sparse work on fire spread on the surface of a composite has employed tests for lateral or downward flame spread. These are relatively slow modes of fire growth and they differ mechanistically from upward flame spread, which tends to be much faster. Good performance in the lateral/downward mode does not necessarily imply good performance in upward spread. The converse, however, is likely to hold true, i.e., resistance to upward spread should carry over to yield resistance to lateral or downward spread.

Suppression of fire growth potential calls for measures which either preclude the heat from an external fire getting to the surface of a composite or which dampen the inherent response of the resin to this heat. At one extreme is total fire insulation of the composite. This has been suggested as a solution for both the hazard of fire involvement and for the threat of structural collapse. A sufficiently thick layer (e.g., 1.97 in. (5 cm)) of fiber insulation can keep the temperature of the composite below its ignition temperature (reducing hazard of fire involvement) and also below its glass transition temperature for periods of 30 minutes or more (reducing threat of structural collapse).

Flame retarded resins are a potential solution to fire growth problems but they only lessen the flammability of a composite. This translates into resistance to a bigger external fire source before fire growth ensues. In unpublished NIST tests, brominated vinyl ester/glass composites exhibited essentially unchanged ignition behavior but required somewhat stronger external heat fluxes to sustain full height flame spread (3.94 ft. (1.2 m)); the increase was from 3-5 kW/m^2 to approximately 10 kW/m^2. Whether this is sufficient depends on the use of the composite and the ignition sources it is likely to experience. Choice of a strongly charring resin such as a phenolic can provide greater benefits if other properties are compatible with the application.

As noted previously, intumescent coatings are an established fire protection technology for non-composite applications. Limited work has been done on their ability to protect composites. These studies looked at the ability of various coatings, including certain intumescents, to delay ignition, lower the rate of heat release, suppress lateral flame spread, and extend the duration of fire resistance of composites in a standard temperature-time exposure. These studies revealed that only a limited minority of commercial coatings have the needed ability to remain in place during intense heat exposures characteristic of large fires (References 6.6.18.2(a)-(c)).

Flammability requirements for transport aircraft are prescribed in Federal Aviation Regulation 25.853. Test methods, additional requirements and other information needed to implement the methods are contained in “Aircraft Materials Fire Test Handbook” DOT/FAA/AR-00/12 (Reference 6.6.18.2(d)).

6.6.18.2.1 ASTM E 84 - Surface burning characteristics of building materials
(Reference 6.6.2.1)

Interior applications of composites in earth-based structures are likely to come under existing building or construction code requirements. Most frequently this means a requirement for some specified level of performance in the ASTM E 84 tunnel test.

The E 84 tunnel measures the spread of flame in a concurrent air flow. The tunnel does not use a radiant heater to preheat the sample, but instead the air that is flowing through the system is heated by the ignition burners and by the flame as it advances down the length of the sample. This hot air passing over the surface of the sample provides the necessary energy to bring the unburnt material up to its ignition temperature.

The ignition source for the apparatus is two burners located below the sample at the 12 in. (305 mm) position. The burners project a methane diffusion flame upward which impinges on the sample for about 2.95 ft. to 3.93 ft. (0.9 to 1.2 m) down the length of the sample from the 12 in. (305 mm) position. The layout of the test chamber and components can be found in Figure 6.6.18.2.1.

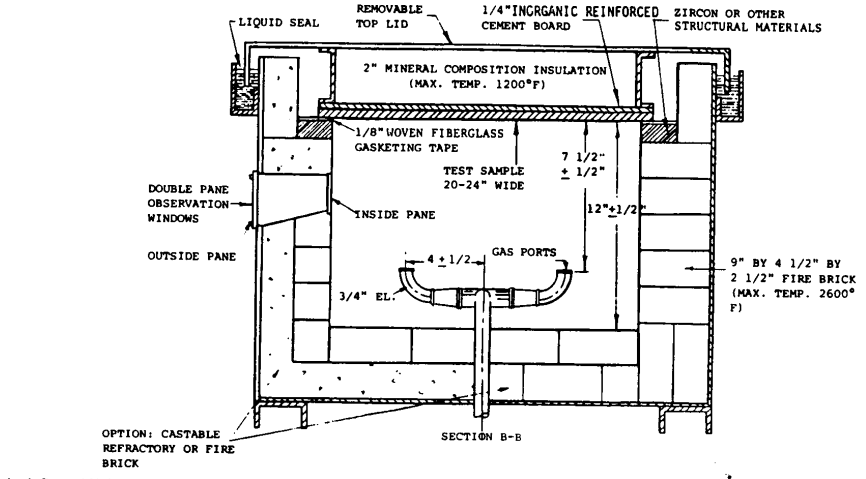
The sample is rectangular in shape, measuring 23.95 ft. x 1.67 ft. (7.3 x 0.51 m). The sample and holder become the roof of the tunnel when in place. The remaining walls are lined with fire brick. Thermocouples are located at the 13.12 ft. and 23.62 ft. (4.0 m and 7.2 m) positions. Fresh air flow is regulated with a damper at the inlet end of the tunnel. The flow rate is controlled so that the velocity in the tunnel is 47.64 in. (121 cm/s).

Attached to the exhaust end of the tunnel is a steel exhaust system. Air flow through the exhaust system is a continuation of the flow through the test chamber. Measurements of smoke obscuration are

taken in the exhaust stack. Reduction of light transmittance is measured using a photometer system. The light path is across the stack, perpendicular to the flow of the exhaust gases.

Values which are measured include time for flame to travel a measured distance, exhaust gas temperature, and percent light transmitted. Values that are often calculated include flame spread and smoke development indexes.

The test has been shown to rank "well-behaved" materials in the same order as the fire behavior measured in full scale enclosures. The term "well-behaved" here means essentially materials which behave like wood in a fire (i.e., materials which char and stay in place on the top of the tunnel for the majority of the test time). Correctly ranking the order of fire behavior of materials in a given type of full-scale test is a minimum requirement for a test method.



Inch-Pound Units	SI Units	Inch-Pound Units	SI Units
¼ in.	6.3 mm	20 in.	508 mm
2 in.	51 mm	9 by 4½ by 2½ in.	230 by 115 by 65 mm
4 ± ½ in.	102 ± 13 mm	1200°F	649°C
7½ ± ½ in.	191 ± 13 mm	2600°F	1427°C
12 ± ½ in.	305 ± 13 mm		

FIGURE 6.6.18.2.1 *Test furnace cross-section for ASTM E 84 Test Method.*

6.6.18.2.2 ASTM E 162 - Surface flammability of materials using a radiant heat energy source (Reference 6.6.18.2.2)

Another bench scale measurement of flame spread is ASTM E 162. The procedure in ASTM E 162 involves the measurement of a flame spread index (Is) which is a product of rate of energy released and average flame spread velocity in the downward direction. Although these quantities change with time as the material burns, the index is formulated to be a constant in order to provide a common scale for ranking different materials.

A sample is fixed in the test apparatus such that it is at an angle to the radiant heat source. This forces ignition at the specimen's upper edge, and if there is flame spread, it progresses downward.

A specimen 6 inches wide by 18 inches long and no greater than 1.0 inch thick is placed in the sample holder. This is located in front of a 12-inch by 18-inch radiant panel using air and gas as the fuel supply. The radiant panel consists of a porous refractory material and should be capable of operating up to

1500°F (815°C). A small pilot flame about 2 inches long is applied to the top center of the specimen at the start of the test. The test is completed when the flame front has traveled 15 inches or after an exposure time of 15 minutes.

The exposure time and whether the specimen was destroyed are reported, as well as any visual characteristics of the burning, such as running or dripping. An average flame spread index is reported.

6.6.18.2.3 ISO 9705 fire test – full-scale room test for surface products
(Reference 6.6.18.2.3)

At present there are no small scale tests for upward flame spread potential. The closest pertinent test is full-scale and it involves both lateral and concurrent flame spread (an analog of upward flame spread). This is ISO 9705 which has been recommended for interior surface materials (including composites) in high speed craft. This is a full room test and can be quite expensive for assessing composites. As an enclosure test, it may be unnecessarily severe for composites which are utilized in open spaces, such as in bridges or piers. However, for enclosed spaces such as deckhouse on a ship, this test is quite appropriate. The enclosure provides an enhanced heat feedback effect, due to accumulating hot smoke which is not present in an open fire exposure.

This full-scale room corner fire test method was developed to evaluate material potential for flame spread within and beyond a realistic compartment. Although the procedures were developed primarily for lining materials, the test is also applicable to testing complete construction assemblies. The test provides data from the early stages of material ignition to flashover.

The standard room consists of an enclosure 11.81 ft. x 7.87 ft. (3.6 x 2.4 meters) by 7.87 ft. (2.4 meters) high with a doorway centered on one 7.87 ft. (2.4-meter) wall. The ISO 9705 "standard method" uses a propane gas burner at 100 kW for the first 10 minutes and 300 kW for a further 10 minutes. A 176 kW propane burner is specified in a similar proposed ASTM standard. Each method requires the gas burner to be placed in a corner so that its flames contact the walls and ceiling.

The "standard" configuration requires that the candidate material cover the walls (excluding the doorway wall) and the ceiling. The specimens are mounted on a framing or support system comparable to that intended for their field use, using backing materials, insulation, or air gaps, as appropriate to the intended application.

The test determines the extent to which wall and ceiling materials or assemblies may contribute to fire growth. Therefore, instrumentation within the room and exhaust system are specified for measurement of the: (a) heat flux in the room; (b) total heat release generated by the fire; and (c) if flashover occurs, time that flames emerge through the doorway. Provisions are also provided for measurement of smoke and toxic gas hazards.

6.6.18.2.4 ASTM E 1321 - Determining material ignition and flame spread properties
(Reference 6.6.18.2.4)

The LIFT Method, ASTM E 1321, combines two separate test procedures: one to determine ignition and the other to determine lateral flame spread.

The sample holder fixes the specimen in a vertical orientation. A radiant panel is positioned parallel to the sample at a 75° angle from the perpendicular. The layout is represented in Figure 6.6.18.2.4. The ignition test requires samples, 5.90 x 5.90 in. (150 x 150 mm), which are exposed to a nearly uniform heat flux. A series of tests at different flux levels are used to develop an ignition time versus the radiant flux profile. From this profile, the minimum flux for ignition is determined.

The flame spread tests use 5.90 x 31.49 in. (150 x 800 mm) samples. These samples are exposed to a spatially graduated heat flux which is 10 kW/m^2 higher than the minimum flux calculated above at the hot end. The flux decays in a well-defined manner to low levels at the opposite end of the sample. The

specimens are preheated for a time which is based upon ignition test results. A horizontal pilot is ignited after the preheat time is over. The flame spread rate on the sample as a function of heat flux is then recorded.

Data reported includes minimum flux for ignition, surface temperature necessary for ignition, thermal inertia value, a flame heating parameter, and flame front velocities versus heat flux.

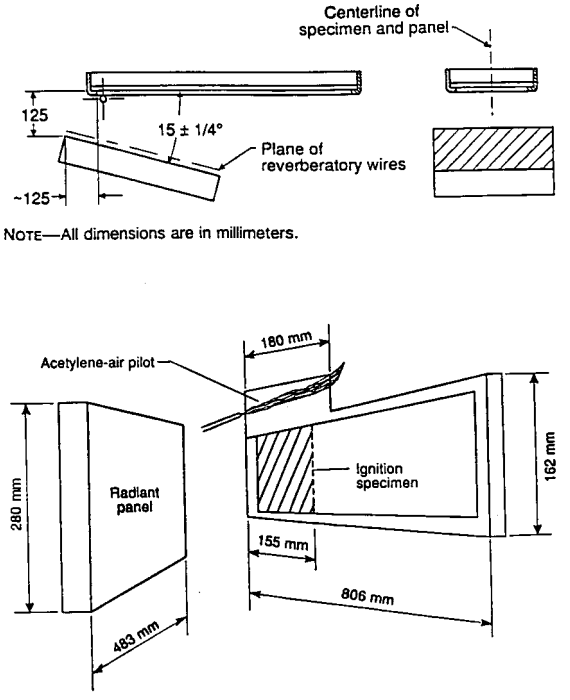


FIGURE 6.6.18.2.4 *Schematic of test method ASTM E1321.*

6.6.18.3 Smoke and toxicity test methods

Combustion gas generation is defined as the gases evolved from materials during the process of combustion. The most common gases evolved during combustion are carbon monoxide and carbon dioxide along with HCL, HCN and others depending upon the chemistry of the matrix resin of a given composite material. Historically, more people have been injured and died from fire combustion products than from direct heat/flame exposure. Various test methods have been developed to assess the toxic potential of smoke from burning materials. These test methods are sensitive to the fire exposure (non-flaming vs flaming). Test methods use either bioassay methodologies (animal testing) or analytical techniques to establish toxic potency of burning materials (References 6.6.18.3(a) and (b)).

6.6.18.3.1 ASTM E 662 - Specific optical density of smoke generated by solid materials
(References 6.6.18.3.1)

The NBS smoke chamber, identified as ASTM E 662, is used to examine the smoke produced by materials in the flaming or non-flaming modes. The sample is exposed either to a radiant heat source alone (non-flaming mode), or in conjunction with a pilot flame (flaming mode). The radiant heat is supplied by an electrical radiant heater. The heater is circular, measuring 76 mm in diameter, and is mounted in a

vertical orientation parallel to the sample. The heater applies a flux of 25 kW/m^2 to the surface of the sample.

Piloted ignition of the sample is accomplished with a multiple flamelet premixed propane/air burner. The burner is located at the bottom of the sample. It is designed so that some of the flamelets will directly impinge on the surface of the sample and some will be projected up parallel to the surface of the sample. The apparatus is shown in Figure 6.6.18.3.1.

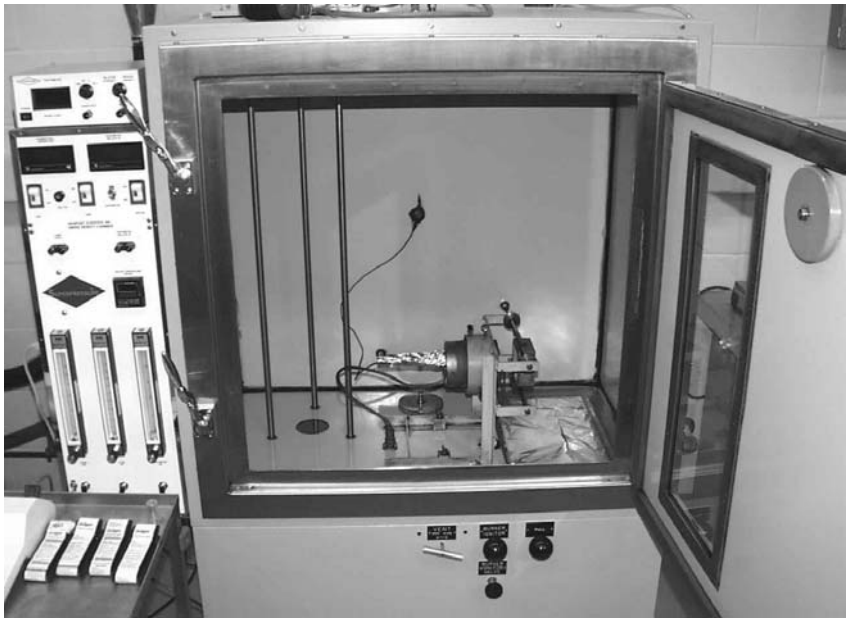


FIGURE 6.6.18.3.1 *Photograph of ASTM E662 smoke chamber.*

The sample is square in shape, measuring 2.99 x 2.99 in. (76 x 76 mm). The thickness may be varied up to 25 mm. The sample is supported in a vertical orientation. The sample, holder, burner, and heater are located inside a test enclosure which measures 2.99 ft. x 2.00 ft. x 2.99 ft. (914 x 610 x 914 mm) high. The enclosure is sealed except for ventilation openings at the bottom and top. The ventilation openings are only open if the pressure inside the chamber goes negative.

Smoke obscuration is measured using a photometric system which transverses a vertical path from the bottom to the top of the enclosure. An incandescent lamp is used for the light source. A photomultiplier tube is used as the receiver.

Values which are measured include externally applied flux and light transmitted. Values which are then calculated include specific optical density.

6.6.18.3.2 NFPA 269 - Developing toxic potency data for use in fire hazard modeling
(Reference 6.6.18.3.2)

The National Fire Protection Association (NFPA) has adopted NFPA 269 for Use in Fire Hazard Modeling. This is a small-scale test method that uses both analytical and bioassay techniques. In the test, a specimen is subjected to electric spark ignition during exposure to a 50 kW/m^2 radiant flux for 15 minutes. The smoke produced is collected for 30 minutes in a sealed chamber. The concentrations of CO, CO_2 and O_2 are measured over the test period, and a value for the concentration-time product (Ct) is calcu-

lated by integration of the area under the concentration-time curves. Measurement of HCN, HCL, and HBr is optional based on the material composition. The Ct product and the mass loss of the test specimen are used to calculate the Fractional Effective Dose (FED) for the test, and this value is used in a calculation to predict the 30-minute LC_{50} of the specimen. LC_{50} is a measure of the lethal toxic potency. It is the concentration of gas or smoke statistically calculated from concentration-response data to produce lethality in 50% of test animals within a specified exposure and post exposure time. This predicted LC_{50} is then confirmed in a comparable test by exposing six rats to the smoke produced by a specimen sized to produce the predicted LC_{50} of the smoke in the exposure chamber. The number of rats which die during the 30-minute exposure period and the subsequent 14 day post-exposure period determine the validity of the predicted LC_{50}. In this manner, it is possible to ensure that the monitored toxicants actually account for the observed effects. The LC_{50} value that is developed is for a pre-flashover fire and additional calculations are provided such that an LC_{50} for a post-flashover fire is calculated. This correction factor is based on the increased CO concentration that occurs during post-flashover fires that are ventilation controlled.

6.6.18.4 Heat release test methods

In recent years, developments in fire research and understanding of fire dynamics have highlighted the importance of heat release rate (HRR) as the primary fire hazard indicator. Fire hazard under a given set of conditions of fuel load, geometric configurations, and ventilation conditions can be expressed in terms of heat release rate and the fire hazard analysis should include the relevant fire response parameter(s) of a material obtained from small scale heat release rate tests. The assessment of potential fire hazard based on heat release rate measurements extends to composite materials also. The rate of heat release, especially the peak amount, is the primary characteristic determining the size, growth, and suppression requirements of a fire environment (References 6.6.18.4(a) and (b)).

6.6.18.4.1 ASTM E-1354 - Heat and visible smoke release rates for materials and products using an oxygen consumption calorimeter
(Reference 6.6.18.4.1)

ASTM E-1354 measures the response of a small sample of material exposed to controlled levels of radiant heating and is used to determine the heat release rates, ignitability, mass loss rates, effective heat of combustion, and visible smoke development. Commonly referred to as the cone calorimeter, this bench scale fire test method involves an application of the oxygen consumption principle and is schematically shown in Figure 6.6.4.1. The oxygen consumption principle states that for most combustibles there is a unique constant, 13.1 MJ/kg O_2, relating the amount of heat released during a combustion reaction and the amount of oxygen consumed from the air. Thus, using this principle, it is only necessary to measure the concentration of oxygen in the combustion system along with the flow rate. The air-flow past the specimen is generally set at 24 liter/s. This results in a highly fuel-lean combustion condition.

Specimens of a material or product to be tested are cut into a 3.94 x 3.94 in. (100 x 100 mm) size. The thickness depends on the type of product tested and can range from .24 to 1.96 in. (6 to 50 mm). The specimen edges are protected from burning, and the specimen can be oriented either horizontally or vertically. The specimen is heated by an electric heater in the shape of a truncated cone, hence, the name cone calorimeter. The irradiance to the specimen can be set to any desired value from zero to 110 kW/m^2, but specific thermal insults of 25, 50, 75, and 100 kW/m^2 are required. These thermal insults correspond to a small Class A fire, a large trash can fire, a significant fire, and an oil pool fire. Piloted ignition of the specimen is provided by an electric spark. Since a uniform, controlled irradiance is provided, the ignition times themselves, as measured, constitute a suitable test for ignitability. The specimen is mounted on a load cell, and its mass, along with all other instrument data, is recorded to provide mass loss rate data. The smoke measuring system is comprised of a He-Ne laser beam projected across the exhaust duct. The monochromatic light is monitored by a solid-state detector. A second detector serves as a reference to guard against effects of drift and of laser power fluctuations. The optical system is designed to be self-purging and does not use optical windows. Full specification of test conditions requires specifying the irradiance, the specimen orientation, the use of spark ignition, the test irradiance, and any special specimen preparation techniques.

The data derived from tests in the cone calorimeter constitute a very large set and can be analyzed in a multitude of ways. The data reported include the following:

(a) Peak rate of heat release (kW/m^2)
(b) Rates of heat release averaged over various time periods, starting with the time of ignition (kW/m^2)
(c) Effective heat of combustion (MJ/kg). This will be less than the oxygen-bomb value of the heat of combustion since the combustion is incomplete (as it is in real fires)
(d) Percent specimen mass lost (%)
(e) Time to ignition (s)
(f) Average smoke obscuration (m^2/kg). Smoke production from a material has the rational units of m^2, representing the extinction cross-section of the smoke. This is normalized by the amount of specimen mass lost (kg)
(g) Average yields of each of the measured gas species (kg/kg)

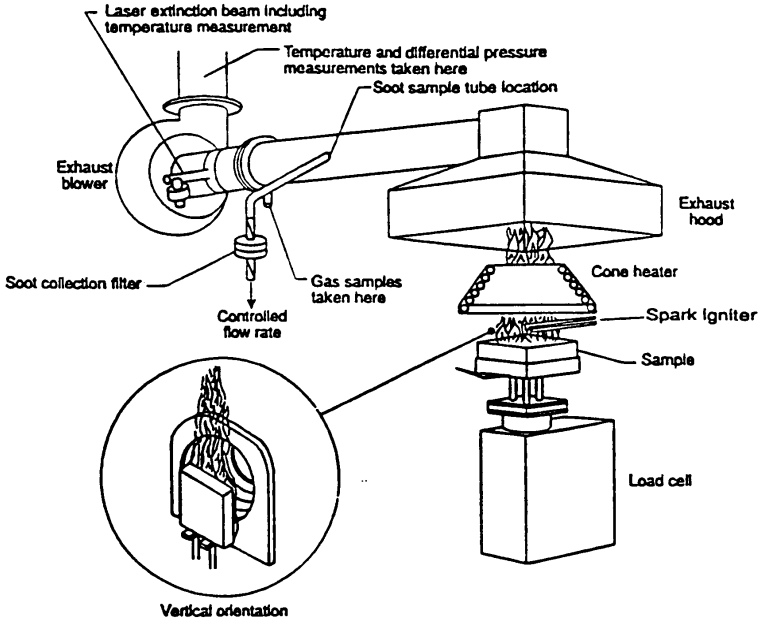


FIGURE 6.6.18.4.1 *Schematic of ASTM E 1354.*

6.6.18.4.2 ASTM E 906 – Heat and visible smoke release rates for materials and products (Reference 6.6.18.4.2)

ASTM E 906 is based on a thermopile method where the temperature rise is used to determine the heat release rate of materials. This thermopile method measures the heat release of materials at a radiant heat flux of 35 kW/m^2. The apparatus consists of a combustion chamber inside an insulated metal box. The radiant source comprises four silicon carbide heating elements of nominal resistance of 1.4 ohms. Specimens are exposed to the radiant heater source inside the chamber at an irradiance of 35 kW/m^2.

Samples are 59 x 59 in. (150x150 cm) in size and a minimum of three specimens are tested for each material. The ignition is caused by a pilot flame from a methane burner placed above the specimen

holder. The total airflow rate through the apparatus is 40 liter/s airflow of which 10 liter/s passes through the combustion chamber. The remaining 30 liter/s flows through the hollow wall section. The temperatures of inflowing air and outflowing gases are measured with a thermopile consisting of 5 type K thermocouples. The hot junctions of the thermocouples are distributed along the diagonals of the stack above a baffle plate. The increase in temperature of the gases exiting the stack with respect to the air temperature entering the combustion zone gives a measure of the heat release rate of the material.

6.6.18.5 Fire resistance test methods

The intensity and duration of a fire can vary over a wide range. It is important to have some understanding of the resistance of building assemblies to different fire threats in order to choose those which more closely match the potential fire threat in a given compartment. Fire resistance here means the ability of a material to continue to serve its structural role during a fire (References 6.6.18.5(a) and (b)).

6.6.18.5.1 ASTM E-119 - Fire tests for building construction and materials
(Reference 6.6.18.5.1)

One test method for fire resistance is ASTM E-119, which uses what is often referred to as the standard time-temperature curve. Underwriters Laboratory utilizes this test method to provide fire ratings for all assemblies used in building construction.

In this test, structural components are subjected to a heated furnace environment for the desired duration. If the endpoint criteria are not reached prior to the end of the test period, the assembly is rated as acceptable for that test period, e.g., 30 or 60 minutes. The furnace is heated in such a manner so that the temperature inside the furnace follows a standard time-temperature curve. This curve, shown in Figure 6.6.18.5.1, is intended to relate to conditions in a fully developed room fire. Assemblies may be tested with or without load. If the assembly is tested under load, the assembly is loaded to induce maximum design stress levels calculated based on theory. Floor and roof assemblies and bearing walls are always tested under load. In addition, a second specimen must be exposed to a hose stream to simulate manual fire fighting and rapid cooling.

Sample sizes for this test are specified as follows: bearing walls and partitions, 9.3 $m^{2;}$ nonbearing walls and partitions, 9.3 m^2; columns, 2.7 m; floors and roofs, 16.7 m^2.

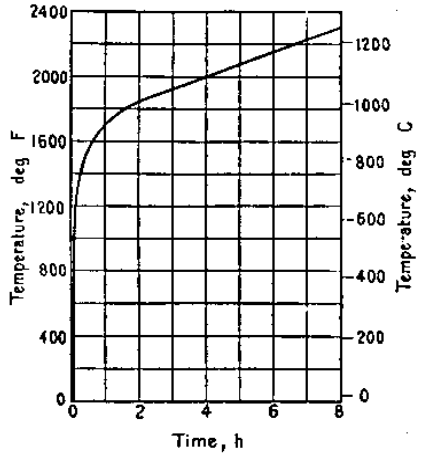


FIGURE 6.6.18.5.1 *ASTM E-119 Time temperature curve.*

6.6.18.5.2 ASTM E-1529 - Determining effects of large hydrocarbon pool fires on structural members and assemblies and UL 1709 - Rapid rise fire tests of protection materials for structural steel (References 6.6.18.5.2(a) and (b)).

One of the distinguishing features of a post flashover fire is the rapid development of high temperatures and heat fluxes that can subject exposed structural members to thermal shock much more readily than they see in ASTM E-119. The ASTM E-1529 fire curves address this issue. The performance of structural members and assemblies exposed to fire conditions resulting from large, free-burning fluid-hydrocarbon-fueled pool fires is the focus of this test. The exposure scenario which is simulated by this test is the condition of total, continuous engulfment of a member or assembly in the luminous flame area of a large free-burning-fluid-hydrocarbon pool fire.

The test setup provides an average surface heat flux on all exposed surfaces of the test specimen of 158 ± 8 kW/m^2. This heat flux is attained within the first 5 minutes of test exposure and maintained for the duration of the test. The test environment reaches a temperature of at least 1500°F (815°C) after the first 3 minutes of the test and remains between 1850°F and 2150°F (1010°C and 1176°C) at all times after the first 5 minutes of the test. Because hydrocarbon pool fires often occur in outdoor environments, procedures for accelerated weathering and aging tests are set forth to simulate weathering and aging in outdoor environments. Fire endurance ratings are given for the time period during which the assembly withstands the fire scenario without allowing the passage of flame or hot gases capable of igniting cotton waste.

The temperature conditions described in UL 1709 (Standard for Rapid Rise Fire Tests of Protection Materials for Structural Steel) are similar to those described above for ASTM E-1529. However, heat fluxes described in UL 1709 (204 ± 16 kW/m^2) are higher than those described in ASTM E-1529 (158 ± 8 kW/m^2). The UL 1709 fire curve rises to at least 1500°F (815°C) after the first 3 minutes of the test and is between 1850°F (1010°C) and 2150°F (1180°C) at all times after the first 5 minutes of the test. In contrast to ASTM E-1529 and UL 1709, the ASTM E-119 fire curve rises to only 1000°F (538°C) at the end of the first 5 minutes, and to 1700°F (927°C) at the end of 60 minutes.

6.7 ELECTRICAL PROPERTY TESTS

6.7.1 Introduction

In certain applications, the electrical properties of a composite are important. The properties that are of interest include dielectric constant, dielectric strength, volume resistivity, surface resistivity, are resistance, dissipation and loss factors. The values can be affected by temperature and environment, as well as the type of curing agent, filler, and fiber used in the composite. The following ASTM test methods can be used for determining the electrical properties of polymer matrix composite laminae and laminates:

ASTM D 149 "Standard Test Method for Dielectric Breakdown Voltage and Dielectric Strength of Solid Electrical Insulating Materials at Commercial Power Frequencies". Method for determining the dielectric strength of solid insulating materials.

ASTM D 150 "Standard Test Method for A-C Loss Characteristics and Permittivity (Dielectric Constant) of Solid Electrical Insulating Materials". Method used for determining the relative permittivity, dissipation factor, loss index, power factor, phase angle, and loss angle of solid insulating materials when the standards are lumped impedances.

ASTM D 495 "Standard Test Method for High-Voltage, Low-Current, Dry Arc Resistance of Solid Electric Insulation". This test method is intended for preliminary screening of material and should not be used in material specifications.

ASTM D 2303 "Standard Test Methods for Liquid-Contaminant, Inclined Plane Tracking and Erosion of Insulating Materials". Test methods for the quantitative evaluation of the relative ability of insulating

materials to withstand the action of electrical discharges on the surface, similar to what may occur in service under the influence of dirt and moisture condensed from the atmosphere.

6.7.2 Electrical permittivity

This section is reserved for future use.

6.7.3 Dielectric strength

This section is reserved for future use.

6.7.4 Magnetic permeability

This section is reserved for future use.

6.7.5 Electromagnetic interference

This section is reserved for future use.

6.7.6 Electrostatic discharage

This section is reserved for future use.

6.8 STATIC UNIAXIAL MECHANICAL PROPERTY TESTS

6.8.1 Introduction

Section 6.8 discusses test methods for determining the static uniaxial mechanical properties of laminated composites. The purpose of this section is to provide brief commentaries on the most commonly used methods, to alert the reader to the limitations of the various methods, and to encourage uniformity in the use of standard test methods with the ultimate goal of combinability of experimental data obtained from multiple sources. The reader is referred to Chapter 8 for statistical data analysis requirements for reporting of data to MIL-HDBK-17.

The section reflects the current dynamic state of test methods development for composite materials. Many of the methods were originally developed for testing of reinforced plastics, and modifications have been (or are being) made for applicability to advanced composites. In recent years there has been a tendency for users to unilaterally modify existing standards without a formal standardization process, leading to uncontrolled test results. In general, these modified standards are not discussed in Section 6.8 except where a specific modification is in common use, and where discussion of the technique is deemed constructive. The test methods included are representative of procedures used in the composite materials industry, and were selected after review of standards documents and user material specifications. Specific test methods may cover lamina-level testing, laminate-level testing, or both, depending upon the test method. The scope of each test method is discussed in the appropriate section.

It is important to make a distinction between methods that are discussed in Section 6.8, and methods for data submittal to MIL-HDBK-17:

- Test methods used by contractors are agreed upon with customers and/or certifying agencies. Section 6.8 reviews many methods in order to provide the reader with awareness of the broad range of procedures in common use. Some of these have been formally standardized (ASTM and other standards) and some are "common practice" methods. Some have distinct limitations, and these are indicated as a matter of information. Mention or omission of a particular method does not, of itself, require or restrict usage. Specific methods are included to allow the user to

perform tests consistent with industry practice; however, inclusion of these standards should not be considered an endorsement of any standard or organization by MIL-HDBK-17.

- When submitting data to MIL-HDBK-17 for consideration for inclusion in Volume 2 of the Handbook, specific methods must be used. Tables at the end of most subsections of 6.8 indicate which methods are acceptable for such submittals. These methods have been chosen in accordance with the criteria given in Section 2.5. Readers are encouraged to also use these methods in contract and internal work to promote standardization.

When selecting and using a particular mechanical strength test method, the importance of obtaining the proper failure mode cannot be overemphasized. While universal definitions of "proper" and "valid" have not been established for most types of tests, further analysis must be employed when unexpected or questionable modes are observed or suspected. If the type of failure is different from what is expected from the test, the data may not represent the property being evaluated. Furthermore, if the failure mode varies within a group of specimens, statistical analysis of the resulting data will not be meaningful due to the introduction of an additional source of variability not related to the property being tested. Therefore, it is crucial that failure modes be reported, and that data be disqualified and discarded when analysis has indicated an unacceptable mode.

It should be noted that failure mode analysis is not necessarily limited to physical examination of the failed test specimens. Other evidence might be obtained from review of additional factors such as:

1. Bending curves from back-to-back strain gage data
2. A check of test machine and/or test fixture alignment
3. A review of the exact procedure used to install and properly align the specimens in the test fixture
4. A check for possible damage to, or malfunction of, the test fixture

ASTM has begun to incorporate failure mode examples and codes into its standard test methods. For example, the 1993 revision of ASTM D 3039 (*Standard Test Method for Tensile Properties of Polymer Matrix Composite Materials*) depicts nine types of failures of the specimen, and defines a three-character coding system that describes various failures. The first letter of the code identifies the type of failure (angled, grip, delamination, etc.), the second indicates the area of the failure (gage, at grip, etc.), and the third denotes the failure location (top, bottom, middle, etc.). In the particular case of tension testing, a failure of the tab or tab adhesive would be an unacceptable mode since the ultimate tensile strength of the laminate was not measured.

Rather than duplicate failure mode examples within the subsections of Section 6.8, the reader is advised to be conscientious regarding the documentation of failure modes, and to refer to examples provided within specific test methods where such examples exist.

6.8.2 Tensile properties

In-Plane Tensile Properties:

Lamina

$E_1^t,\ F_1^{tu},\ \varepsilon_1^{tu},\ \nu_{12}^{tu}$

$E_2^t,\ F_2^{tu},\ \varepsilon_2^{tu},\ \nu_{21}^{tu}$

Laminate

$E_x^t,\ F_x^{tu},\ \varepsilon_x^{tu},\ \nu_{xy}^{tu}$

$E_y^t,\ F_y^{tu},\ \varepsilon_y^{tu},\ \nu_{yx}^{tu}$

Out-of-Plane Tensile Properties:

Lamina

$E_3^t,\ F_3^{tu},\ \varepsilon_3^{tu},\ \nu_{31}^{tu},\ \nu_{32}^{tu}$

Laminate

$E_z^t,\ F_z^{tu},\ \varepsilon_z^{tu},\ \nu_{zx}^{tu},\ \nu_{zy}^{tu}$

6.8.2.1 Overview

The basic physics of most tensile test methods are very similar: a prismatic specimen with a straight-sided gage section is gripped at the ends and loaded in uniaxial tension. The principal differences between these tensile test specimens are the specimen cross-section and the load-introduction method. The cross-section of the specimen may be rectangular, round, or tubular; it may be straight-sided for the entire length (a "straight-sided" specimen) or width- or diameter-tapered from the ends (a larger area) into the gage section (a smaller area).[1] Straight-sided specimens may, in some cases, utilize tabbed load application points.

There are three notable exceptions to the uniaxially loaded prismatic specimen: 1) a sandwich beam test that relies upon gross flexure of a sandwich beam to create an in-plane stress state in the facesheets, making the tensile facesheet, in effect, the specimen; 2) a ring test that applies, via a fixture, a diametrical expansion (or an approximation of such) to a narrow, high radius-to-thickness ratio ring, creating a membrane (in-plane) tensile stress in the ring; and 3) a solid-laminate curved beam test that applies, via a fixture, an opening bending moment, producing a through the thickness tensile stress in the bend region.

While there are a number of existing or developing standards for in-plane tensile properties of laminated materials, this is not the case for out-of-plane properties. Test methods potentially suitable to become standards for through the thickness tensile properties of laminates have only recently begun to receive substantial attention, and so are relatively immature.

By changing the specimen configuration, many of the tensile test methods are able to evaluate different material configurations, including unidirectional laminates, woven materials, and general laminates. However, some specimen/material configuration combinations are more robust (less sensitive to specimen preparation and testing variations) than others. The least robust (most user-sensitive) configuration is the unidirectional specimen. As an example, fiber/load misalignment in a 0° unidirectional specimen, which can occur due to either specimen preparation or testing problems, or both, can reduce strength as much as 30% due to an initial 1° misalignment. This specimen is also very sensitive to load-introduction upsets and requires a high degree of laboratory sophistication, both in specimen preparation as well as testing, to achieve satisfactory results. And bonded end-tabs, which were introduced in the late 1960's to *minimize* load-introduction problems in high-strength unidirectional materials, can actually *cause* premature specimen failure (even in non-unidirectional specimens), if not applied and used precisely and with great art. Since most 0° unidirectional specimens fail with an explosive shatter that obscures the true failure mode, physical evidence of poor testing/specimen preparation practices is usually unavailable.

These difficulties with the testing of unidirectional materials have led to the increased use of a much more robust $[90/0]_{ns}$-type laminate specimen (also known as the "crossply" specimen). From the laminate strength of a crossply specimen (when the lamina elastic properties are known), the equivalent unidirectional F_1^{tu} lamina strength can be derived, using the procedure discussed in Section 2.4.2. When previously undocumented improvements in testing technique are combined with use of crossply test specimens, much simpler untapered tabs, or even tabless specimens, are now feasible, allowing laboratories that are generally qualified, but inexperienced in unidirectional testing, to produce results equivalent to the best attainable unidirectional data. While unidirectional testing is still performed, and in certain cases may be preferred or required, a straight-sided, tables, $[90/0]_{ns}$-type specimen is now generally believed to be the lowest cost, most reliable configuration for lamina tensile testing of unidirectional materials. This straight-sided tabless configuration also works equally well for non-unidirectional material forms and for

[1]Though there have been many different types of tapered specimens, they are often called, as a class, Adogbone≅ specimens.

other general laminates. Another advantage is that, unlike with 0° unidirectional specimens, $[90/0]_{ns}$-type specimen failures do not usually mask indicators of improper testing/specimen preparation practices.

6.8.2.2 In-plane tension test methods

6.8.2.2.1 Straight-sided specimen tension tests

1) ASTM D 3039/D 3039M, Standard Test Method for Tensile Properties of Polymer Matrix Composites
2) ISO 527, Plastics --- Determination of Tensile Properties
3) SACMA RM 4, Tensile Properties of Oriented Fiber-Resin Composites
4) SACMA RM 9, Tensile Properties of Oriented Cross-Plied Fiber-Resin Composites
5) ASTM D 5083, Standard Test Method for Tensile Properties of Reinforced Thermosetting Plastics Using Straight-Sided Specimens

ASTM Test Method D 3039/D 3039M (Reference 6.8.2.2.1(a)), originally released in 1971, is the original standard test method for straight-sided rectangular specimens. As a result of a major re-write of D 3039, approved in 1993, tabs were made optional, and a significant number of previously ambiguous, undocumented, and/or optional test and reporting parameters were clarified, documented, and/or made mandatory. ISO 527 (Reference 6.8.2.2.1(b)) parts 4 and 5 (currently in the draft international standard phase) and the two SACMA (Suppliers of Advanced Composite Materials Association) tensile test methods, SRM 4 (Reference 6.8.2.2.1(c)) and SRM 9 (Reference 6.8.2.2.1(d)) are substantially based on ASTM D 3039 and, as a result, quite similar.

While there are still a number of minor differences between ASTM D 3039 and ISO 527, there is a coordinated effort underway to harmonize ASTM D 3039 and ISO 527 and make them technically equivalent. SRM's 4 and 9, while originally intended to be restricted subsets of ASTM D 3039, deviate from ASTM D 3039 enough that they are not exactly equivalent test methods; an ASTM/SACMA harmonization effort is being discussed but has not yet begun.[1] The last of the straight-sided test methods, ASTM D 5083 (Reference 6.8.2.2.1(e)), is the straight-sided equivalent of the ASTM D 638 dogbone tension test for plastics (discussed in Section 6.8.2.2.3). While ASTM D 5083 is conceptually similar to ASTM D 3039, D 5083 was not developed for use with advanced composites, and therefore, cannot be recommended.

In all of these test methods, a tensile stress is applied to the specimen through a mechanical shear interface at the ends of the specimen, normally by either wedge or hydraulic grips. The material response is measured in the gage section of the specimen by either strain gages or extensometers, and the elastic material properties subsequently determined.

End tabs, if used, are intended to distribute the load from the grips into the specimen with a minimum of stress concentration. A schematic example of an appropriate failure mode of a multidirectional laminate using a tabbed tensile specimen is shown in Figure 6.8.2.2.1(a). However, design of the tabs remains somewhat of an art, and an improperly designed tab interface will produce an unacceptable proportion of failures near the tab and result in very low specimen strengths. For this reason a single standard tab design has not been mandated by ASTM, although, when tabs are necessary, the easier-to-apply, less expensive, unbeveled 90° tabs are preferred if the results are acceptable. Recent comparisons confirm that success of a tab design is more dependent on use of a sufficiently ductile adhesive than on the tab angle. An unbeveled tab applied with a ductile adhesive will outperform a tapered tab that has been applied with a insufficiently ductile adhesive. Adhesive selection is therefore most critical to bonded tab use.

The simplest way to avoid bonded tab problems is to not use them. Many laminates (mostly non-unidirectional) can be successfully tested without tabs, or with friction tabs. An example of a high-strength carbon/epoxy material being tested in an untabbed, $[90/0]_{ns}$-type laminate configuration using an emery

[1]ASTM D 3039 contains bending and failure mode restrictions not present in SRM=s 4 and 9, and is different in other respects like thickness measurement, conditioning, and data reporting. ASTM D 3039 is also significantly more detailed than SRM=s 4 and 9. The sum of these differences may produce a different test result.

cloth interface in finely serrated wedge grips is shown in Figure 6.8.2.2.1(b). Flame-sprayed unserrated grips have also been successfully used in tabless tension testing.

FIGURE 6.8.2.2.1(a) *Typical tension failure of multi-directional laminate using a tabbed specimen.*

Other important factors that affect tension testing results include control of specimen preparation, specimen design tolerances, control of conditioning and moisture content variability, control of test machine-induced misalignment and bending, consistent measurement of thickness, appropriate selection of transducers and calibration of instrumentation, documentation and description of failure modes, definition of elastic property calculation details, and data reporting guidelines. These factors are described in detail, and controlled, where appropriate, by ASTM D 3039/D 3039M. While ISO 527 parts 4 and 5 and SRM's 4 and 9 are similar to ASTM D 3039/D 3039M in most respects, they do not provide the same degree of guidance or control as ASTM D 3039. For this reason ASTM D 3039 is preferred.

In summary, with proper attention to detail and reasonable care the straight-sided specimen test is generally straightforward and gives good results. However, test parameters must be properly selected for the material and configuration under test, which requires training and experience.

Limitations of the straight-sided specimen tensile test:

Bonded Tabs: The stress field near the termination of a bonded tab is significantly three-dimensional, and critical stresses tend to peak at this location. Design of bonded tabs for the purpose of minimizing peaking stresses is not well-understood and is material and configuration dependent; improperly designed tabs can significantly degrade results. As a result, tables or unbonded tabbed configurations are becoming more popular, when the resulting failure mode is appropriate.

Specimen Design: There are, particularly within ASTM D 3039, a large number of specimen design options included in the standard, needed to cover the wide range of material systems and lay-up configurations within the scope of the test method. These options can be very confusing to the novice, and can lead to the selection of an inappropriate specimen design that negatively affects test results.

Specimen Preparation: Specimen preparation is very important to the test results. While this can probably be said to be true for almost all composite mechanical tests, it is particularly important for unidirectional tests, and unidirectional tension tests are no exception. Fiber alignment, control of specimen taper, and specimen machining (while maintaining alignment) are the most critical steps. For very low strain-to-failure material systems or test configurations, like the 90° unidirectional test, flatness is also particularly important. Edge machining techniques (avoiding machining-induced damage) and edge surface finish are also particularly critical to strength results from the 90° unidirectional test.

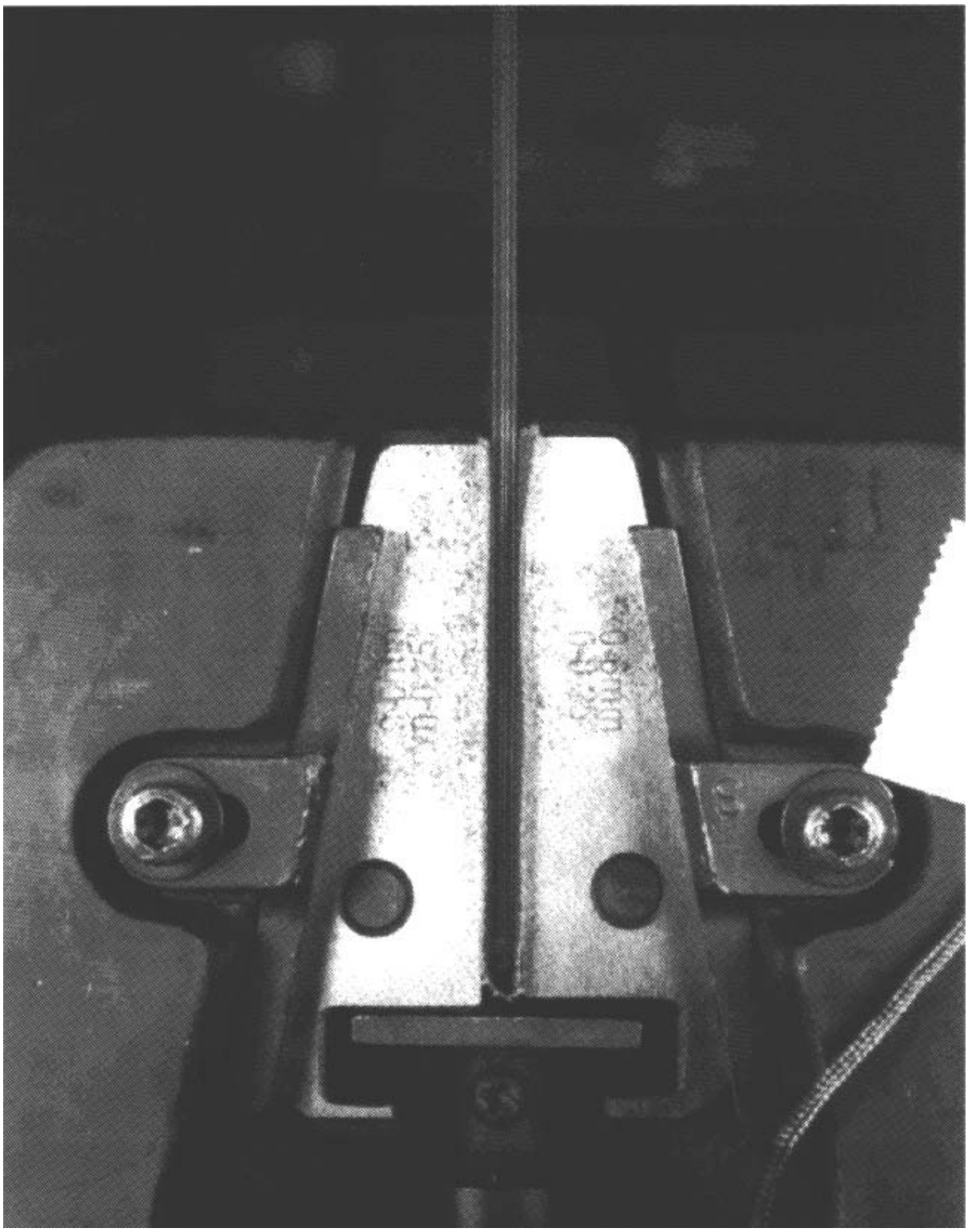


FIGURE 6.8.2.2.1(b) *Tension testing of untabbed specimen using an emery-cloth gripping interface.*

6.8.2.2.2 Filament-wound tubes

ASTM Standard Test Method D 5450/D 5450M, Transverse Tensile Properties of Hoop Wound Polymer Matrix Composite Cylinders

ASTM D 5450 describes a test for 90° tensile properties, specifically for a hoop-wound unidirectional cylinder. This test method is discussed in more detail in Section 6.12.1 on test methods for filament wound materials.

6.8.2.2.3 Width tapered specimens:

1) ASTM Standard Test Method D 638, Tensile Properties of Plastics
2) SAE AMS “Bowtie” Tension Specimen

ASTM Test Method D 638 (Reference 6.8.2.2.3(a)), developed for and limited by scope to use with plastics, uses a flat, width-tapered tension specimen with a straight-sided gage section. Despite its heritage, this specimen has also been evaluated on and applied to composite materials. The specimen taper is accomplished by a large cylindrical radius between the wide gripping area at each end and the narrower gage section, resulting in a shape that justifies the specimen nickname of the “dogbone” specimen. The taper makes the specimen particularly unsuited for testing of 0° unidirectional materials, since about half of the gripped fibers terminate prior to the gage section, resulting in failure by splitting at the radius due to inability of the matrix to shear the load from terminated fibers into the gage section.

While the ASTM D 638 specimen configuration has sometimes been successfully used for fabric reinforced composites and with general non-unidirectional laminates, some material systems remain sensitive to the stress concentration at the radius. While, for its intended use with plastics, the specimen is molded to shape, for laminated materials the specimen must be machined, ground, or routed to shape. The specimen also has the drawback of having a relatively small gage volume and is poorly suited for characterization of coarse weaves with repeating units larger than the gage width of 0.25-0.50 in. (6.4-13 mm). The standardized procedure, due to the intended scope, does not adequately cover the testing parameters required for advanced composites.

The bowtie tension test specimen, so-called because of its planform shape with a reduced cross-section, is similar in many respects to the ASTM D 638 specimen, though it has never been released as a standard test method. The bowtie specimen has achieved indirect standardization through use in several SAE AMS composite material (fabric-based) specifications[1]. It is also still contained within a number of existing corporate internal material specifications for fabric-based materials, though it is rarely used now in new material specifications. With the geometric similarity to the ASTM D 638 specimen come a similar set of limitations and restrictions. The shape fundamentally restricts use to fabric reinforced materials and/or non-unidirectional laminates. Specimen preparation is extremely important since the reduced cross-section is prepared by machining, routing, or grinding, and both surface finish of the edge and machining of the tangent radii at the transition region to the reduced area are critical. The specimen also does not work well with coarse fabrics, since the gage section is only 0.5-in. (13 mm) wide.

To its credit, the bowtie specimen is reportedly somewhat less sensitive to failures in the transition section than the D 638 specimen, and has also been employed as a resource of last resort, particularly when the severity of non-ambient test environments creates otherwise difficult gripping problems for straight-sided specimens.

Other width-tapered specimen configurations have been proposed, but to date each has been shown, after study, to have at least one shortcoming that renders the method undesirable for general application, and so will not be further discussed.

[1]The four known SAE specifications containing the bowtie coupon at the time of this writing were: AMS 3844A (Reference 6.8.2.2.3(b)), AMS 3845A, AMS 3847B, and AMS 3849A. Only the first, as an example, is completely referenced.

Limitations of the width-tapered tension tests for advanced composites:

Standardization: While the ASTM D 638 test is standardized, it was not developed for advanced composites, and is primarily applicable to relatively low modulus, unreinforced materials, or low reinforcement volume materials incorporating randomly oriented fibers. The bowtie test is standardized only in the sense of continued use in a limited number of SAE AMS material specifications. It has not been standardized for general use.

Specimen preparation: Special care is required to machine the taper into a laminated coupon.

Cost: Specimen fabrication is more expensive than untabbed straight-sided coupons.

Stress state: The radius transition region can dominate the failure mode and result in reduced strength results. The width-tapered specimen is not suitable for unidirectional laminates, and is limited to fabrics or non-unidirectional laminates when gage section failures can be attained.

Limited gage section volume: The limited gage width makes it unsuitable for coarse fabrics.

6.8.2.2.4 Split-disk ring tension test

ASTM Standard Test Method D 2290, Apparent Tensile Strength of Ring or Tubular Plastics and Reinforced Plastics by Split Disk Method

Procedure A (Procedures B and C apply only to plastics) of ASTM D 2290 (Reference 6.8.2.2.4) loads a hoop-wound narrow ring using a split-disk loading fixture that applies a hoop-direction tensile stress to the test ring. This test method was developed in the early years of composites, primarily for tensile properties of filament wound materials. It has long since been superseded by more reliable and more representative test methods. The disadvantages will not be dwelled upon, but include the material form/process limitation, the presence of a unaccounted bending moment at the fixture split, the extremely small gage volume, and the inability to monitor strain response. This test is not recommended for MIL-HDBK-17 data, but it still sees some limited usage as a quality control test in the filament winding industry.

6.8.2.2.5 Sandwich beam test

ASTM Standard Test Method C 393, Flexural Properties of Flat Sandwich Constructions

The sandwich beam test, shown schematically in Figure 6.8.2.2.5 is standardized as ASTM C 393 (Reference 6.8.2.2.5). While primarily intended as a flexure test for sandwich core shear evaluation, the scope also allows use for determination of facing tensile strength. While this use is not well documented within the test method, it has been used for tension testing of composite materials, particularly for 90° properties of unidirectional materials, or for fiber-dominated testing in extreme non-ambient environments.

An example of practical use of this test method for 90° unidirectional tape properties follows. A piece of 0.5-in. thick, 1/8-in. cell, 8.1 lbm/ft^3 (13 mm thick, 3 mm cell, 130 kg/m^3) aluminum honeycomb core is bonded to the test laminate using a suitable adhesive. A compressive faceskin is also bonded to the other side of the core, normally during the same bonding step. To minimize thermal expansion problems from dissimilar materials, the compressive faceskin is often chosen to be of the same material and orientation, but at twice the thickness of the tensile faceskin to assure failure in the tensile faceskin. The test specimen is then cut with a wet-diamond saw from the sandwich laminate. Specimen dimensions are 1 inch (25 mm) wide and 8 inch (200 mm) long, with the core ribbon direction aligned with the length of the specimen. The test setup uses a support span of 7 inch (180 mm) and a four-point loading span of 3 inch (76 mm). The load is both applied and reacted at all points using 1-inch (25 mm) square, 1/8-inch (3 mm) thick rubber pads, which are in turn each loaded by a 1/4-inch (6 mm) thick steel loading plate of the same area. The load is applied to each loading plate via a 1/2-inch (13 mm) diameter steel roller that rides in a transverse slot in the loading plate. This loading mechanism distributes the load into the beam

and prevents out-of-plane crushing of the core. The specimen and loading fixturing are shown schematically in Figure 6.8.2.2.5.

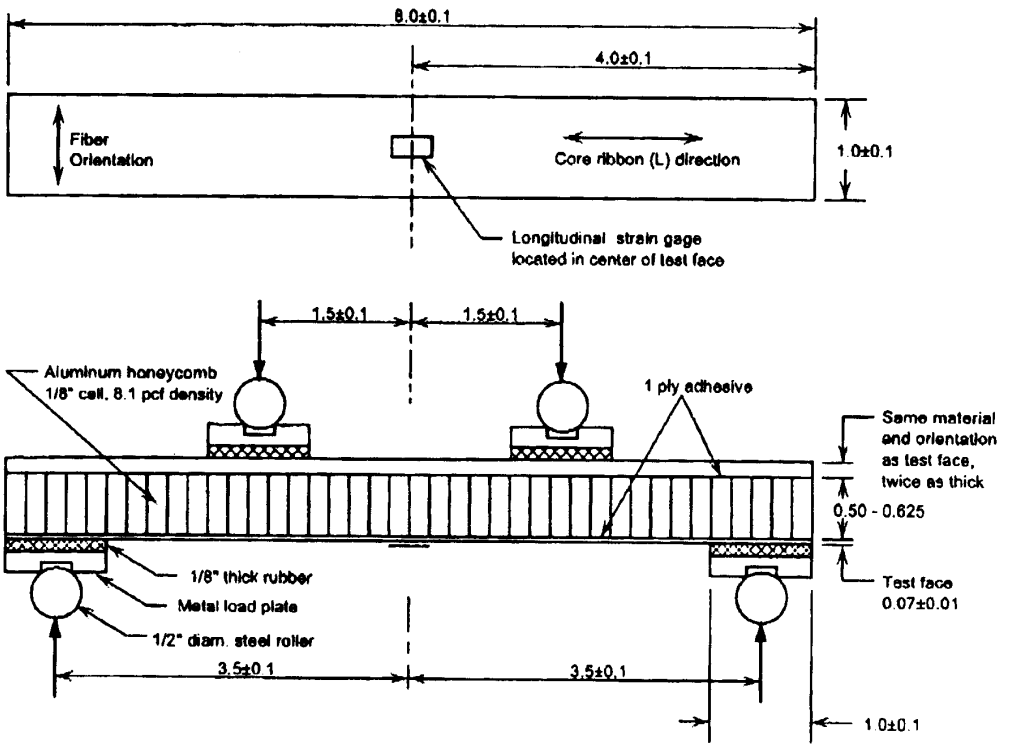


FIGURE 6.8.2.2.5 *Schematic of sandwich beam test coupon and fixturing, for 90° unidirectional tensile testing of laminated materials.*

This test specimen is claimed by some to be less susceptible to handling and specimen preparation damage than D 3039-type 90° specimens, resulting in higher strengths and less test-induced variation. However, the one-sided environmental conditioning of this specimen is a problem, since the required conditioning times are longer by a factor of four or more, and such conditioning can create adhesive bond failures. Adhesive selection is, therefore, important and masking of the adhesive from the environmental conditioning may be required. In such cases, conditioning travelers are required that must be twice the test skin thickness in order to simulate the single-sided exposure of the specimen itself.

Limitations of the sandwich beam test for tensile properties:

Cost: Specimen fabrication is relatively expensive.

Stress State: The effect on the stress state of the sandwich core has not been studied in tension, and could be a concern.

Standardization: While this test technically is standardized, its practical application and limitations are not well studied or documented.

Conditioning: Conditioning is more difficult, as described above.

6.8.2.3 Out-of-plane tensile test methods

6.8.2.3.1 Introduction

There are currently no published standards for out-of-plane tensile test methods specifically relating to composites. Two basic approaches are presently in use, or being studied, by the aerospace industry include: direct out-of-plane loading of a laminated specimen bonded between two fixture blocks (based on modifications to similar non-composite test methods) and indirect out-of-plane loading of a curved beam. Both concepts are being considered for possible standardization in composite use by ASTM.

6.8.2.3.2 Direct out-of-plane loading adaptations of ASTM C 297/C 633/D 2095

1) using square cylinder loading blocks, or
2) using circular cylinder loading blocks

Three similar ASTM standard test methods already used for out-of-plane loading of other material systems have been adapted to composites: ASTM C 297 (Reference 6.8.2.3.2(a)), ASTM C 633 (Reference 6.8.2.3.2(b)), and ASTM D 2095 (Reference 6.8.2.3.2(c)). In the adaptations to composites a laminated specimen is bonded to cylindrical metal loading blocks which are pulled in the out-of-plane direction by a tensile test machine. The metal loading cylinders are either square or circular. The square specimens are typically 2-in. (50 mm) in width, while the circular specimens range in diameter from 0.8-in. to 2-in (20-50 mm). Strength is determined simply by dividing maximum load prior to failure by the specimen gage area.

If the specimen is sufficiently thick, strain gages may be used to determine elastic modulus. A thick specimen may also allow a reduced diameter gage section, which may be required if the out-of-plane strength exceeds the bond strength of the specimen/loading-block interface.

It has been argued by some that a circular specimen achieves a more uniform stress distribution (lower stress concentration). However, either configuration is extremely sensitive to specimen preparation factors, especially surface finish of the specimen edge and alignment of the load and loading blocks. Two typical specimen configurations are shown in Figure 6.8.2.3.2(a).

Each of the ASTM test methods uses a different method of introducing the load to the loading blocks. ASTM C 297 uses what is essentially a universal joint at each end. ASTM C 633 (circular only) applies a thread to the opposite end of each loading block and depends upon test machine alignment to eliminate bending. ASTM D 2095 uses a fixture that eliminates one of the bending degrees of freedom at one end, and the other bending degree of freedom at the other end. These three approaches are shown in Figures 6.8.2.3.2 (b-d).

Limitations of the direct out-of-plane tensile test methods:

Standardization: Despite the existence of three similar standards intended for use on other material systems, there is currently no standard for application of the methodology to laminated composites. This approach is still being studied.

Cost: Due to tight tolerances required for repeatable representative data, specimen preparation is relatively expensive.

Specimen preparation: Results are extremely sensitive to alignment of the loading blocks during bonding, as well as the machining quality and surface finish of the laminate edges. This implies that the laminate itself must be flat. An additional consideration is CTE-mismatch induced thermal stresses caused by a significant difference between the laminate in-plane CTE and the end-block CTE. This is especially important during end-block bonding, as well as during non-ambient testing.

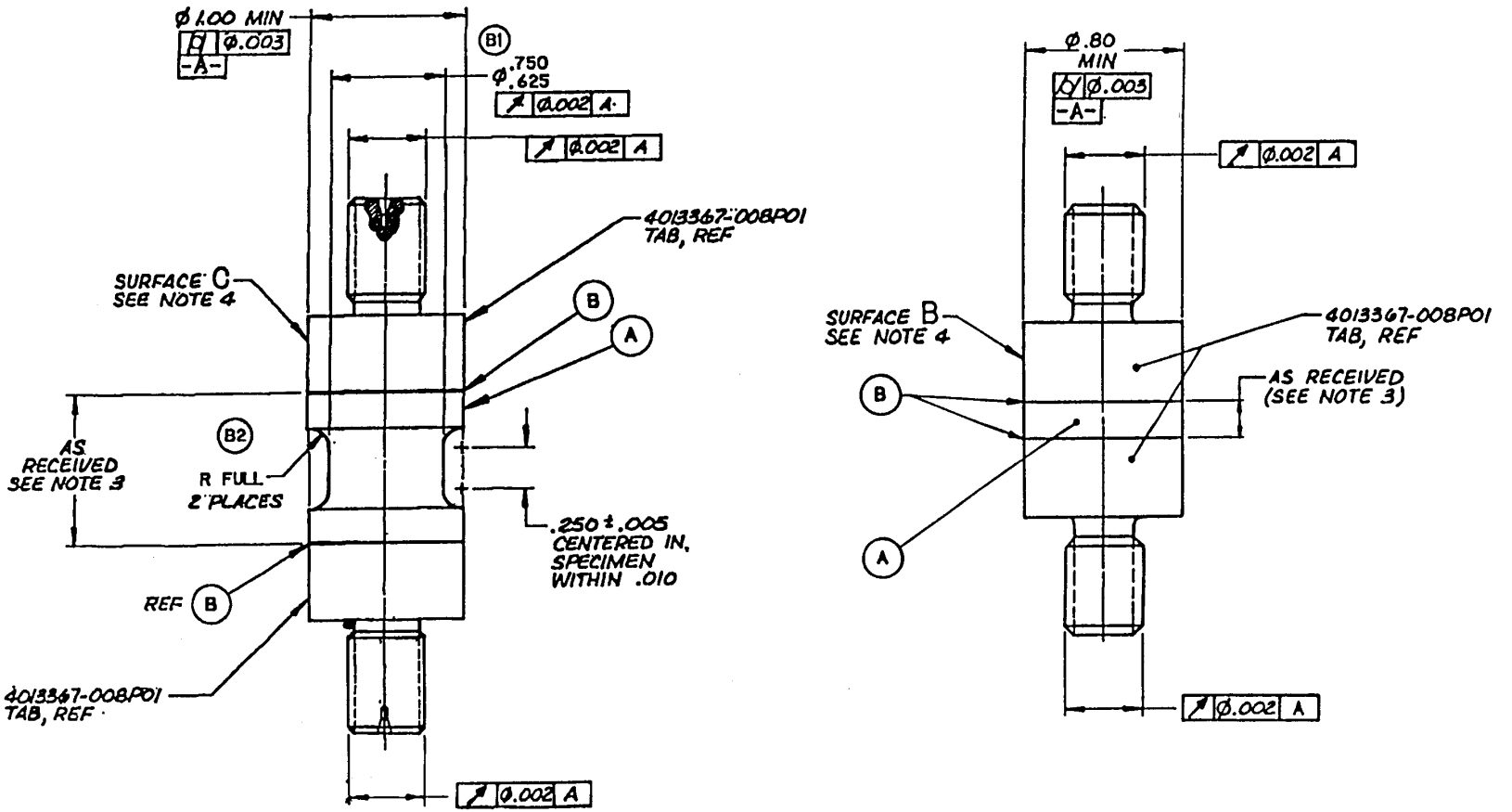
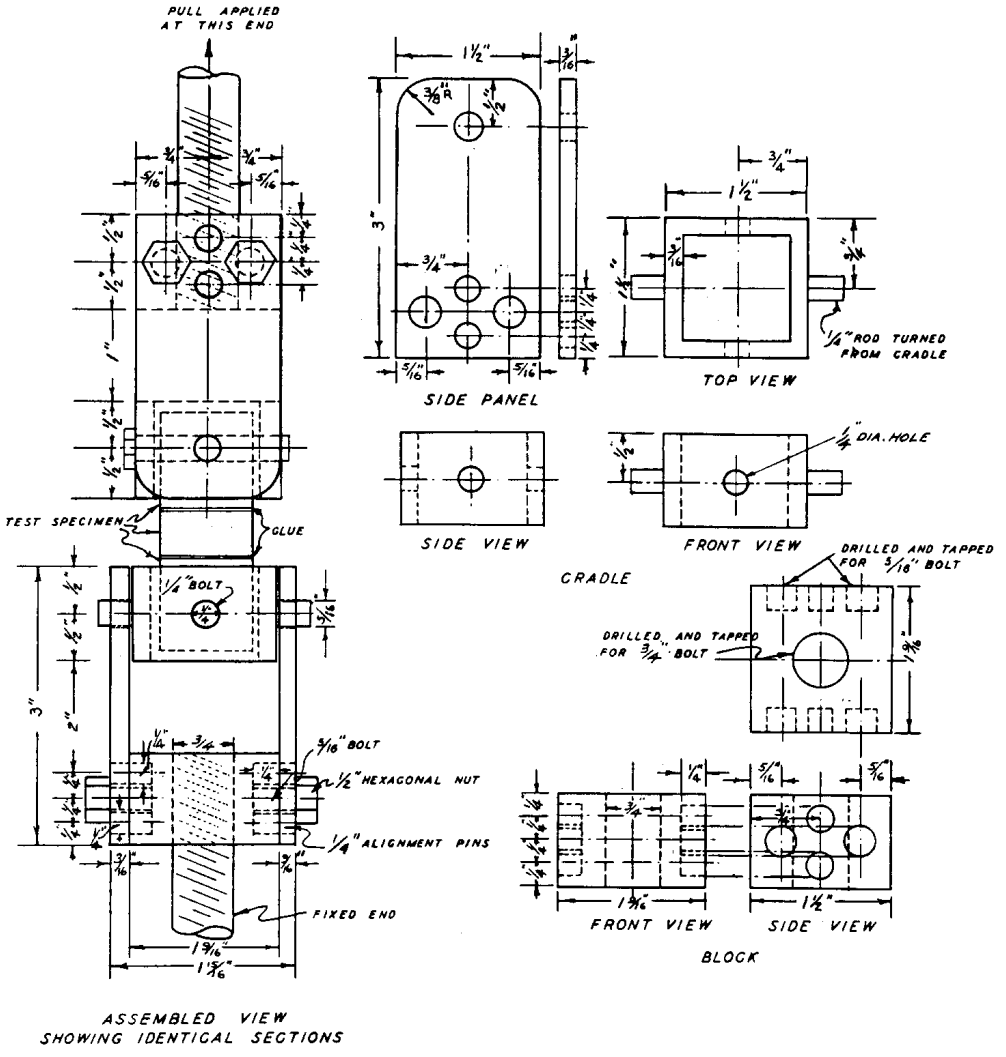


FIGURE 6.8.2.3.2(a) *Two typical direct out-of-plane tensile specimen configurations.*



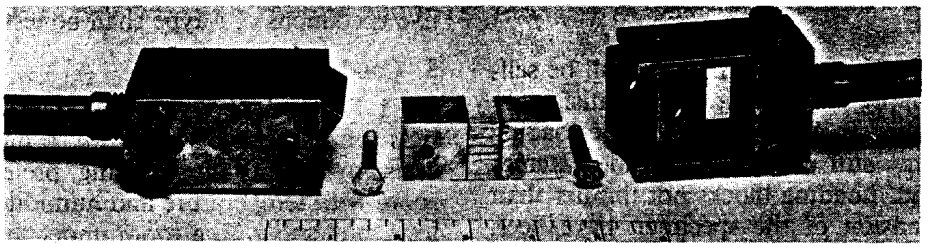
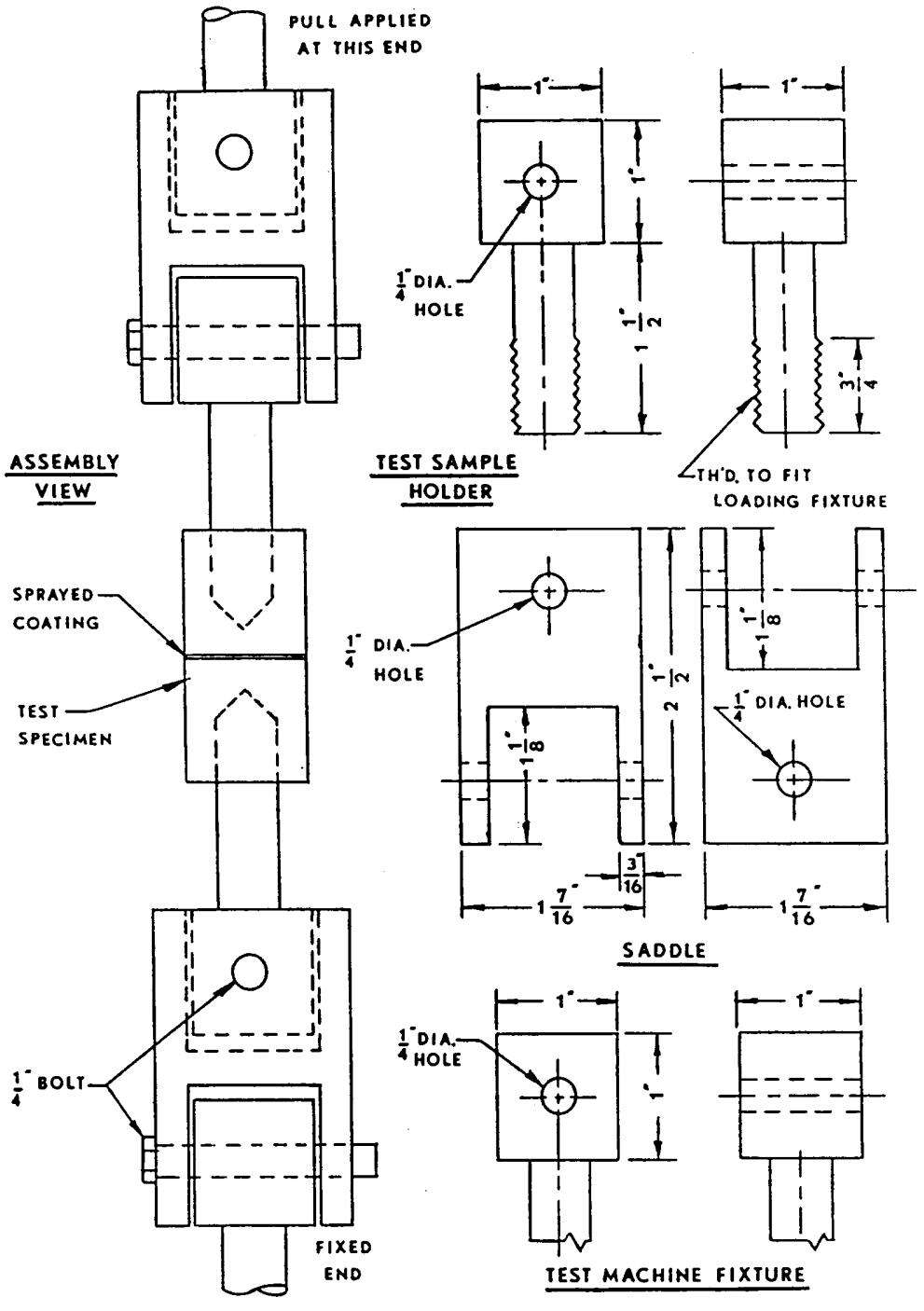


FIGURE 6.8.2.3.2(b) *The ASTM C 297 loading fixture.*



Metric Equivalents

in.	3/16	1/4	3/4	1	1 1/8	1 7/16	1 1/2	2 1/2
(*mm*)	(4.8)	(6.4)	(19)	(25.4)	(29)	(37)	(38)	(64)

FIGURE 6.8.2.3.2(c) *ASTM C 633 end-blocks.*

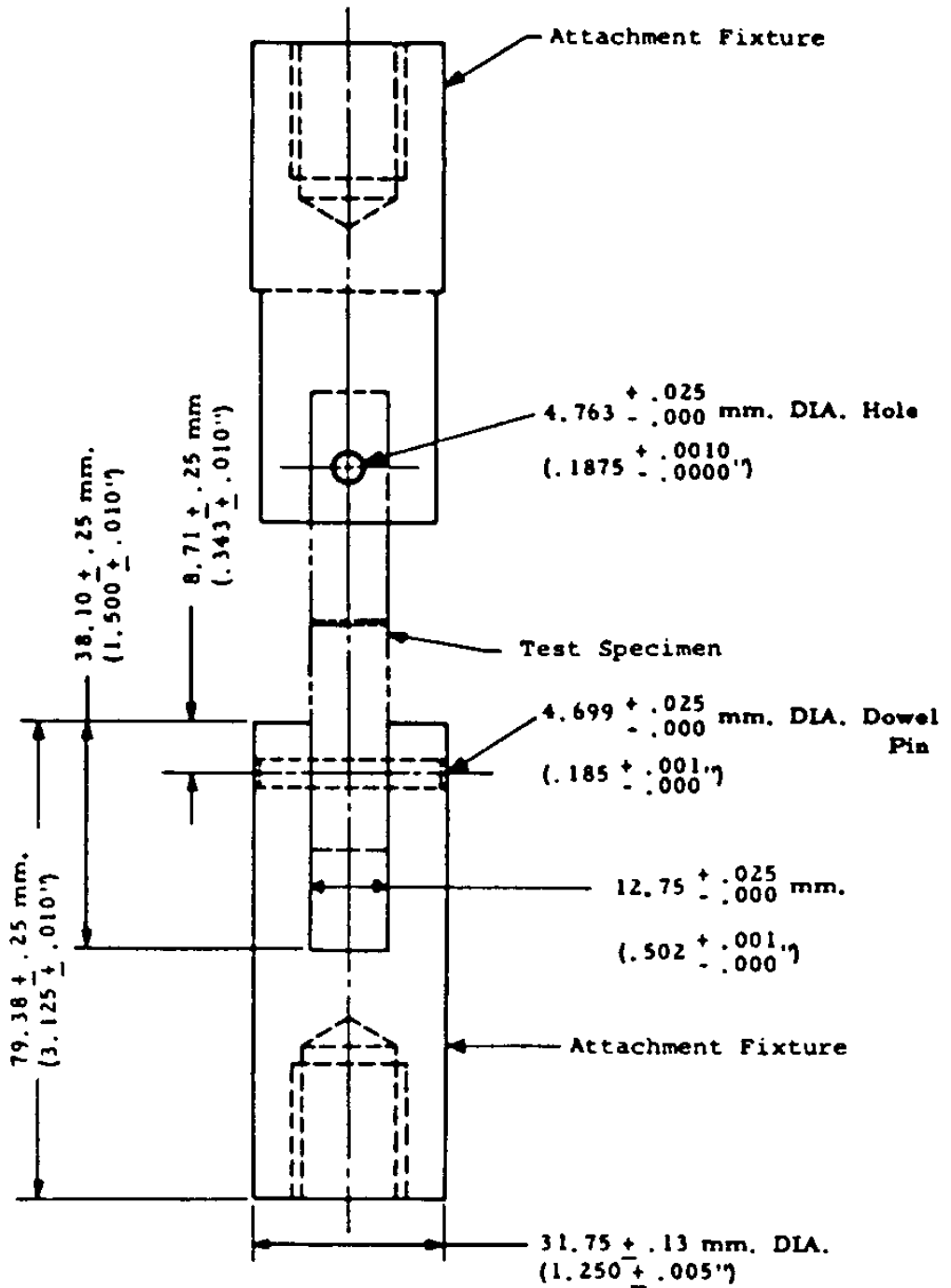


FIGURE 6.8.2.3.2(d) *The ASTM D 2095 loading fixture.*

6.8.2.3.3 Curved beam approach to out-of-plane tensile strength

This technique takes advantage of the out-of-plane tensile loading induced in the elbow of a curved laminate beam subjected to an opening moment. Several researchers have published investigations of different variations of this technique, exploring specimen size, specimen shape (90° small radius or "C"-shaped), and loading methods (attachment fixture used to apply an opening tensile load, or a four-point flexural fixture) (e.g., Reference 6.8.2.3.3). Typical specimen configurations are conceptually illustrated by Figure 6.8.2.3.3.

Limitations:

Standardization: Currently non-standard, although it is being evaluated for possible standardization by ASTM D-30.

Inconsistent results: Early investigations indicate results are strongly geometry and size dependent.

Material response: Unlike the direct out-of-plane loading method with a thick laminate, the specimen cannot be instrumented for out-of-plane strain and therefore modulus cannot be determined.

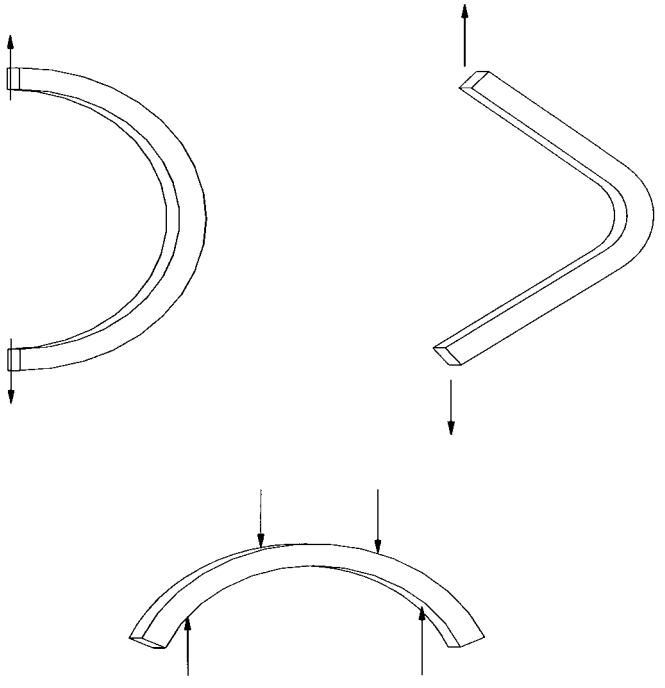


FIGURE 6.8.2.3.3 *Curved beam loading approaches.*

6.8.2.4 Tension test methods for MIL-HDBK-17 data submittal

Data produced by the following test methods (Table 6.8.2.4) are currently being accepted by MIL-HDBK-17 for consideration for inclusion in Volume 2.

TABLE 6.8.2.4 *Tension test methods for MIL-HDBK-17 data submittal.*

	Symbols	Fully Approved, Interim, and Screening Data	Screening Data Only
LAMINA PROPERTIES			
0° In-Plane Strength	F_1^{tu}, ε_1^{tu}	D 3039, SRM 4, SRM 9 (crossply only)	---
0° In-Plane Modulus, Poisson's Ratio	E_1^t, ν_{12}^t	D 3039, SRM 4	---
90° In-Plane Strength	F_2^{tu}, ε_2^{tu}	D 3039, SRM 4, D 5450	---
90° In-Plane Modulus	E_2^t	D 3039, SRM 4, D 5450	---
Out-of-Plane Strength	F_3^{tu}, ε_3^{tu}	(no recommendation)	---
Out-of-Plane Modulus, Poisson's Ratios	E_3^t, ν_{31}^t, ν_{32}^t	(no recommendation)	---
LAMINATE PROPERTIES			
x In-Plane Strength	F_x^{tu}, ε_x^{tu}	D 3039	---
x In-Plane Modulus, Poisson's Ratio	E_x^t, ν_{xy}^t	D 3039	---
y In-Plane Strength	F_y^{tu}, ε_y^{tu}	D 3039	---
y In-Plane Modulus	E_y^t	D 3039	---
Out-of-Plane Strength	F_z^{tu}, ε_z^{tu}	(no recommendation)	---
Out-of-Plane Modulus, Poisson's Ratios	E_z^t, ν_{zx}^t, ν_{zy}^t	(no recommendation)	---

6.8.3 Compressive properties

In-Plane Compressive Properties

Lamina

$E_1^c,\ \nu_{12}^c,\ F_1^{cu},\ \varepsilon_1^{cu}$

$E_2^c,\ \nu_{21}^c,\ F_2^{cu},\ \varepsilon_2^{cu}$

$E_3^c,\ \nu_{31}^c,\ F_3^{cu},\ \varepsilon_3^{cu},\ \nu_{32}^c$

Laminate

$E_x^c,\ \nu_{xy}^c,\ F_x^{cu},\ \varepsilon_x^{cu}$

$E_y^c,\ \nu_{yx}^c,\ F_y^{cu},\ \varepsilon_y^{cu}$

$E_z^c,\ \nu_{zx}^c,\ F_z^{cu},\ \varepsilon_z^{cu},\ \nu_{zy}^c$

6.8.3.1 Overview

The compressive response of composite materials has been the subject of research efforts and test programs since the early 1970's. Yet there remain numerous methods in use for testing composites in compression and no consensus as to which should be recommended.

Compression tests are conducted on composite materials, utilizing appropriate instrumentation, to determine compressive modulus, Poisson's ratio, ultimate compressive strength and/or strain-at-failure. These properties are determined through use of test fixturing that is typically designed 1) to introduce a uniform state of uniaxial stress in the specimen test section, 2) to minimize stress concentrations, 3) to be as simple to use and fabricate as possible. and 4) to minimize specimen volume. Compressive data are used for various purposes including research, quality control and generation of design allowables.

Measures of quality for a particular compressive test method include low coefficients of variation for strength and modulus, as well as the value of the modulus obtained relative to that from other compression test methods. While relative compressive strength is also often used as another measure of compression test quality, the inherent differences in compression response between the different compression tests mean that the test fixture, the resulting failure modes, and the application must be considered along with the resulting strengths. Compressive strengths from some methods may be considered "artificially high" due to fixture/coupon restraints that may suppress certain "real world" failure modes. Typically, fixtures are designed to induce failure in the test section and to intentionally inhibit some failure modes such as end brooming and column buckling that, if permitted to occur, would result in "artificially low" strengths. This tradeoff between just enough restraint versus too much restraint, and artificially high versus artificially low compressive strength, is the reason for the myriad of possible test methods and the lack of agreement on one acceptable method. There are differences of opinion on how to balance these tradeoffs. Final selection of a compressive test method depends on the goals of the testing program.

The measured compressive strength for a single material system has been shown to differ when determined by different test methods. Other parameters found to be significant contributors to the variations in results include fabrication practices, control of fiber alignment, improper and/or inaccurate specimen machining, improper tabbing procedures if tabs are used, poor quality of the test fixture, improper placement of the specimen in the test fixture, improper placement of the fixture in the testing machine, and an improper test procedure.

A review of the numerous compression test methods available reveals they can be broadly classified into three groups: 1) those that introduce load into the specimen test section through shear, 2) those that introduce load into the specimen test section through direct compression (end-loading), and 3) those that introduce load into the specimen test section through a combination of end-loading and shear. The two compressive test methods for fiber-reinforced composites currently published by ASTM Committee D-30 in D 3410-95 (Reference 6.8.3.1(a)) and the one method in D 5467-97 (Reference 6.8.3.1(b)) introduce load into the test section of the specimen primarily by shear. ASTM D 695 (Reference 6.8.3.1(c)),

SACMA SRM-1R-94 (Reference 6.8.3.1(d)), and SRM-6-94 (Reference 6.8.3.1(e)) utilize end loading. ASTM D 6484 (Reference 6.8.3.1(f)) is a combined loading test method.

Compressive test methods can be further classified as having a supported or an unsupported test section. An unsupported test section is defined as one with nothing in contact with the surfaces of the specimen in the test section throughout the entire compression test. A supported test section is one with support on the specimen faces and/or edges in the test section provided by the test fixture or ancillary equipment. All of the test methods discussed in this section utilize specimens with unsupported test sections, with the exception of ASTM D 5467, the Sandwich Beam method. A more complete discussion of compression test methodology and a description of test methods not covered here can be found in References (6.8.3.1(g) - (*l*)).

6.8.3.2 In-plane compression tests

The in-plane compressive test methods described below are typically used to generate the ultimate compressive strength, strain-at-failure, modulus, and Poisson's ratio of axially or transversely loaded unidirectional composite specimens, over a typical thickness range of 0.040 to 0.400 in. (1 to 10 mm). With the exception of the D 5467 (sandwich beam) method, all of the test methods discussed below will also accommodate specially orthotropic laminates, including $[0/90]_{ns}$ style laminates. The testing of $[0/90]_{ns}$ laminates has become a particularly popular means of eliminating specimen- and fixture-related sensitivities associated with the use of unidirectional specimens. If lamina compressive data are desired from $[0/90]_{ns}$ laminates, data reduction procedures are required. A discussion of the use of [0/90] laminates for determining lamina properties, and the associated data reduction methods, can be found in Section 2.4.2, and in SACMA SRM 6.

The test methods discussed here can often be used for specimen thicknesses greater than those indicated above. Additional information on testing laminates thicker than 0.400 in. (10 mm) can be found in Volume 3, Chapter 6.

General Limitations of In-Plane Compression Testing

Test Method Sensitivity - Measured compressive strength for a single material system has been shown to differ when determined by different test methods. Such differences can be attributed to specimen alignment effects, specimen geometry effects and fixture effects, even though efforts have been made to minimize these effects. Examples of the differences in test results between the two procedures in ASTM D 3410-95 and the one procedure in ASTM D 5467-97 can be found in References 6.8.3.2(a) and (b).

Material and Specimen Preparation - Compressive modulus, and especially compressive strength, are sensitive to poor material fabrication practices, damage induced by improper specimen machining and lack of control of fiber alignment. Fiber alignment relative to the specimen coordinate axis should be maintained as carefully as possible, although no standard procedure to ensure this alignment exists. Procedures found satisfactory include the following: fracturing a cured unidirectional laminate near one edge parallel to the fiber direction to establish the 0° direction, or laying in small filament count tows of contrasting color fiber (aramid in carbon laminates and carbon in aramid or glass laminates) parallel to the 0° direction either as part of the prepreg production or as part of panel fabrication.

6.8.3.2.1 ASTM D 3410/D 3410M, Compressive Properties of Polymer Matrix Composite Materials With Unsupported Gage Section by Shear Loading

Two compression procedures are published by ASTM in Test Method D 3410 (Reference 6.8.3.1 (a)) and have historically been called the Celanese (D 3410, Procedure A), and the IITRI (Illinois Institute Technology Research Institute, D 3410, Procedure B). The Celanese and IITRI procedures, as with many other published procedures, originally carried the names of the organizations under which the procedure

was developed. The Celanese and IITRI procedures address the use of tabbed or untabbed rectangular specimens and transfer load via wedge-type grips.

Limitations of ASTM D 3410

Material Form - Limited to continuous-fiber or discontinuous-fiber reinforced composites for which the elastic properties are specially orthotropic with respect to the test direction.

Test Fixture Characteristics - Although both Procedures A and B transmit load to the specimen via tapered wedge grips, the wedge surfaces in Procedure A are conical and those in Procedure B are flat. The conical wedges in Procedure A are known to be prone to cone-to-cone seating problems (Reference 6.8.3.2(a)). The flat wedge grip design used in Procedure B was employed to eliminate this wedge-seating problem (Reference 6.8.3.2(a)). A fixture characteristic that can have a significant effect on test results is the surface finish of the mating surfaces of the wedge grip assemblies. Since these surfaces undergo sliding contact they must be polished, lubricated, and free of nicks and other surface damage.

Strain Measuring Devices - While compressometers are not ruled out, available space considerations make use of strain gages essentially required. Back-to-back gages are required for a minimum number of specimens for both Procedures A and B.

ASTM D 3410, Procedure A

Procedure A is not used very extensively, and is being considered for removal from ASTM D 3410. Thus it will not be discussed further here. Details are available in ASTM D 3410-95.

ASTM D 3410, Procedure B

The fixture for this test method was designed principally to eliminate the seating problems associated with the conical wedge grips in Procedure A (Reference 6.8.3.2(a)). In place of conical wedge grips, the fixture for this test method consists of flat wedge grips seated in a rectangular housing (Figure 6.8.3.2.1). The fixture for this method is much larger and heavier than that for Procedure A, and can accommodate much larger specimens. The test specimen used in this fixture is typically a tabbed specimen of rectangular cross section with recommended dimensions of 5.5 - 6.0 in. (140 - 155 mm) long, 0.50 - 1.0 in. (10 - 25 mm) wide, and with a of 0.5 - 1.0 in. (10 - 25 mm) gage length. Specimens tested using this procedure have a minimum required thickness, specified as a function of gage length, material moduli, and expected material strength. As with Procedure A, the load applied to the fixture is transferred from the wedge grips to the specimen tabs through shear, and from the tabs to the test specimen through shear. The complex stress state in the tabbed region of the specimen changes to uniaxial compression in the specimen test section. Compression strength is determined from load at failure while modulus and strain-at-failure are determined when strain gages or compressometers are employed.

Limitations of ASTM D 3410, Procedure B

Tabbing and Tolerances - The data resulting from this test method have been shown to be sensitive to the flatness and parallelism of the tabs, so care should be taken to assure that the specimen tolerance requirements are met. This may require precision grinding of the tab surfaces after bonding them to the specimen. The fixture used for this procedure must also be precision machined and assembled, and accurately installed in the testing machine.

6.8.3.2.2 ASTM D 6484, Compressive Properties of Polymer Matrix Composite Laminates Using a Combined Loading Compression (CLC) Test Fixture

As the title implies, this test method applies a combination of end loading and shear loading to the test specimen. A typical test fixture is shown in Figure 6.8.3.2.2. It consists of four blocks clamped in

pairs to either end of the test specimen. The surfaces of the fixture blocks in contact with the specimen are roughened, to increase the effective coefficient of friction and hence the shear load transfer. By adjusting the torque applied to the four screws in each pair of blocks, the ratio of shear loading to end loading can be controlled. The goal is to apply sufficient torque so that the ends of the specimen are not crushed or otherwise fractured by the end loading, but only a minimum more than necessary. Increasing the clamping force on the ends of the specimen increases the through-thickness stresses induced, and the axial stress concentration at the gage section ends of the fixture blocks. While this is the goal, it has been shown (Reference 6.8.3.2.2.(a)) that there is considerable tolerance on the upper end of screw torque (clamping force).

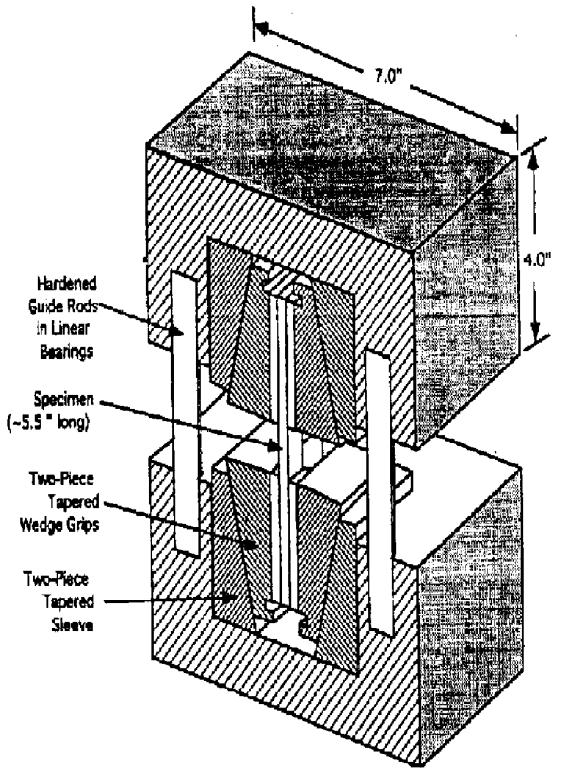


FIGURE 6.8.3.2.1 *Schematic of ASTM D 3410-95 Procedure B test fixture and specimen.*

Because of the favorable load introduction aspects of combined loading, it is possible to test many types of composite materials and laminates without the use of tabs. Tabs always introduce an additional stress concentration in the specimen at the ends of the gage section (References 6.8.3.2.2(b)-(d)). Eliminating tabs also significantly reduces the cost of specimen preparation, and eliminates several inherent factors that are potential sources of error and data scatter. These include variations in tabbing material thickness and adhesive bondline thickness. As an example, untabbed $[0/90]_{ns}$ cross-ply laminates cannot be reliably tested using end loading methods. Crushing of the specimen ends is likely. Such laminates are readily tested using combined loading.

The standard CLC specimen is 5.5 in. ((140 mm) long. This produces a 0.50 in. (12.7 mm) gage length (unsupported length). Longer or shorter gage sections can be obtained by simply altering the total length of the specimen. A specimen width of 0.50 in. (12.7 mm) is recommended for most applications. However, the standard fixture will accommodate a specimen of any width up to 1.2 in (30.5 mm). A specimen thickness on the order of 0.080 to 0.100 in. (0.020 to 0.025 mm) is commonly used. However,

the fixture will accommodate a specimen of any practical thickness. Specimens that are too thin will buckle. Thick specimens will end-crush if the orthotropy ratio of the material being tested is too high (a high enough shear component of the combined loading cannot be attained).

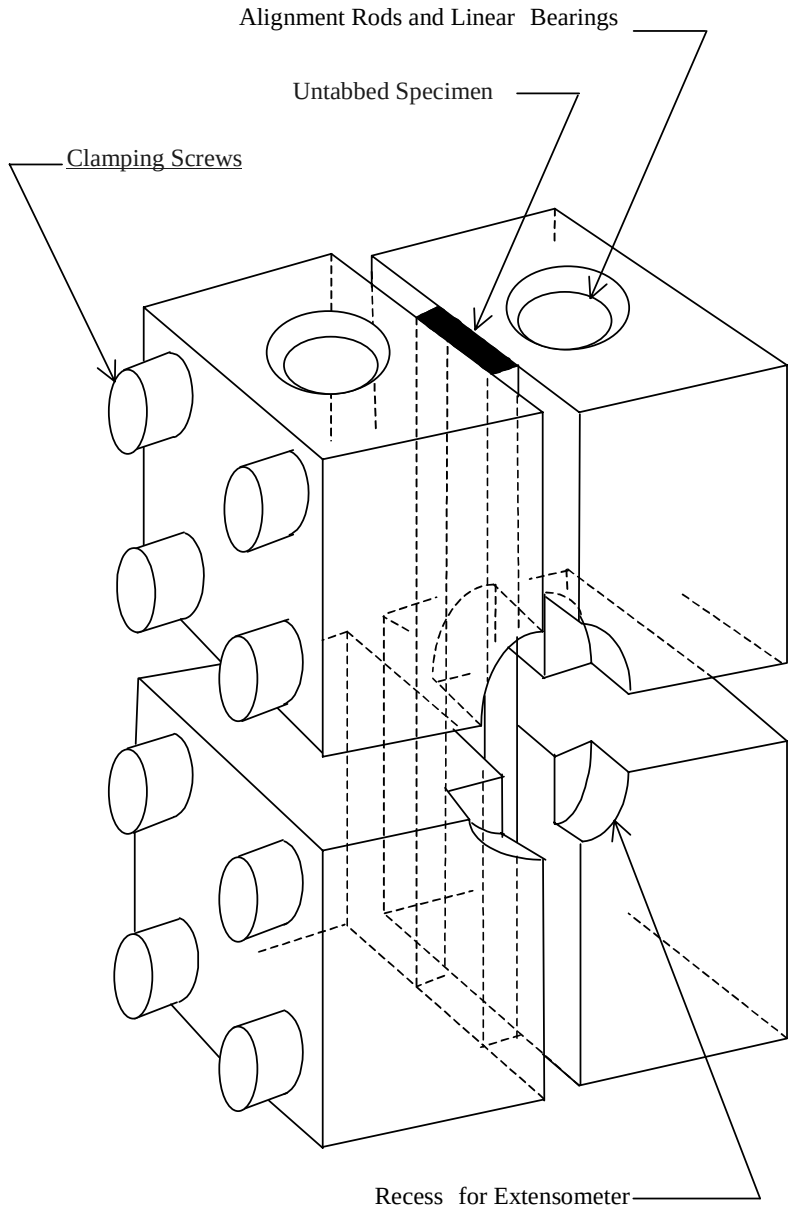


FIGURE 6.8.3.2.2 *Schematic of ASTM D 6484 test fixture with an untabbed specimen installed.*

As for ASTM D 3410, tabbed specimens, even unidirectional composites, can be tested. There is a slight advantage of using combined loading rather than shear loading of tabbed specimens in that the clamping forces do not have to be as high and thus the stress concentrations previously noted are not as high.

Material Forms - most specially orthotropic laminate configurations can be tested using untabbed specimens. Exceptions are laminates with high percentages of 0° plies. Testing laminates with more than 50 percent 0° plies may induce end crushing. Untabbed fabric and braided composites can also be tested. Highly orthotropic unidirectional composite specimens must be tested with tabs.

Test Fixture Characteristics - The test method relies on a high effective coefficient of friction at the specimen-fixture interface, to transmit a significant shear force while keeping the clamping force to a minimum. Thermal-sprayed tungsten carbide particle grip surfaces perform well. Each pair of fixture end blocks must be properly machined and well matched at their outer ends so that when clamped on to the specimen ends they form flat planes perpendicular to the axis of the test specimen. The specimens must also be prepared with flat ends perpendicular to the specimen axis. A specimen is installed in the fixture such that each end of the specimen is flush with the outer ends of a pair of clamping blocks. Then when a compressive force is applied via flat platens, this force is transmitted both by the fixture end blocks and the specimen ends.

Strain Measuring Devices - A typical Combined Loading Compression test fixture is provided with a recess on one side so that a compressometer can be attached to one edge of the specimen in the gage section (Figure 6.8.3.2.2). However, as in ASTM D 3410, the unsupported section is typically only about .50 in (12.7 mm) long, limiting the attachment space available. Attaching extensometers to each face of the test specimen, as recommended to monitor specimen bending and buckling under load, is even more difficult because of the long reach length required. Thus, strain gages are commonly used instead. These can have a very short active gage section length and thus fit into a confined space. Back-to-back gages are required for a minimum number of specimens, to check for specimen bending and buckling.

Limitations of ASTM D 6484

Specimen Dimensions - The standard fixture is designed to grip each end of the untabbed specimen over a length of 2.5 in. (63.5 mm). Thus the specimen must be longer than 5.0 in. (127 mm) in order to establish a gage section. The maximum specimen width the standard fixture will accommodate is 1.2 in. (30.5 mm). As specimen thickness is increased, the through-thickness distribution of the axial compressive stress will become more nonuniform. This may dictate a practical upper limit even though the fixture can accommodate thicker specimens.

Material Forms - Highly orthotropic composite materials cannot be tested using untabbed specimens. The very high clamping forces required to prevent end crushing induce stress concentrations of unacceptable magnitudes at the gage section ends of the clamping blocks.

6.8.3.2.3 ASTM D 5467, Compressive Properties of Unidirectional Polymer Matrix Composites Using a Sandwich Beam

The sandwich beam method (Reference 6.8.3.1(b)) consists of a honeycomb-core sandwich beam that is loaded in four-point bending, placing the upper face sheet in compression (Figure 6.8.3.2.3). The compression face sheet (upper sheet) is a 6-ply unidirectional laminate and the lower face sheet should be the same material and twice as thick. The two face sheets are separated by, and bonded to, a deep aluminum honeycomb core. The upper face sheet is designed to fail in compression when the beam is subjected to four-point bending. The beam is loaded to failure in bending, resulting in the measurement of compressive strength, compressive modulus and strain-at-failure if strain gages or compressometers are employed.

Limitations of ASTM D 5467

Material Form - This test procedure is limited to unidirectional materials.

Specimen Complexity - The sandwich beam specimen is much larger, and specimen preparation is more complex and expensive, than for those in ASTM D 3410 and ASTM D 6484.

Poisson's Ratio - The validity of Poisson's ratio obtained from this method has been questioned due to anticlastic bending.

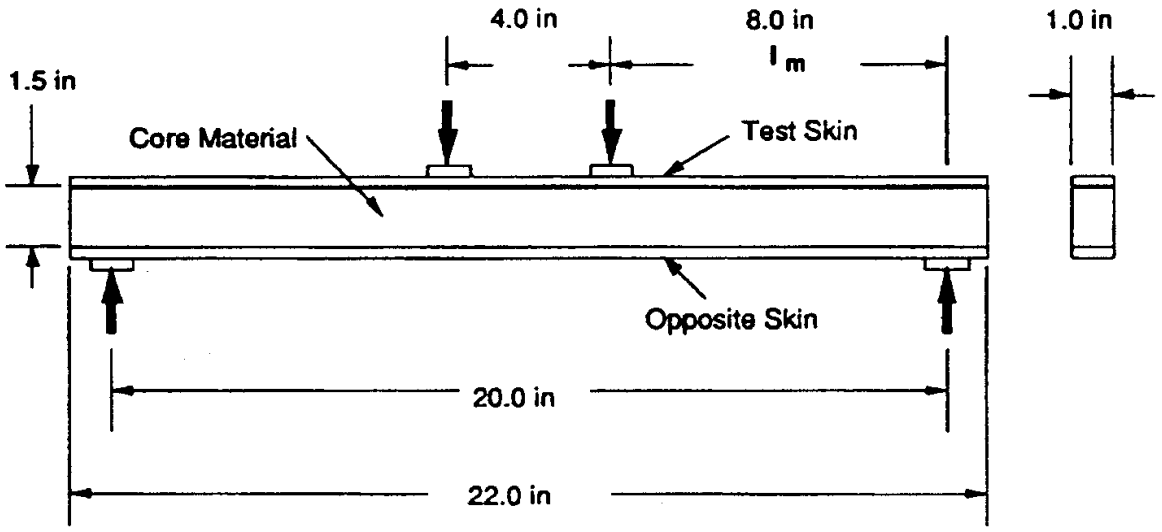


FIGURE 6.8.3.2.3 *Schematic of ASTM D 5467 specimen and loading configuration.*

6.8.3.2.4 ASTM C 393, Flexural Properties of Flat Sandwich Constructions

This test method (Reference 6.8.3.2.4) is one of a series designed to test sandwich constructions, and covers the determination of the properties of flat, sandwich constructions subjected to flatwise flexure in the same manner as ASTM D 5467. While D 5467 is designed to provide data for the compressively loaded face sheet only, ASTM C 393 is used to determine the flexural and shear stiffness of the entire sandwich, shear modulus and shear strength of the core, or compressive or tensile strength of the face sheets. There are no limitations on the core or skin materials for this test method, the specimen is rectangular in cross section, and the core, face, and span geometries are determined to achieve the desired failure mode as a function of material properties. While not widely used for the determination of composite material properties, this test method does allow for the design of a test specimen not covered by ASTM D 5467. Caution should be exercised in using this test for composite material properties since the equations for determining the material properties may not be applicable for some specimen geometries or core/face sheet combinations.

The use of this test method to determine the tensile properties of [90°] laminates is covered in Volume 1, Section 6.8.2.2.5.

Limitations of ASTM C 393

Material Form - This test method is not limited in the material form of the core material or face sheet material. Equations for determining the material properties may not be applicable for some specimen geometries or core/face sheet combinations.

Specimen Geometry - This test method is limited to rectangular sandwich construction and the core, face, and span geometries are allowed to vary in order to achieve the desired failure mode as a function of material properties. Equations for determining the material properties may not be applicable for some specimen geometries or core/face sheet combinations.

6.8.3.2.5 ASTM D 695, Compressive Properties of Rigid Plastics

This method was developed by ASTM Committee D-20 for compression testing of unreinforced and reinforced rigid plastics. Two types of specimens can be used for this method. The first is typically used for unreinforced plastics and is in the form of a right cylinder or prism whose length is twice its principal diameter or width. Preferred specimen sizes are 0.50 in. by 0.50 in. by 1 in. (12.7 mm by 12.7 mm by 25.4 mm) for a prism and 0.50 in. dia. by 1 in. (12.7 mm dia. by 25.4 mm) for a cylinder. Smaller diameter rods or tubes may also be tested provided they are of sufficient length to allow a specimen slenderness ratio of 11:1 to 16:1. The specimen is tested by placing it between the hardened steel faces of a compressive subpress and loading it to failure.

The second test specimen configuration in the standard is documented as being for "reinforced plastics, including high-strength composites and highly orthotropic laminates" < 0.125 in. (6.4 mm) thick. It uses a flat, untabbed dogboned specimen 3.13 in. (79.5 mm) long having a 0.50 in. (12.7 mm) reduced width test section. Two I-shaped (anti-buckling) support plates, slightly shorter than the specimen, and with longitudinal grooves, are lightly clamped to the faces of the specimen, at most a negligible portion of the applied force is transmitted through the support plates (a redundant load path). After positioning the specimen between the support plates, a compressive load is applied directly to the ends of the specimen until failure, to determine compression strength.

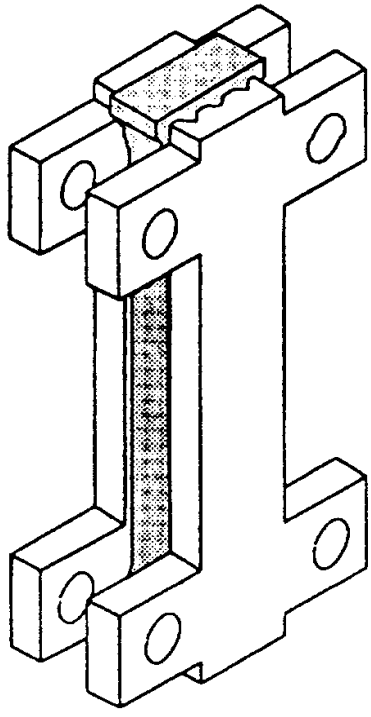


FIGURE 6.8.3.2.5 *Schematic of ASTM D 695 specimen and test fixture.*

The flat dogboned specimen, fixture-supported method was evaluated in a D-30 round robin for [0] AS/3501 and [0] E-Glass/1002 laminates (Reference 6.8.3.2(b)). The conclusion from this study was that this test method is not adequate for determining the compressive strength of high-modulus composite materials in the forms studied in this investigation. (Other forms, such as E-glass fabric-reinforced com-

posites, can be successfully tested using this test method.) In an attempt to modify this portion of the ASTM D 695 test method for use with high-modulus composites, a straight-sided, tabbed coupon has been developed. In addition, an L-shaped base to support the fixture-specimen assembly has also been added to the test method. These modifications were not made by ASTM, nor incorporated into ASTM D 695. A discussion of these modifications is included below in the section on the SACMA SRM 1R test method.

Limitations of the ASTM D 695 Compressive Test Method

Material Form - The published scope of this document states that it is limited to unreinforced and reinforced rigid plastics, including high-modulus composites. But round-robin testing conducted by ASTM Committee D-30 found this method to be unacceptable for the measurement of strength of high modulus composites (Reference 6.8.3.2(b)). However, it should be noted that there was some question as to the validity of the round-robin testing (Reference 6.8.3.2(b)).

6.8.3.2.6 SACMA SRM 1R, Compressive Properties of Oriented Fiber-Resin Composites

A variation of the ASTM D 695 "Standard Test Method for Compressive Properties of Rigid Plastics" for high-modulus continuous fiber composites has been developed and documented by SACMA as SRM 1R (Reference 6.8.3.1(d)). While essentially retaining the simple fixturing of the D 695 method, the variation utilizes straight-sided tabbed specimens for compression strength and an L-shaped base for support of the fixture-specimen assembly. A separate, untabbed specimen must be used for the measurement of modulus. Both specimens are shorter than the D 3410 specimens, being 3.18 in. (80 mm) long, 0.5 in. (6.4 mm) wide and 0.040 to 0.120 in. (1 to 3 mm) thick. Although the test section is unsupported, it is very short (0.188 in. (4.8 mm)).

This test method tends to produce slightly higher average values of compression strength (5 to 10 percent) than ASTM D 3410 Procedures A and B. One probable reason is the slightly more uniform stress state in the gage section produced by the end loading. However, as for ASTM D 695, a fixture-induced redundant load path through the lateral supports can be significant if the clamping force is too high. SRM-1R specifies that the clamping screws be torqued to 6-10 in-lb (0.7-1.0 Joules). This is discussed further in (Reference 6.8.3.1(*l*)). A schematic of this test method with the compressive strength specimen in place is shown in Figure 6.8.3.2.6.

Limitations of the SACMA SRM 1R Compressive Test Method

General - Separate strength and modulus specimens are required for this test method. The short specimen gage length results in a small test section volume for the strength specimen.

Material Form - The specific scope of this test method is interpreted by the Handbook coordination group to be for 0° direction properties of unidirectional specimens, and 0° and 0° direction properties of fabric specimens only. This test method is applicable to fabric-based materials only when the unit cell size of the specimen weave/braid is smaller than the 0.188 inch (4.8 mm) gage length of the specimen.

Compressive Strength - Measured compressive strengths obtained using this test method are typically higher than those obtained using the methods in ASTM D 3410, as previously discussed. It is important to avoid a redundant load path induced by using clamping screw torques that are too high.

Strain-at-failure - This test method will not provide strain-at-failure since the gage region of the strength specimen is not large enough for a strain gage, and the (untabbed) modulus specimen geometry is not suitable for loading to failure. Consequently, stress-strain response, including monitoring of specimen bending strains as commonly done to assess proper gage section loading, cannot be observed over most of the actual stress-strain response.

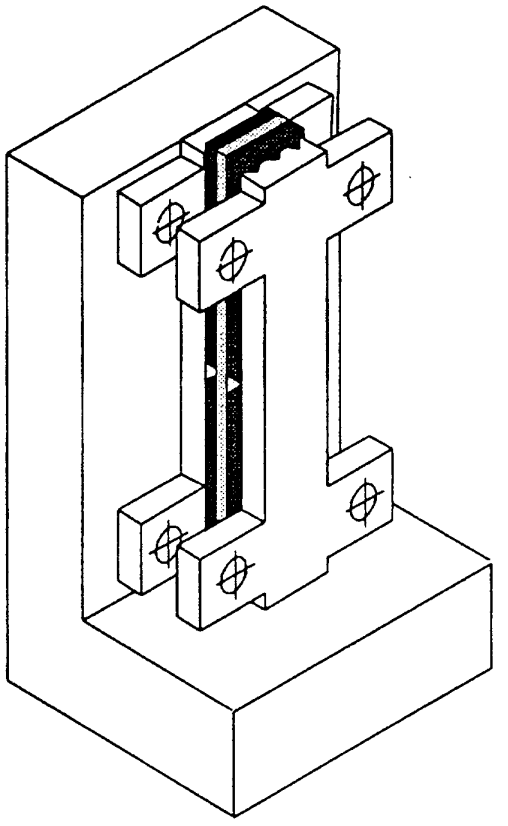


FIGURE 6.8.3.2.6 *Schematic of SACMA SRM 1-88 test fixture and specimen.*

6.8.3.2.7 SACMA SRM 6, Compressive Properties of Oriented Cross-Plied Fiber-Resin Composites

This test method (Reference 6.8.3.1(e)) is identical to SACMA SRM 1R with the exception that it is limited in material form to cross-plied laminates. This limitation is applied since the method is intended for the determination of unidirectional composite compressive strength by applying a back-out factor to the strength determined from the cross-plied laminate.

The procedure is to test a $[90/0]_{ns}$ (e.g., n=3 or 6) laminate[1] to failure in compressive as outlined in this method (which is the same as in SACMA SRM 1R). The compressive strength of this laminate is then multiplied by a factor to determine the effective strength of the unidirectional plies that make up the $[90/0]_{ns}$ laminate, as follows:

$$\sigma_{c,uni} = F \times \sigma_{c,lam}$$

$$\text{where } F = \frac{2E_{11}}{E_{11} + E_{22}}$$

The moduli (E_{11} and E_{22}) of the unidirectional composite material must be determined by separate tests of a unidirectional composite material. A more complete discussion of the limitations and assumptions associated with the use of cross-ply laminate data to back out unidirectional composite data is presented in Section 2.4.2. The strength back-out factor in SACMA SRM 6 is an approximation and is not the same as the factors recommended in Section 2.4.2.

[1] For example, for a carbon/epoxy composite, n = 3 is typically used for the laminate lay-up when a prepreg material with an areal weight greater than or equal to 100 g/m^2 is tested, and n=6 for areal weights less than 100 g/m^2.

Limitations of the SACMA SRM 6 Compressive Test Method

General - Separate strength and modulus specimens are required for this test method. The short specimen gage length results in a small test section volume for the strength specimen. The use of the back-out factor in this test method assumes linear elastic response of the material it is being applied to.

Material Form - Limited to cross-plied, polymer matrix composites reinforced with oriented, continuous fibers, and made primarily of prepreg or similar product forms. The short gage length prohibits its use for fabric-based and braided materials when the unit cell size of the specimen weave/braid is larger than the 0.188 in. (4.8 mm) gage length.

Compressive Strength - As for ASTM D 595 and SACMA SRM 1R, a fixture-induced redundant load path through the lateral supports can be significant if the clamping force is too high. As for SRM-1R, SRM 6 specifies that the clamping screws be torqued to 6-10 in-lb. (0l7-1.0 Joules). This is discussed further in (Reference 6.8.3.1(*l*)).

Strain-at-failure - This test method does not provide strain-at-failure since the gage region of the strength specimen is not long enough for a strain gage, and the (untabbed) modulus specimen geometry is not suitable for loading to failure. Consequently, stress-strain response, including monitoring of specimen bending strains, as is commonly done to assess proper gage section loading, cannot be observed over most of the actual stress-strain response.

6.8.3.2.8 Through-thickness compression tests

Due to an historical lack of need for through-thickness compressive data, there are no standardized or widely accepted test methods to determine the through-thickness (z-direction) compressive strength, modulus or Poisson's ratio of composite laminates. These data have been reported to a limited extent in the literature (References 6.8.3.2.8(a) and (b)), and simple rectilinear specimens cut from thick-section laminates have been used to obtain these properties.

6.8.3.3 Compressive test methods for developing MIL-HDBK-17 data submittal

Data provided by the following test methods (Table 6.8.3.3) are currently being accepted by MIL-HDBK-17 for consideration for inclusion in Volume 2.

TABLE 6.8.3.3 *Compressive test methods for MIL-HDBK-17 data submittal.*

	Symbol	Fully Approved, Interim and Screening Data	Screening Data Only
Lamina Properties			
0° In-Plane Strength	F_1^{cu}, ε_1^{cu}	D 3410, D 6484, D 5467, SRM 1R[1,2], SRM 6[1,2]	---
0° In-Plane Modulus, Poisson's Ratio	E_1^c, ν_{12}^c	D 3410, D 5467[3], SRM 1R[2]	---
90° In-plane Strength	F_2^{cu}, ε_2^{cu}	D 3410, D 6484, SRM 1R[1,2,4]	---
90° In-Plane Modulus, Poisson's Ratio	E_2^c, ν_{21}^c	D 3410, D 6484, D 5467[3], SRM 1R[2,4]	---
Out-of-Plane Strength	F_3^{cu}, ε_3^{cu}	no recommendation	---
Out-of-Plane Modulus, Poisson's Ratio	E_3^c, ν_{31}^c, ν_{32}^c	no recommendation	---
Laminate Properties			
x In-Plane Strength	F_x^{cu}, ε_x^{cu}	D 3410, D 6484	---
y In-Plane Strength	F_y^{cu}, ε_y^{cu}	D 3410, D 6484	---
x In-Plane Modulus, Poisson's Ratio	E_x^c, ν_{xy}^c	D 3410, D 6484	---
y In-Plane Modulus, Poisson's Ratio	E_y^c, ν_{yx}^c	D 3410, D 6484	---
Out-of-Plane Strength	F_z^{cu}, ε_z^{cu}	no recommendation	---
Out-of-Plane Modulus, Poisson's Ratio	E_z^c, ν_{zx}^c, ν_{zy}^c	no recommendation	---

[1] Not approved for ε_1^{cu} nor ε_2^{cu}

[2] Not approved for fabric-based materials when the unit cell size of the specimen weave/braid is larger than the 0.188 inch (4.8 mm) gage length.

[3] Not approved for ν_{12}^c nor ν_{21}^c

[4] Approved for fill direction properties in cross-plied fabric based specimens only.

6.8.4 Shear properties

6.8.4.1 Overview

In-Plane Shear Properties: [1,2]
G_{12}, F^{so}_{12}, F^{su}_{12}, γ^{su}_{12}
G_{xy}, F^{so}_{xy}, F^{su}_{xy}, γ^{su}_{xy}

Out-of-Plane Shear Properties:
G_{23}, F^{so}_{23}, F^{su}_{23}, γ^{su}_{23},
G_{yz}, F^{so}_{yz}, F^{su}_{yz}, γ^{su}_{yz}

G_{31}, F^{so}_{31}, F^{su}_{31}, γ^{su}_{31}
G_{zx}, F^{so}_{zx}, F^{su}_{zx}, γ^{su}_{zx}

Short-Beam Strength Properties:
F^{sbs}_{31}, F^{sbs}_{zx}

Shear testing of composite materials has proven to be one of the most difficult areas of mechanical property testing in which to define a rigorously correct test, especially in the out-of-plane direction. A number of test methods have been devised, only some of which are described herein. Many of these methods were originally developed for materials other than continuous fiber reinforced composites, such as metal, plastic, wood, or adhesive. Several of the methods are not yet fully standardized for composite materials, and none of the methods is without deficiency or limitation, though some are clearly more desirable than others.

While there is general agreement regarding the accuracy of shear modulus measurements (for properly conducted tests), the biggest difficulty with shear testing of composites is determination of shear strength. The presence of edge effects, material coupling effects, nonlinear behavior of the matrix or the fiber/matrix interface, imperfect stress distributions, or the presence of normal stresses make shear strength determination from existing shear test methods highly questionable; depending on the test, results may under- or over-estimate shear strength. Due to this uncertainty, shear strength data to be used structural applications should be reviewed on a case-by-case basis for each given application.

A growing body of experience with composite shear testing, both published and unpublished, has led to a greater understanding of the strengths and weaknesses of each test method. At the Fall 1991 ASTM Committee D30 meetings, discussions in the D30.04.03 Section on Shear Test Methods led to the first two of the following conclusions. During the Spring 1993 meetings, this group added the third conclusion. These philosophies are being included in existing and future ASTM standard shear test methods:

1. There are no known standard (or non-standard) test methods that are capable of producing a perfectly pure shear stress condition to failure for every material system, although some test methods can come acceptably close on specific material systems, as judged by the end-user for a given engineering purpose.
2. The strengths resulting from test methods that do not consistently produce a reasonable approximation of pure shear, or that do not fail via a shear failure mode, should not be termed “shear strength.”
3. Since ultimate strength values from existing shear tests are no longer believed able to provide an adequate criterion for comparison of material systems, the addition of an offset strength is now recommended (0.2% offset, unless otherwise specified).

[1] Note that shear properties generally assume subscript independence, where, for example, $F^{su}_{23}=F^{su}_{32}$, *etc.* For common engineering materials in the principal material coordinate system, this is a commonly accepted and generally accurate assumption. However, in unbalanced, multi-directional laminates, the shear stiffness, shear strength, or both, can be direction dependent. In such laminates this arises primarily from the difference in behavior between tension and compression of the fibers that are oriented off-axis relative to the loading direction.
[2] Transverse isotropy, a common assumption for many material systems, implies that $G_{12}=G_{13}$.

With the highly nonlinear stress-strain behavior of many filamentary composites, and especially with high-elongation material systems, it is common to terminate a shear test prior to actual specimen failure. Following the lead of MIL-HDBK-17, ASTM D30 currently recommends ending shear testing at 5% shear strain, if failure has not previously occurred. The rationale for this is included in the following discussions.

Practical usage in structural laminates---Typical structural laminates are designed to have fibers aligned with major load-carrying directions. In the case of shear, the shear loads are usually carried in fiber tension or compression by plies oriented at the appropriate angle. Since, from basic mechanics of materials, the shear strain in the matrix of a given ply cannot be more than twice the fiber axial strain of another ply oriented at 45° to the first ply, we can see that an upper bound on the useful engineering value of shear strain is twice the tensile or compressive fiber strain. As the most ductile structural fibers currently fail well below 2.5%, a practical upper limit for shear strain in a structural laminate would be 5%. Terminating shear test data at this shear strain value is a practical recommendation that saves time in testing and yields a more structurally attainable, and therefore more meaningful, lower-bound estimate of ultimate shear strength.

Limitations of common shear test methods---There are kinematic limitations with both the ±45° tensile shear test and the V-notched beam (Iosipescu) shear test, due to excessive scissoring of the fibers. The work of Kellas et al (Reference 6.8.4.1) showed that the initial condition of ±45° ply geometry changes significantly at high shear strains. Based on their estimate of the relationship between fiber scissoring and shear strain, the test results for these tests become questionable past 5% shear strain, which then becomes a practical upper strain limit for these test methods.

A different issue that results in a similar restriction involves the use of typical strain gages. If gages are used for strain measurement, as is feasible with some tests and required by others, the typical gage limit of about 3% extensional strain equates to roughly 6% shear strain, making this a practical limit for shear strain measurement that is similar in value to the kinematic restriction.

Laminate Testing --- Certain shear test methods, like the ±45° tensile shear test, are by their very nature capable of testing only certain types of laminates. And, as difficult as determination of ultimate material shear strength is for current shear test methods, shear strength of a multi-directional laminate is even more problematic. While several of the shear test methods discussed herein are capable of determining a substantial portion of a laminate stress-strain curve and with it a shear modulus, there is no standard test method that has been shown to adequately determine the ultimate shear strength of a multi-directional laminate. A modification of ASTM D4255 (rail shear test method) using bonded, tapered, tabs have been suggested for shear strength testing of multi-directional laminates, but interest to date has not been sufficient to either standardize this modification or allow MIL-HDBK-17 to recommend it for widespread use. MIL-HDBK-17 Testing Working Group will continue to follow developments in this area.

6.8.4.2 In-plane shear tests

6.8.4.2.1 ±45° tensile shear tests

1) ASTM D 3518/D 3518M-94, Test Method for In-Plane Shear Response of Polymer Matrix Composites by Tensile Test of a ±45° Laminate
2) SACMA SRM 7R-94, In-plane Shear Stress-Strain Properties of Oriented Fiber-Resin Composites.

This test (References 6.8.4.2.1(a) and (b)) for in-plane shear properties consists of a modified ASTM Test Method D3039 tensile test of a specimen having a ply lay-up of the [±45]ns family. Away from the gripping region the in-plane shear stress in this specimen can be shown to be a simple function of the average applied tensile stress, allowing for straightforward calculation of the shear response of the material. This test method has the advantages of a simple test specimen, requires no fixturing, and measurement of strain can be performed using either extensometers or strain gages.

Originally applied only to unidirectional materials, the 1994 release of the standard now includes many woven fabric materials, but the test method is inherently restricted to determination of properties in the 1-2 material plane. The SACMA version has historically been a restricted subset of the ASTM standard; though there have been minor differences between current releases of the two methods in the past. However, the versions listed herein have several significant differences. The 1994 SACMA test method does not include several significant changes that were made to D 3518 in 1994, and though the basic physics of the test remain identical, the details of data reduction are now distinctly different so the two version are not fully equivalent. D 3518 now defines the chord modulus from 2000 to 6000 shear microstrain (versus 500-3000 in SRM 7R-94), and terminates the test at 5% shear strain (or failure, whichever comes first), while SRM 7R-94 still defines strength based only on ultimate load. D 3518 also has added an offset strength, which SRM 7R-94 does not include.

Good modulus agreement has been shown between the ±45° test method and other shear test methods (References 6.8.4.2.1(c) through (e)), although the stress-strain response has been shown to be underestimated at shear strain levels above 1.3% (Reference 6.8.4.2.1(f)). There is a feeling by many in the aerospace composite structures community that while the stress state of this specimen may not be “pure,” it does respond in a manner that mimics the actual stress state and ply interaction within a structural laminate. The resulting response yields an “effective” shear modulus that may be preferred by the designer.

Previous versions of the ASTM standard lacked sufficient definition of several test parameters that have since been found to have significant effects on the ultimate strength of this specimen. It has been shown (Reference 6.8.4.1) that this specimen does not fail due to in-plane shear, but rather due to complicated interactions that are sensitive to material toughness, ply stacking sequence, ply count, ply thickness, edge effects, and surface ply constraints. The 1994 release of D 3518 provides additional controls that improve this situation:

- “Ultimate shear strength” has been replaced with “shear stress at 5% shear strain”, since it is now agreed that this test cannot determine a true ultimate material strength. This new quantity is analogous to the old ultimate strength value, but since it is calculated differently, for many material systems they will not be exact equivalents, and may be significantly different.
- Adds an offset shear strength (a more meaningful quantity for material comparison than the previous “ultimate” shear strength).
- Terminates the test at 5% shear strain, if the specimen has not already failed by rupture.
- Changes the chord shear modulus to use a strain range (2000-6000 shear microstrain) that is consistent with the tensile chord modulus strain range (1000-3000 microstrain).
- Provides requirements for ply lay-up that assure that the most brittle modes of failure will be avoided, and increase the likelihood that data comparisons will be more meaningful.

Refer to the references, or to the discussion within the ASTM standard itself, for more details.

Limitations of the ±45° tensile shear test:

Material and Laminate Form---Limited to materials available in a fully balanced and symmetric ±45° specimen. As discussed above, the stacking sequence, ply count, and ply thickness have a direct effect on specimen strength. Low ply count laminates and repeating (or very thick) plies have a deleterious effect on strength and are restricted in the new standard.

Inhomogeneous Materials---The material is assumed homogenous with respect to the size of the test section. Material forms with features that are relatively coarse with respect to the test section width, such as woven or braided textiles with a coarse repeating pattern, require a larger, currently nonstandard, specimen width.

Impurity of Stress State---The material in the gage section is not in a state of pure in-plane shear, as an in-plane normal stress component is present throughout the gage section, and a complex stress

field exists near the free edges. Although the specimen is believed to provide reliable initial material response, and can establish shear stress-strain response well into the nonlinear region, the calculated shear stress at failure does not represent the material strength, which is why the ASTM standard now terminates the test at 5% shear strain.

Effects of Large Deformation---The extreme fiber scissoring that can occur in this specimen in ductile specimens changes the fiber orientation progressively with increasing strain, conflicting with the fiber orientation assumptions used in the calculation of results. This is a second reason why the test is now terminated 5% shear strain.

6.8.4.2.2 Iosipescu shear test

ASTM D 5379/D 5379-93, Test Method for Shear Properties of Composite Materials by the V-Notched Beam Method.

The V-notched beam shear test (often called the Iosipescu test in the literature) has been standardized for composites by ASTM Committee D-30 in ASTM D 5379/D 5379M-93 (Reference 6.8.4.2.2(a)). The concept for the v-notched beam shear test for strength and modulus was originally identified in the late 1950's and early 1960's by Arcan (References 6.8.4.2.2(b) through 6.8.4.2.2(d)) and Iosipescu (Reference 6.8.4.2.2(e) through 6.8.4.2.2(g)) for use on metals. Subsequent usage was limited until detailed investigations were begun on an improved specimen and fixture at the University of Wyoming under NASA funding during the early 1980's (References 6.8.4.2.2(h) and (i)). The fixture was subsequently modified (References 6.8.4.2.2(j) and (k)), and this latter Wyoming configuration formed the basis for the ASTM standard. This method has been investigated extensively; see References 6.8.4.2.2(l) through 6.8.4.2.2(r)) for additional investigations. Early historical perspectives are given in References 6.8.4.2.2(h) and 6.8.4.2.2(s). However, the remainder of the discussion focuses on the configuration that has been standardized.

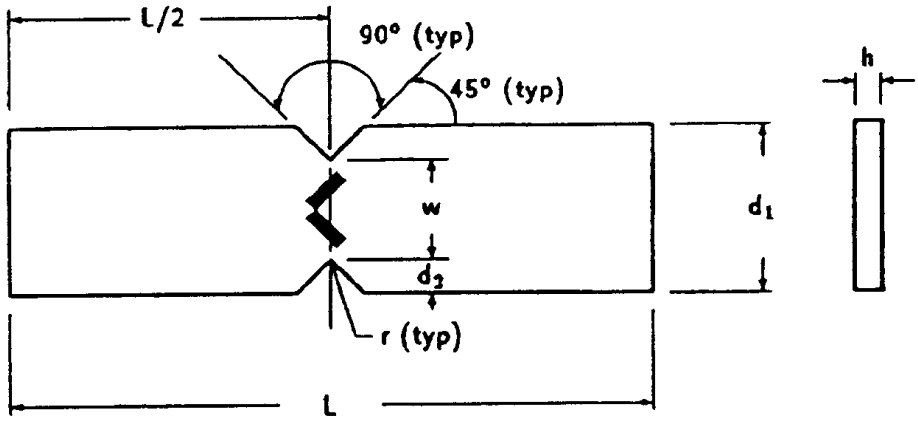
In this method, a material specimen in the form of a rectangular flat strip with symmetrical centrally located v-notches, shown schematically in Figure 6.8.4.2.2(a), is loaded in a mechanical testing machine by a special fixture, shown schematically in Figure 6.8.4.2.2(b). Either in-plane or out-of-plane shear properties may be evaluated, depending upon the orientation of the material coordinate system relative to the loading axis.

While the standard only addresses determination of properties in the material coordinate system, the initial stress-strain response of general multi-directional laminates may also be determined. However the method of load introduction into the specimen is generally not capable of reacting the much higher loads sustainable by a multi-directional laminate, and therefore for most material systems this test method is limited, for multi-directional laminates, to characterization of elastic modulus and the initial portion of the stress-strain curve. A few multi-directional materials have been successfully tested, such as the discontinuously reinforced, multi-directional, molded material commonly called sheet-molding compound (SMC), but such materials remain the exception rather than the rule.

The specimen is inserted into the fixture with the notch located along the line-of-action of loading via an alignment tool that centers the specimen in the fixture. The upper head of the fixture is attached to and driven downward by the cross-head of the testing machine, while monitoring load. The relative displacement between the two fixture halves loads the notched specimen. By placing two strain gage elements, oriented at $\pm 45^\circ$ to the loading axis, in the middle of the specimen (away from the notches) and along the loading axis, the shear response of the material can be measured.

The object of the VNB concept can be seen in the idealization of the applied loading as asymmetric flexure, shown in the shear and bending moment diagram of Figure 6.8.4.2.2(c). The specimen gage area is in the region of constant shear and zero moment. The specimen notches influence the shear strain along the loading direction, making the shear distribution more uniform than would be seen without

the notches.[1] The degree of uniformity in the shear distribution is a function of material orthotropy; the best overall in-plane shear results have been obtained on [0/90]ns-type laminates. However, while the point-loading idealization indicates constant shear loading and zero bending moment in the gage section of the specimen, in practice the fixture applies distributed loads to the specimen that contribute to an asymmetry in the shear strain distribution and to a component of normal stress which is particularly deleterious to [90]n specimens.



Nominal Specimen Dimensions		
d_1	=	20.0 mm [0.75 in.]
d_2	=	4.0 mm [0.15 in.]
h	=	as required
L	=	76.0 mm [3.0 in.]
r	=	1.3 mm [0.05 in.]
w	=	12.0 mm [0.45 in.]

FIGURE 6.8.4.2.2(a) *V-notched beam test coupon schematic.*

Limitations of the v-notched beam shear test:

Inhomogeneous Materials--- The material is assumed homogeneous with respect to the size of the test section. Materials that have relatively coarse features with respect to the test section dimensions, such as fabrics using large filament count tows (such as tows of 12000 filaments or more) or certain braided structures, should not be tested with this specimen size.

Uniformity of Strain Field--- The calculations assume a uniform shear strain state between the notches. The actual degree of uniformity varies with the level of material orthotropy and the direction of loading. A new strain gage grid configuration has recently been developed specially for use with this test method. The active grid on this gage extends from notch-to-notch and provides an improved estimation of the average strain response. When using conventional strain gages the most accurate measurements of in-plane shear modulus for unidirectional materials have been shown to result from the [0/90]ns specimen.

[1] An isotropic beam in shear has a parabolic shear stress profile.

Load Eccentricity--- Twisting of the specimen during loading can occur, affecting strength results, and especially, elastic modulus measurement. It is recommended that at least one specimen of each sample be tested with back-to-back rosettes to evaluate the degree of twist.

Determination of Failure--- Failure is not always obvious in certain materials or configurations. See the standard test method (Reference 6.8.4.2.2(a)) for more information.

Instrumentation: Strain gages are required.

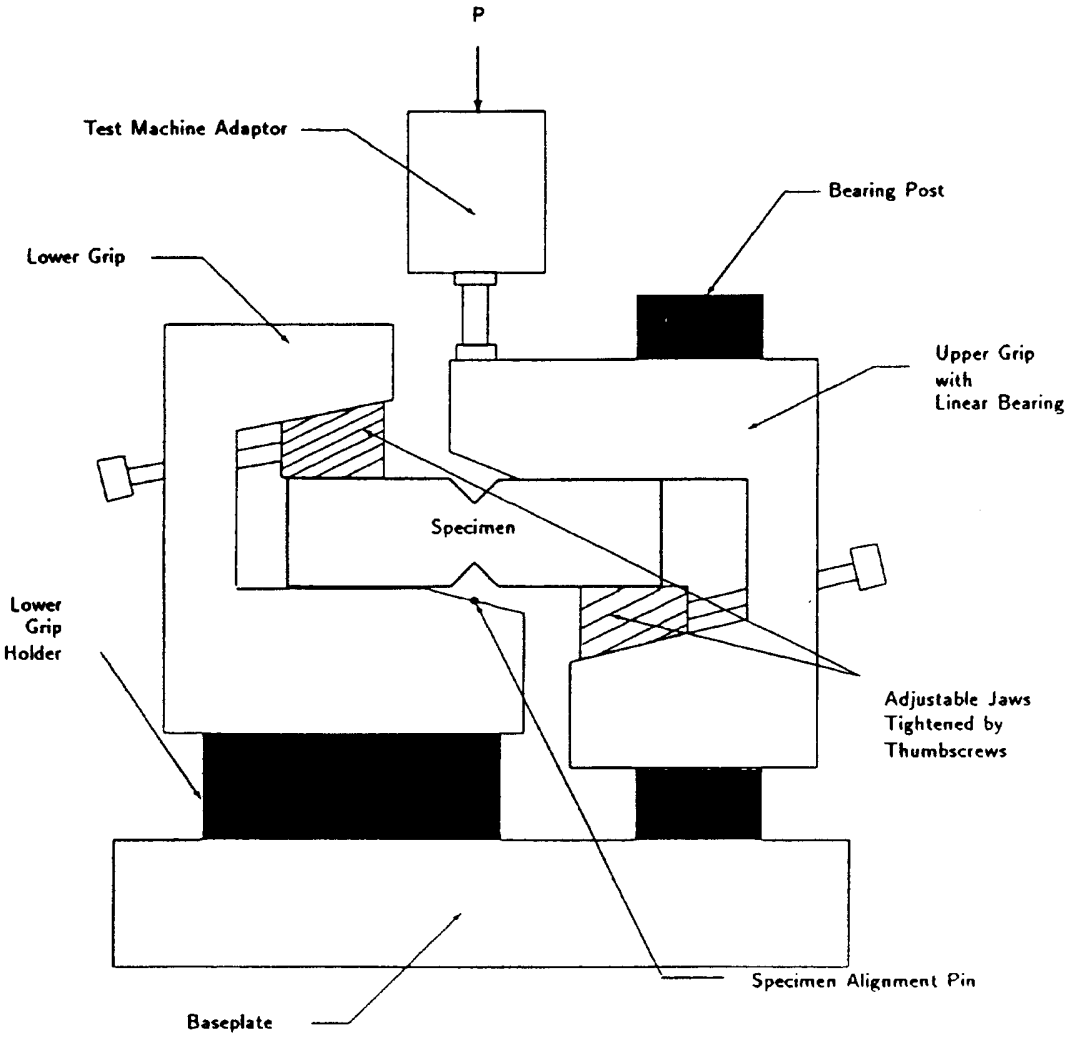
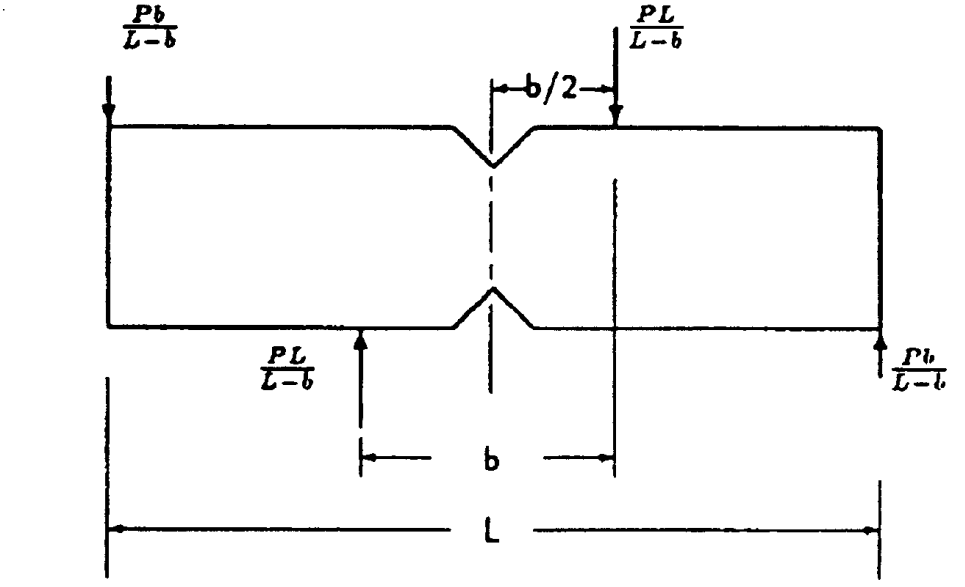
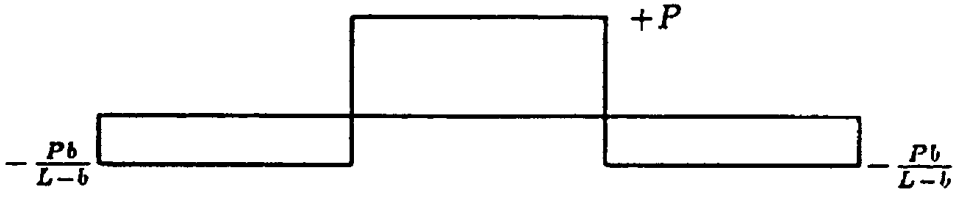


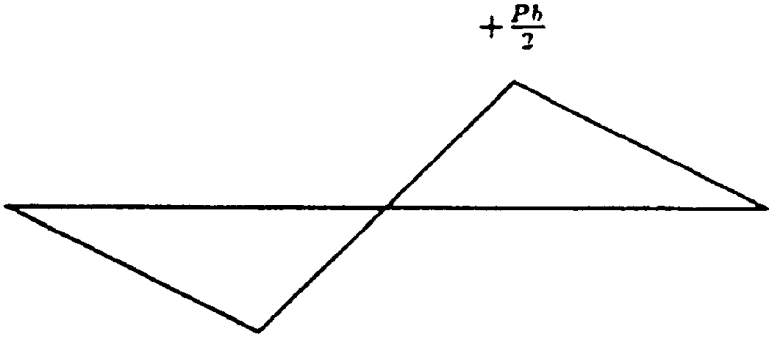
FIGURE 6.8.4.2.2(b) *V-notched beam test fixture schematic.*



Force Diagram



Shear Diagram



Moment Diagram $-\frac{Pb}{2}$

FIGURE 6.8.4.2.2(c) *Idealized force, shear, and moment diagram.*

6.8.4.2.3 Rail shear tests

ASTM D 4255-83, Guide for Testing for In-plane Shear Properties of Composite Laminates.

In 1983, ASTM Committee D-30 published D 4255 (Reference 6.8.4.2.3(a), a standard covering in-plane shear properties of composite laminates by either of two rail shear methods. The round-robin testing conducted by D-30 to understand the precision and bias of these methods found, at the time, a large amount of variability in results between laboratories. While the sources of this variability were not well understood at the time, a standard for this test method was strongly desired since rail shear tests were in wide use. The standard was completed and released, though with caveats, in order to provide a common ground for the users.

Since the initial release of D 4255, several testing factors that could have contributed to the scatter in the initial round-robin data have become better understood, and a revision to this standard is in progress at this writing (1997) that will correct several obvious shortcomings in the initial release. Whether the revised standard will have improved/reduced variability and gain the confidence of new users, particularly in the face of the substantial research that has since improved competing simpler and less expensive test methods, remains to be seen.

While the standard is restricted to in-plane testing, it is capable of testing for either material shear or multi-directional laminate shear properties. However the current version of the standard is limited, as is the D 5379 Iosipescu shear test, to characterization of modulus or initial shear stress-strain response, since the standard means of applying load to the specimen generally cannot sustain the higher strengths of multi-directional laminates. Development of standard rules for application of bonded, tapered tabs to multi-directional laminates is needed, since this approach is one of the most promising for shear strength determination of off-axis configurations. However, more work remains to be done before a bonded, tapered, tab modification to D 4255 can be standardized or recommended by MIL-HDBK-17.

As the shear stress state is not uniform through the specimen, and as failures are often noted to begin outside the center of gage section (such as at the restrained corners of the plate) this test as currently standardized does not always produce reliable shear strength data (Reference 6.8.4.2.3(b)). The three-rail test has a purer state of stress (Reference 6.8.4.2.3(c)), although it requires a larger specimen size of approximately 150 mm by 150 mm (6 in. by 6 in.)

Limitations of the D 4255-83 rail shear tests:

Specimen Size: Both version require larger specimens than other shear tests.

Instrumentation: Strain gages are required.

Stress State: The stress state is known to be non-uniform, and the failure mode is typically influenced by non-shear failures starting outside of the gage section.

Data Scatter: High data scatter from round-robin tests cast doubt upon the ability of these methods to produce repeatable data, at least in their current form.

6.8.4.2.4 Ten-degree off-axis shear test

This method, first reported by Chamis and Sinclair (Reference 6.8.4.2.4) uses a straight-sided, rectangular unidirectional tensile specimen with the fiber oriented at ten degrees to the loading direction (Figure 6.8.4.2.4). Note that the material specimen is limited to unidirectional filamentary laminates. This specimen, like the ASTM D 3518 specimen above, is also not under a state of pure shear and suffers from the effects of a combined stress state. This test produces results of generally higher modulus and significantly lower strengths than the other shear test methods such as ASTM Test Methods D 3518 or D

5379. This test method is inherently restricted to evaluation of the shear response in the 1-2 plane, and is therefore not applicable to shear evaluation of laminates.

Limitations of the 10° off-axis shear test:

Material Form: Limited to unidirectional laminates.

Stress State: Known to have a significantly biased stress state producing an overly stiff initial response and premature failure.

Lack of Standardization: Has never been standardized.

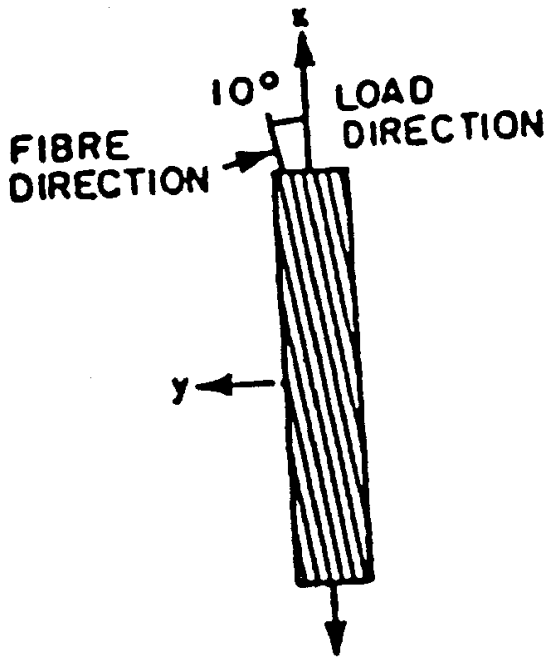


FIGURE 6.8.4.2.4 *Ten-degree off-axis tension shear test.*

6.8.4.2.5 Tube torsion tests

1. ASTM E143-87, Test Method for Shear Modulus at Room Temperature
2. MIL-STD-375, Test Method for In-Plane Shear Properties of Hoop Wound Polymer Matrix Composite Cylinders
3. ASTM D 5448/D 5448M-93, Test Method for In-Plane Shear Properties of Hoop Wound Polymer Matrix Composite Cylinders

Torsion testing of tubes has been standardized by ASTM since 1959 by Test Method E143-87 (Reference 6.8.4.2.5(a)). While broad in scope, and technically not exclusive of composites, Test Method E143 was primarily developed for metals. However the concept has also been applied to composites, where the challenge becomes in applying load to the specimen without producing a grip-induced failure; a typical gripping arrangement is shown in Figure 6.8.4.2.5. A torsion test specifically for wound composite tubes was developed and released as Military Standard, MIL-STD-375 (Reference 6.8.4.2.5(b)). MIL-STD-375 was submitted to ASTM for non-military standardization, and with minor changes was approved as ASTM D 5448/D 5448M-93. Test Method D 5448 (Reference 6.8.4.2.5(c)) consists of a 100 mm (4 in.) nominal

diameter hoop-wound tube, which is gripped at each end and twisted via a fixture until failure. This test has been shown to produce good results and is the theoretical ideal for determining both in-plane shear strength and modulus. Note that since the MIL-STD test method has since been withdrawn by the U.S. DOD it should no longer be referenced; it has been superseded by the ASTM test method.

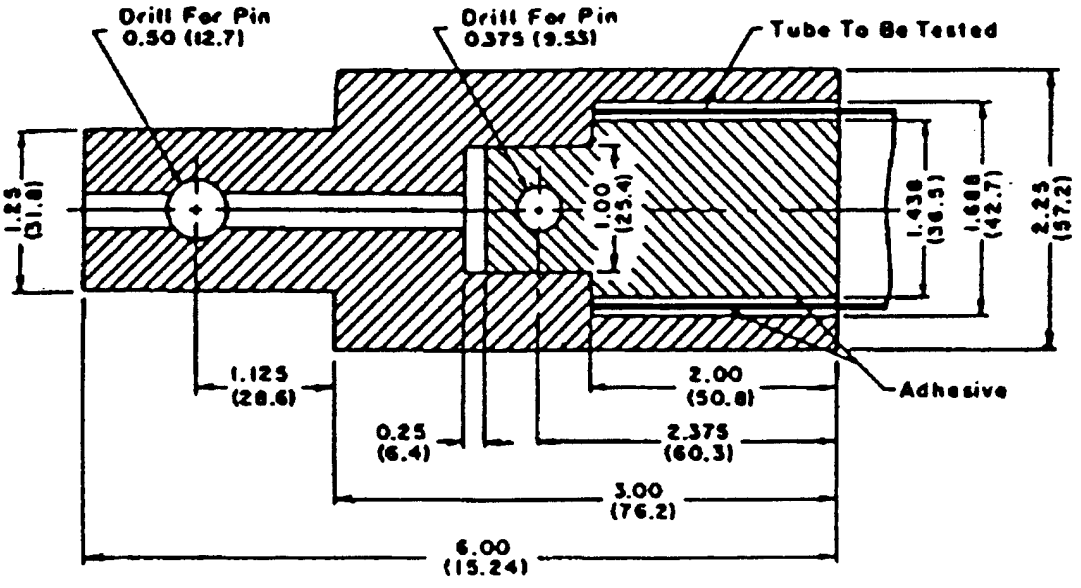


FIGURE 6.8.4.2.5 *Typical torsion tube shear fixture (Reference 6.8.4.2.5(d)).*

While not within the scope of current test standards, these tests, which are inherently restricted to in-plane usage, can be applied to laminate testing as well as lamina testing. However, gage section specimen failures may be difficult to achieve in the multi-directional laminates, due to the higher loads developed in the presence of off-axis fibers. Loading point modifications will usually be required for laminate testing.

Limitations of the torsional tube methods:

Material Form: If not using filament-wound materials the process required to create the tube may be significantly different than that used in the structure.

Cost of Specimen Fabrication: Fabrication of the specimen can be a significant undertaking requiring unusual expense.

Stress Concentration: A stress concentration exists at the end grips, as noted by Guess and Haizlip (Reference 6.8.4.2.5(d)), tending to result in failures in the gripping area, unless extreme precautions are taken.

Instrumentation: Strain gages are required.

6.8.4.3 Out-of-plane shear tests

6.8.4.3.1 Short-beam strength tests

1) ASTM D 2344-84, Test Method for Apparent Interlaminar Strength of Parallel Fiber Composites by Short-Beam Method

2) SACMA SRM 8R-94, Apparent Interlaminar Shear Strength of Oriented Fiber-Resin Composites by the Short-Beam Method

ASTM Test Method D 2344 (Reference 6.8.4.3.1(a), commonly known as the short-beam strength (SBS) test, attempts to quantify the interlaminar (out-of-plane) shear strength of parallel fiber reinforced composites.[1] The specimen for this test is a short, relatively deep beam cut from a flat laminate. The specimen is mounted as a simply supported beam and loaded at the midpoint of the span of the specimen. The intent is to minimize bending stresses while maximizing out-of-plane shear stresses by using a short, deep "beam."

However, the contact stresses induced at the load points greatly interfere with the strain distribution both through the depth of the beam and axially along the length of the beam. The resulting failure is rarely, if ever, a true pure shear failure but instead results from the complex stress state present in the specimen, as shown by Berg *et al* (Reference 6.8.4.3.1(b)) and others.

Unfortunately this test has commonly been used in the past (and is still used by some) to develop design allowables for structural design criteria. In the absence of any other choice this is understandable, though regrettable. However, the availability of the v-notched beam method, discussed in Section 6.8.4.3.2, makes the use of the short-beam strength test for property determination obsolete.

The short beam strength test should only be used for qualitative testing such as material process development and control. As a quality control test use of laminate configurations other than unidirectional are common, though currently non-standard.

The ASTM standard is currently being revised and updated to allow the standard SBS testing of balanced and symmetric laminates. A related method is SACMA SRM 8R-94 (Reference 6.8.4.3.1(c)).

Limitations of the short beam strength test include:

Stress State: The stress state is known to be significantly disruptive and three-dimensional. The resultant strengths are a poor estimation of the out-of-plane shear strength.

Failure Mode: The failure mode is most often multi-mode.

No Modulus/Material Response: Instrumentation of this specimen is not practical, therefore modulus and stress-strain data cannot be obtained.

6.8.4.3.2 Iosipescu shear test

This test method and the specimen geometry are described for in-plane shear testing in Section 6.8.4.2.2. When testing for out-of-plane shear properties the orientation of the fibers in the laminate is changed so as to cause a shearing action in the desired transverse plane. This test method is the only acceptable out-of-plane shear test available. The out-of-plane testing of laminates with fibers off-axis to the test direction, such as 3-dimensional textiles, are subject to the same restrictions and limitations that are discussed in the section on in-plane Iosipescu testing (6.8.4.2.2).

6.8.4.3.3 ASTM D 3846-79, Test Method for In-Plane Shear Strength of Reinforced Plastics

ASTM Test Method D 3846 (Reference 6.8.4.3.3), despite the title, is *not* normally used as an in-plane shear strength test (using the most common definition of in-plane in the terminology of advanced composites) but is in fact an out-of-plane shear strength test and as such is covered in this section on out-of-plane shear tests.

[1] A currently fully equivalent, but more restricted, subset of ASTM D2344 has been promulgated by the composite materials suppliers as SRM 8R-94 (Reference 6.8.4.3.1(c)). However, it is expected that, barring a parallel revision to SRM 8R-94, the two documents will diverge as a result of the on-going revision to ASTM D2344.

This test is primarily intended for use on randomly-dispersed fiber-reinforced thermosetting sheet plastics as a substitute to the short-beam strength test, Test Method D 2344 (Reference 6.8.4.3.1(a)), described in Section 6.8.4.3.1. The test consists of a doubly notched specimen loaded compressively in a supporting jig (the same fixture used in the Test Method D 695 compression test). Failure occurs in out-of-plane shear in the plane of the specimen between the two centrally located opposing square notches. While this specimen can be (and has been) used for testing continuous-fiber laminated reinforced plastics, it is not recommended for use on advanced composite laminates. The notches, which are machined into the specimen to force failure of the laminate in shear, were found by Herakovich *et al* (Reference 6.8.4.2.2(n)) to negatively influence the stress distribution in the specimen. As a result, a non-uniform, multiaxial stress state exists in the gage section, making a true strength calculation suspect at best.

Limitations of the D 3846 notched compression test:

Stress State: A highly three-dimensional, non-uniform stress state in the gage section cause strength values from this test to be unusually poor estimations of the true out-of-plane shear strength.

No Modulus/Material Response: Instrumentation of this specimen is not practical, therefore modulus and stress-strain data cannot be obtained.

6.8.4.4 Shear test methods for MIL-HDBK-17 data submittal

Data produced by the test methods in Table 6.8.4.4 are currently being accepted by MIL-HDBK-17 for consideration for inclusion in Volume 2.

TABLE 6.8.4.4 *Shear test methods for MIL-HDBK-17 data submittal.*

Property	Symbols	Fully Approved, Interim, and Screening Data	Screening Data Only
In-Plane Shear Strength (lamina)	F_{12}^{so}, F_{12}^{su}	D 3518 SRM 7 D 5379 D 5448	---
In-Plane Shear Strength (laminate)	F_{xy}^{so}, F_{xy}^{su}	---	---
In-Plane Shear Modulus (lamina)	G_{12}	D 3518 SRM 7 D 5379 D 4255 D 5448	---
In-Plane Shear Modulus (laminate)	G_{xy}	D 5379 D 4255	---
Out-of-Plane Shear Strength	F_{23}^{so}, F_{23}^{su} F_{31}^{so}, F_{31}^{su}	D 5379	---
Out-of-Plane Shear Modulus	G_{31}, G_{23} G_{xz}, G_{yz}	D 5379	---
Short Beam Strength	F_{31}^{SBS} F_{zx}^{SBS}	---	D 2344 SRM 8

6.8.5 Flexural properties

There is not a recommended test method for determining the flexural properties of composite laminates. Even though there are approved flexural test methods, there is some debate as to the validity of the results.

Within the aerospace industry, flexure testing is primarily used for quality control. ASTM Test Method D 790, "Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Materials", was originally written for plastics but has since been modified and approved for composites (Reference 6.8.5). In some cases, ASTM Test Method C 393, "Flexure Test of Flat Sandwich Constructions", has been adapted for use with composite laminates (Reference 6.8.2.2.5).

6.8.6 Fracture toughness properties

6.8.6.1 Overview

Fracture in structural solids, such as wood, glass, metals, rock and concrete, is usually initiated by some crack or notch-like flaws, which cause high stresses in the neighborhood of such flaws. Inglis (Reference 6.8.6.1(a)) pointed out the significance of the localized concentration of stress near the tip of a sharp notch. A criterion of fracture based on the first law of thermodynamics was proposed by Griffith (Reference 6.8.6.1(b)), who postulated that the reduction in strain energy due to propagation of a crack is used to create new crack surfaces. Strain energy release rate, G, is defined as the reduction in strain energy (or increase in potential energy) due to an infinitesimal self-similar extension of the crack and catastrophic propagation of the crack will occur when this rate reaches a critical value, G_c. For a through crack of length 2a in a thin or a thick plate, subjected to a tensile stress σ, the energy release rate can be expressed in terms of σ, a and the properties of the material. Irwin (Reference 6.8.6.1(c)) pointed out that in isotropic materials, three independent kinematic movements are possible, by which the upper and lower crack surfaces can displace with respect to each other. These movements are schematically depicted in Figure 6.7.8.1. Only the first mode (Mode I or opening mode) was considered by Griffith. Irwin showed that the crack tip stresses can be expressed by a three parameter set of equations. These parameters, K_I, K_{II}, K_{III}, called the Mode I, Mode II and Mode III stress intensity factors, are functions of the crack dimensions and the applied loads and critical values of these parameters govern the phenomenon of unstable crack growth. The concept of failure based on the critical value of a stress intensity factor has been shown to be equivalent to that proposed by Griffith in terms of the critical strain energy release rate (G_{Ic}, G_{IIc}, or G_{IIIc}). Irwin also suggested the use of the critical value of the total energy release rate as the parameter governing failure, provided failure occurs by self-similar crack propagation. These concepts have also been extended to orthotropic materials (Reference 6.8.6.1(d)).

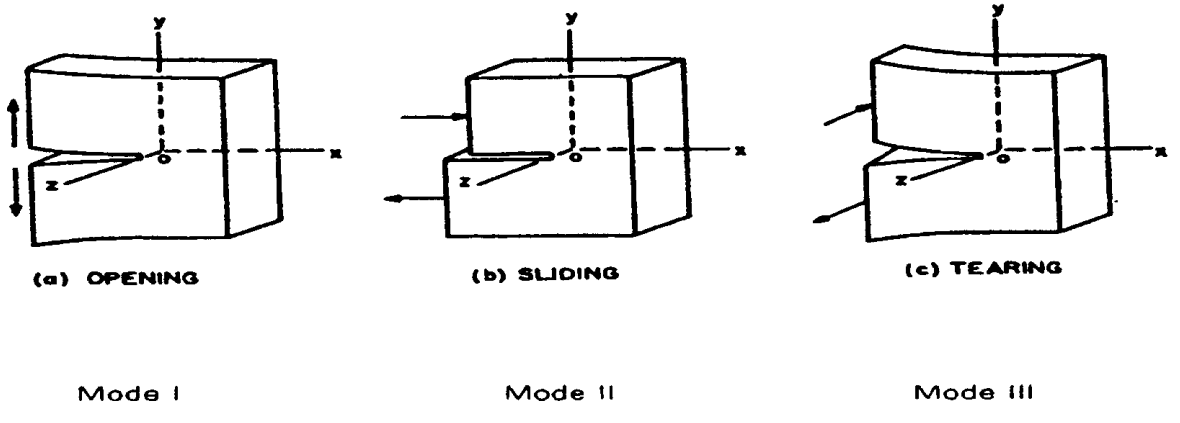


FIGURE 6.8.6.1 *Basic modes of crack extension.*

Use of fracture mechanics has gained wide acceptance in predicting failure in metal structures (References 6.8.6.1(e)-(g)) and various test methods have been developed for determining K_{Ic} (or G_{IC}) as well as crack growth resistance curves for cases when stable crack growth is possible. Further, over the years, there has emerged a fracture mechanics design procedure for fatigue of metal aeronautical structures, which is based on periodic inspection for monitoring visible cracks and predicting residual life using crack growth laws of the power law type (References 6.8.6.1(h) and (i)). Various attempts have been made to use fracture mechanics based methods for predicting failure of thin laminates with through cracks or notches (Reference 6.8.6.1(j)). However, it has been found that linear elastic fracture mechanics treating the thin laminates as orthotropic or isotropic plates is not useful because of considerable subcritical surface damage near the crack tips. Semi-empirical corrections are often employed for predicting strength of thin notched laminates (Reference 6.8.6.1(k)). On the other hand, fracture mechanics approaches appear to yield better results for thick laminates containing through cracks or deep surface flaws (References 6.8.6.1(l) through 6.8.6.1(n)) . It has also been shown that crack growth resistance or the R-curve concept, originally proposed for modeling stable crack growth (with increasing load) in metals, is useful for predicting fracture in notched chopped fiber composites (Reference 6.8.6.1(o)). Although use of fracture mechanics in the problems just described has been very limited, it is now being widely used in the industry for dealing with various problems involving delamination fracture. Delaminations (in resin rich regions between the plies in a laminate) can exist as manufacturing defects or can be created due to various reasons; namely, (i) coalescence of small voids at interfaces, (ii) foreign object impact and (iii) peculiar stress fields near discontinuities such as free edges, holes, ply drops, transverse ply cracks or bonded joints. The basic concepts of delamination fracture are the same as those discussed earlier. However, the strain energy release rate, which is the energy released due to infinitesimal extension (as described earlier for through cracks in a plate) of a delamination is commonly used for prediction of catastrophic fracture and various test methods have been proposed for determination of its critical value (often called the toughness) for each of the three modes of loading (I, II, and III as shown in Figure 6.8.6.1). Some tests have also been devised for determining the criteria of failure (mode interaction) under mixed mode conditions. The next section gives some general discussions on the test methods and use of the properties in practical applications. Subsequent sections deal with some of the test methods.

6.8.6.2 General discussion

It should be noted that although the subject is quite advanced at this point and various attempts have also been made to obtain (i) R curves for modeling stable delamination growth under increasing load and (ii) delamination growth law for predicting delamination extension under cyclic loading, only one test method has been standardized. The test specimens usually contain an implanted delamination in the form of a nonadhesive insert and have been used widely for unidirectional glass or carbon fiber reinforced composites. The tests are designed such that delamination growth direction coincides with the fiber direction. Toughness values or other characteristics may sometimes vary depending on the tendency of the delamination to wander around various phases of multiple phase matrix materials. Also, brittle matrix composites with tough adhesive interleaves may yield different properties depending on the region where the delamination propagates, i.e., interleaves, brittle matrix or the interface. Use of non-unidirectional specimens with implanted delaminations between two off-axis plies of the same orientation or between two plies of different orientations may cause the delamination to shift its path through ply cracks and interpretation of data for such cases is difficult. It is also likely that the properties will differ if the delamination propagates in a direction other than the fiber direction, even though it remains coplanar. Woven fabric composites may show more scatter as compared to that for unidirectional laminates and increasing tendency of stable delamination growth (R-curve) because of the typical structural arrangement in such materials.

The main reason for observed resistance to delamination growth (for stable growth commonly observed in Mode I tests) in unidirectional brittle matrix composites is the phenomenon of fiber bridging across the delamination plane. Such bridging is caused by fiber nesting that is typically present in unidirectional composites and, hence, the toughness value for initiation should be identified separately from those at later stages of delamination growth. This value should be representative of the toughness for a natural delamination and it is often used to obtain a conservative design criterion. R-curves obtained from tests are not commonly used to obtain generic material property data. They are, however, often used to

compare the degree of fiber matrix bonding between specimens, panels or batches of the same material or to compare composites with the same fiber, but different matrices. Poor bonding usually results in greater fiber bridging and, hence, a greater increase in G_c with delamination length.

In materials not strongly influenced by fiber bridging, a competing mechanism may dominate. When the delamination starts to grow from the insert, a pop-in type of behavior (yielding higher toughness values than that for subsequent growth) is observed. For Mode I tests, the first point is neglected in such cases and results for subsequent growth (made possible by displacement controlled tests) are utilized. For Mode II or mixed mode tests, failure is usually catastrophic and for this reason precracking (extension of the delamination beyond the insert) by Mode I loading, wedge insertion, or other methods is usually employed.

Most of the tests utilize beam type specimens with a single delamination tip. As the load is increased, load point deflections are measured and delamination growth is observed visually or using other aids or devices. In some cases, catastrophic delamination growth is observed (as in Mode II tests) and the maximum load reached is noted. Otherwise (in displacement controlled tests), load-displacement plots remain linear up to a point beyond which the delamination extends and a load drop occurs. In some materials, the onset of nonlinearity may be noticed before any delamination growth is noticeable. Such behavior may occur due to inelastic material response or subcritical damage growth ahead of the tip. The measured values of loads, deflections, and delamination lengths at the point of onset of nonlinearity or delamination growth are utilized to compute the critical energy release rates associated with fracture or subcritical damage growth. In some cases, approximate closed form expressions of energy release rates in terms of load, deflection, delamination length and/or material (or beam) stiffnesses are utilized for data reduction. When the deflection can not be measured and there is some uncertainty about the stiffness, an alternate approach is to first perform a compliance calibration on the specimen (or similar specimens) where the compliance

$$C = \frac{\delta}{P} \qquad 6.8.6.2(a)$$

δ being the deflection associated with the applied load P, for various values of delamination, length a is fitted to an exact or approximate relation (based on the principles of mechanics or a polynomial). The energy release rate is given by

$$G = -\frac{1}{b}\frac{dU}{da} \qquad 6.8.6.2(b)$$

or

$$G = \frac{1}{b}\frac{dV}{da} \qquad 6.8.6.2.(c)$$

where b is the specimen width and a the delamination length. U and V are the strain and potential energies, respectively, both expressed in terms of displacements.

Now,

$$U = \frac{1}{2}P\delta = \frac{1}{2}\frac{\delta^2}{C} \qquad 6.8.6.2.(d)$$

and

$$V = -\frac{1}{2}P\delta = -\frac{1}{2}\frac{\delta^2}{C} \qquad 6.8.6.2.(e)$$

Therefore, it follows that for displacement controlled tests (δ prescribed)

$$G = \frac{\delta^2}{2bc^2}\frac{dC}{da} = \frac{P^2}{2b}\frac{dC}{da} \qquad 6.8.6.2.(f)$$

It can be shown that the same relation holds for load controlled tests. This approach is not suitable for mixed mode tests because the individual components of the energy release rates can not be calculated in this manner, unless their contribution to the total energy release rate is known.

As discussed in the previous section, catastrophic fracture in a single mode can be predicted using the critical strain energy release rate for the particular mode provided, of course, the energy release rate

is constant along the delamination front. If these rates vary over the front, a conservative estimate can be obtained by equating the maximum value of this quantity at a point as determined from finite element or other stress analyses to the critical value (numerous investigations are reported in literature for such calculations, but they are beyond the scope of this section). Such estimates are often adequate for design purposes unless the delamination gets arrested because of structural or other constraints. A similar approach can be employed for estimating stable growth pattern and instability point using the R-curve concept. Growth of such delaminations under cyclic loading can be estimated using experimentally determined power law type growth laws, where the maximum value (or range) of the energy release rate is the controlling parameter. Uses of R-curves for predicting stable crack growth and power laws for estimating growth per cycle are, however, not yet accepted in the industry, since there exists some evidence that they do not always yield generic characterizations (Reference 6.8.6.2(a)).

The problem becomes more complicated when mixed mode conditions are present and the toughness values for the three modes differ significantly from one another. Various fracture criteria have been proposed for prediction of quasistatic fracture. A survey of such criteria for combined action of Modes I and II can be found in Reference 6.8.6.2(b). Power law type growth laws with the maximum value (or the range) of a scalar function f of the energy release rates ($f = G_I + G_{II} + G_{III}$ is a simple example) or the stress intensity factors have been suggested in different studies for modeling growth of delaminations under cyclic loading.

6.8.6.3 Mode I test methods

6.8.6.3.1 Double cantilever beam (DCB) test, ASTM D 5528
(Reference 6.8.6.3.1(a))

The test set up is schematically shown in Figure 6.8.6.3.1(a), which illustrates two types of loading attachments. The specimen is about 5 in. (125 mm) long, 0.8 - 1 in. (20-25 mm) wide and 0.12 - 0.20 in. (3-5 mm) thick. The applied load P to the two arms, the corresponding displacement δ, and typical load-displacement traces obtained are shown in Figure 6.8.6.3.1(b). The numbers on these traces indicate results for various delamination lengths and are obtained as the delamination progresses. NL indicates the onset of nonlinearity, which is usually caused by subcritical crack growth or material nonlinearities. The traces are often utilized to perform a compliance calibration. Various procedures for data reduction, other details, and restriction on specimen dimensions to avoid geometric nonlinearities are documented in References 6.8.6.3.1(a) and (b). As mentioned in the previous section, this test has been found to be adequate for unidirectional specimens. Some care should be taken when it is to be used for other lay-ups or material forms. Midplane symmetry is a requirement for pure Mode I deformation.

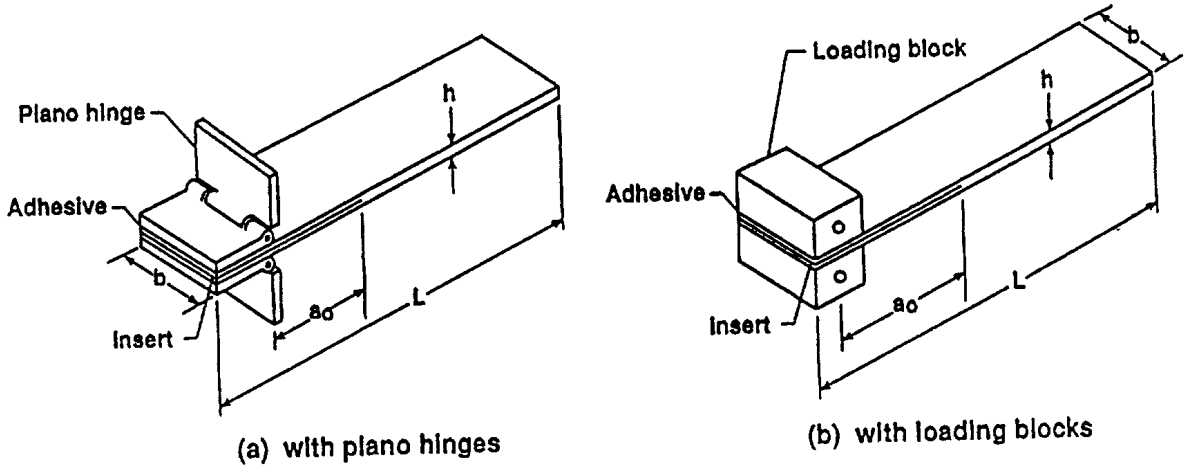


FIGURE 6.8.6.3.1(a) *Double cantilever beam specimen.*

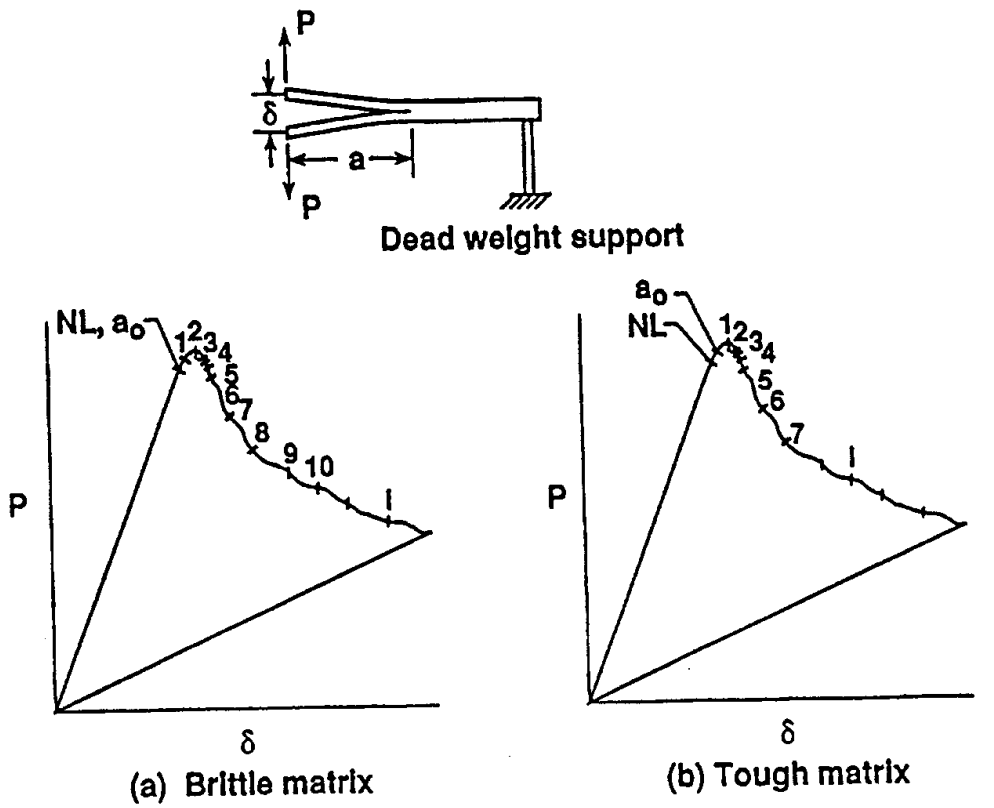


FIGURE 6.8.6.3.1(b) *Load displacement trace from DCB test.*

An asymptotic expression for the energy release rate for large a/h (a being the delamination length and h is the laminate thickness) is given by References 6.8.6.3.1(c) and (d).

$$G_I = \frac{96 P^2 (a + \alpha h)^2}{E_{11} b^2 h^3} \qquad 6.8.6.3.1(a)$$

where b is the beam width, E_{11} is the axial Young's modulus of the unidirectional composite and α is a constant which depends on the ratio of the axial shear and Young's moduli. The relation in Equation 6.8.6.2(e) can also be used when each of the beam arms are balanced midplane symmetric laminates without any bending twisting coupling if E is replaced by the equivalent flexural modulus for the arms. Substituting Equation 6.8.6.2(e) in Equation 6.8.6.2(d) and integrating with respect to a, one obtains the following equation for the compliance C for large a/h.

$$C = C_o + \frac{64 (a + \alpha h)^3}{E_{11} b h^3} \qquad 6.8.6.3.1(b)$$

where C_o is the integration constant. The constant α can be chosen as

$$\alpha \approx 1.45 \sqrt{\frac{E_{11}}{G_{13}}} \qquad 6.8.6.3.1(c)$$

where G_{13} is the through the thickness shear modulus.

It has been suggested (References 6.8.6.3.1(b) and 6.8.6.3.1(d)) that for common glass and carbon fiber reinforced composites C_o can be chosen equal to zero. Also h and the term in the denominator in the second term of Equation 6.8.6.3.1(b) or (a) should be determined by fitting measured compliances to Equation 6.8.6.3.1(b). Therefore, if a straight line is made to fit $C^{1/3}$ versus a plot (a being the abscissa

then the line when extended will cut the abscissa, x at $x = -\alpha h$) and the slope of the line gives the value of $(64/E_{11} b h^3)^{1/3}$. These values can then be substituted in Equation 6.8.6.3.1(a) to calculate G_I. It should be pointed out that in many cases αh can be neglected in comparison to length a and the results of Equations 6.8.6.3.1(a) and (b) are the same as those obtained if the two arms are treated as cantilever beams clamped at the end.

Another method, which is suggested in the ASTM standard (Reference 6.8.6.3.1(a)) for calculation of G, when both P and δ can be measured at the point of delamination propagation, is to use the formula

$$G_I = \frac{3P\delta}{2b(a+\alpha h)} \approx \frac{3P\delta}{2ba} \qquad 6.8.6.3.1(d)$$

The second expression is valid when αh can be neglected in comparison to a. This approach is called the modified beam theory method. Various other data reduction methods have been suggested in the literature (Reference 6.8.6.3.1(b)). It may be noted that if P and δ, measured at the point of onset of non-linearity, are inserted in Equation 6.8.6.3.1(d), the value of G_I yields a conservative estimate of its critical value.

6.8.6.3.2 Other mode I tests

The double cantilever beam test is the most widely used method for determining the Mode I toughness. In cases where joining loading attachments is a problem, wedge insertion between the two beams has been suggested. Another method which has been used is liquid pressure loading over a circular delamination between a thin film bonded to a substrate, which may be useful for some special applications.

6.8.6.4 Mode II test methods

6.8.6.4.1 End notched flexure (ENF) test

The ENF specimen is schematically shown in Figure 6.8.6.4.1. Typically, the specimens are 6 inches (150 mm) long, 1 inch (25 mm) wide and 0.12 - 0.20 inch (3 to 5 mm) thick. The insert is about 1 inch (25 mm) long. The span 2L of the three-point loaded beam is of the order of 4 inches (100 mm) leaving about a 1 inch (25 mm) overhang beyond the end supports. It is designed such that a delamination will propagate through the midplane of a laminate specimen loaded in three point bending (Reference 6.7.8.4.1(a)). The laminate should be symmetric about the midplane. Implanted and precracked delaminations grow in an unstable manner and, therefore, only the toughness for onset of Mode II fracture can be measured. Recently a stabilized version has been proposed (Reference 6.8.6.4.1(b)), where the test is controlled to a constant shear displacement at the delamination front. However, subcritical shear damage ahead of the front may influence the measurements after the onset.

An asymptotic expression for the energy release rate for large a/h (a being the delamination length and h is the laminate thickness) is given by (Reference 6.8.6.4.1(c)) as

$$G_{II} = \frac{9}{2}\frac{P^2(a+\alpha h)^2}{E_{11} b^2 h^3} \qquad 6.8.6.4.1(a)$$

where P is the load at midspan, b and E_{11} are the beam width and axial Young's modulus of the unidirectional composite. α is constant which can be chosen as

$$\alpha \approx 0.065\sqrt{\frac{E_{11}}{G_{13}}} \qquad 6.8.6.4.1(b)$$

G_{13} being the through the thickness shear modulus. Use of similar expressions for laminates whose top and bottom halves have no bending-twisting and shear-extension coupling, but may have bending-extension coupling have also been suggested (Reference 6.8.6.4.1(c)). Using the procedure described in Section 6.8.6.3.1 for the DCB test, the compliance C for large a/h can be shown to be given by

$$C = C_o + \frac{3(a+\alpha h)^3}{E_{11} b h^3} \qquad 6.8.6.4.1(c)$$

where C_o is an integration constant.

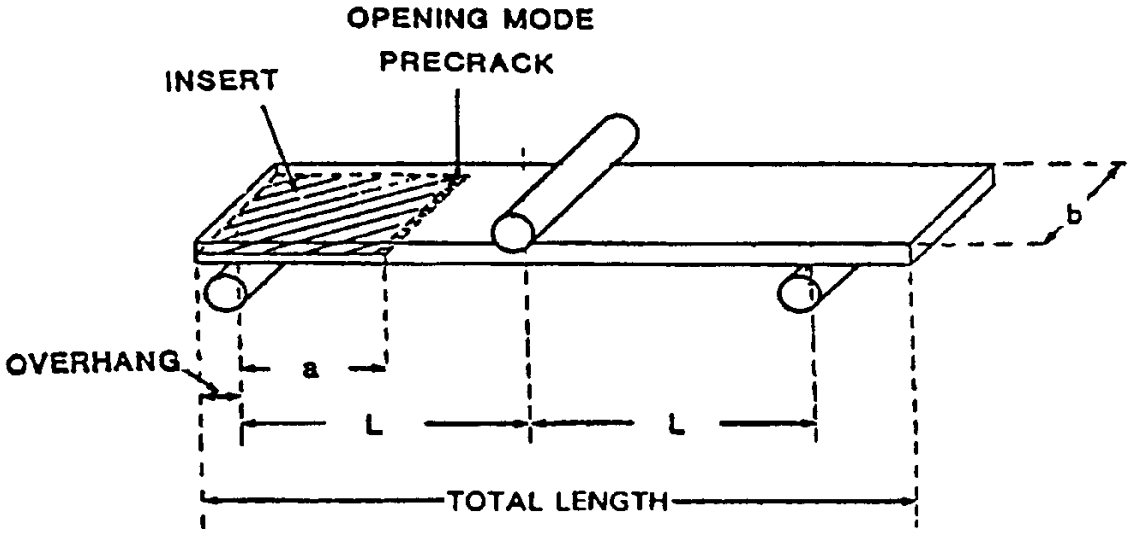


FIGURE 6.8.6.4.1 *Specimen geometry and loading.*

Use of a curve-fitting procedure to fit Equation 6.8.6.4.1(c) with estimated values of α close to that given by Equation 6.8.6.4.1(b) has been suggested in Reference 6.8.6.4.1(c). According to other investigators, C_o can be chosen as the compliance of the beam without any delaminations and fitting a straight line to $C^{1/3}$ vs. length a can, therefore, be performed to determine α and $(3/E_{11} b h^3)^{1/3}$. Compliance measurements are easily performed by taking a long beam with an implanted delamination and shifting the beam on the supports to obtain various values of a.

An ASTM "round robin" test program is planned for the ENF test and ASTM subcommittee D30.06 plans to draft a proposed standard test method based on this test program. Effects of material nonlinearity and tough adhesive interlayers on Mode II fracture have also been studied (Reference 6.8.6.4.1(c)).

6.8.6.4.2 Other mode II tests

Flexural loading of a thick laminated beam of the same form used in the ENF test, but with delaminations implanted between the support and the central load, has also been suggested (see Reference 6.8.6.4.1(c)), but compliance measurements and precracking are difficult for this specimen. However, a wider plate type specimen with implanted delaminations of circular and elliptic shapes has been found to be useful to characterize growth of such delaminations under a combined mode, the contribution from each mode varying along the delamination boundary (Reference 6.8.6.4.2).

6.8.6.5 Mode III test methods

Presently there are no commonly accepted methods for measuring Mode III toughness. A split cantilever beam has been proposed (Reference 6.8.6.5(a)) but there appears to be some Mode II contribution in this specimen (Reference 6.8.6.5(b)). A cone torsion test has been used to characterize adhesive bonds (Reference 6.8.6.5(c)), which showed that Mode III toughness can be higher than that in Mode II.

It is a common practice in composites industry to use Mode II value to estimate the criticality of Mode III fracture.

6.8.6.6 Mixed mode test methods

As discussed in Section 6.8.6.2, in natural delaminations with curved fronts, the energy release rates not only vary along the front, but mixed mode conditions are also present. Mixed mode effects are also present in delaminations with straight fronts (edge delaminations and adhesive joints are examples). In such cases, Mode I and Mode II contributions are dominant, although small Mode III effects may also be present. For this reason, various attempts have been made to characterize delamination fracture under combined action of Mode I and Mode II effects and some of them are discussed next.

6.8.6.6.1 Mixed mode specimen or crack lap shear (CLS)

The CLS specimen (Reference 6.8.6.6.1) was patterned after a similar specimen used for bonded joints. The specimen is a tension coupon, where some of the plies are terminated in the middle of the coupon (Figure 6.8.6.6.1). This specimen has been used widely in the industry. Mode I and II components are computed based on stress analyses (finite element or other methods). Effects of geometric nonlinearities have also been studied.

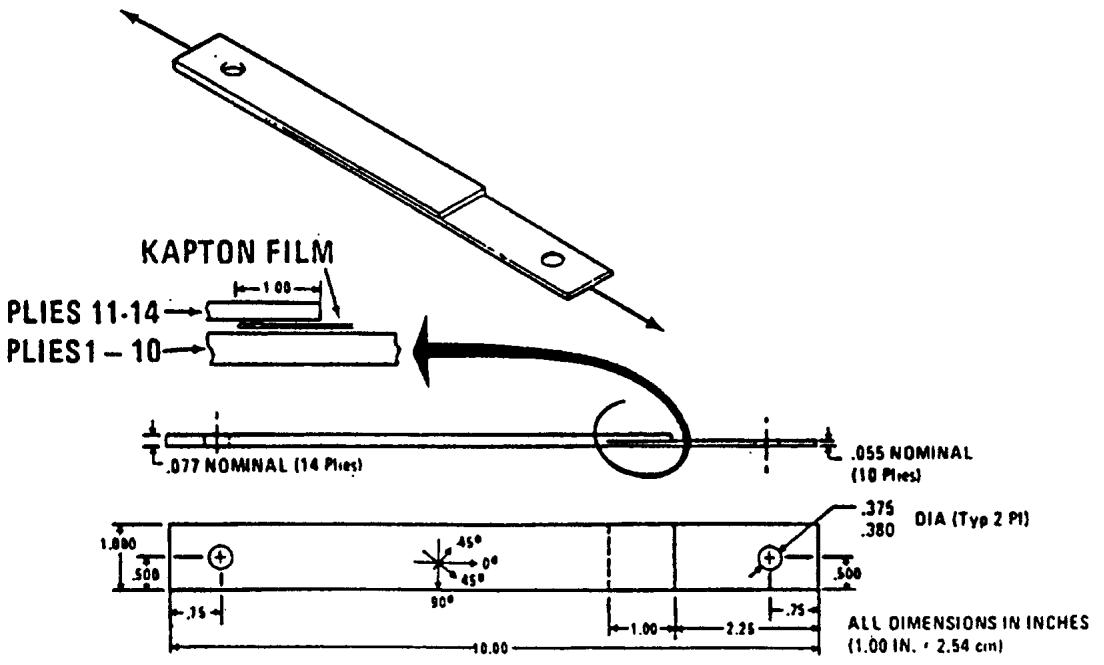


FIGURE 6.8.6.6.1 *Mixed-mode crack lap shear coupon.*

6.8.6.6.2 Mixed mode bending (MMB) test

This test has recently been proposed (References 6.8.6.6.2(a) and (b)) by combining the schemes used for DCB and ENF tests and the specimen is of the same form as that used in those tests. A special loading device is utilized, which can produce a wide range of the ratio of Mode I and Mode II components by changing the lever arm of the device shown in Figure 6.8.6.6.2. Use of the following equations has been suggested

$$G_I = \frac{3P^2}{4b^2h^3L^2E_{11}} A_1 B_1$$
$$G_{II} = \frac{9P^2}{16b^2h^3L^2E_{11}} A_2 B_2 \qquad 6.8.6.6.2(a)$$

A_1, B_1, A_2, and B_2 are expressed by the following equations when a modified linear beam theory is used for analysis

$$A_1 = a^2 + \frac{2a}{\lambda} + \frac{1}{\lambda^2} + \frac{h^2E_{11}}{10G_{13}}$$
$$A_2 = a^2 + \frac{h^2E_{11}}{5G_{13}} \qquad 6.8.6.6.2(b)$$
$$B_1 = (3C - L)^2$$
$$B_2 = (C + L)^2$$

An ASTM "round robin" test program is planned using the MMB specimen. ASTM subcommittee D3.06 plans to draft a proposed standard test method based on the results of this test program.

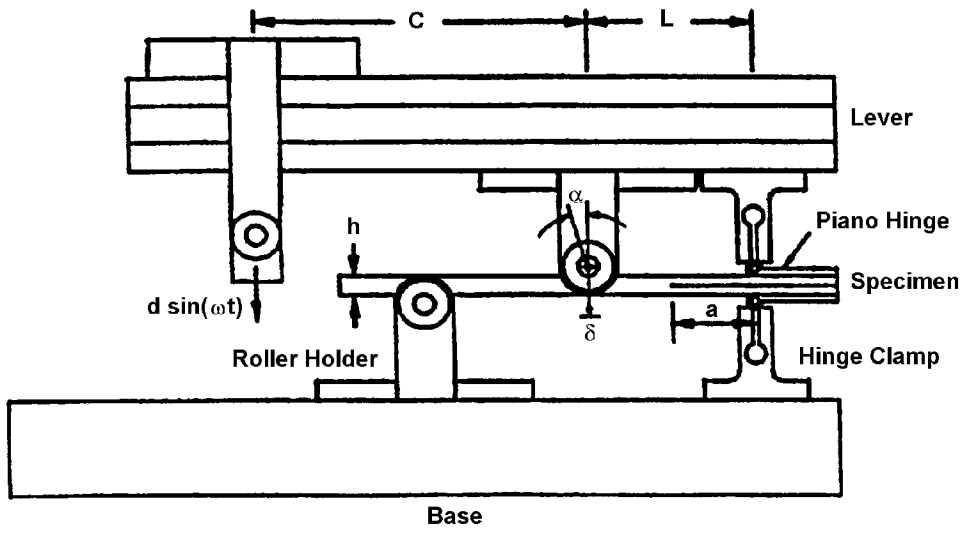


FIGURE 6.8.6.6.2 *Mixed-mode-bearing test setup.*

6.8.6.6.3 Edge delamination test

This test specimen makes use of a $(\pm\theta_2/90_2)_s$ tension specimen, in which the free edge effect causes growth of delamination from the edges (Reference 6.8.6.6.3(a)). However, the delamination growth is neither uniform nor symmetric, since it does not remain in the midplane, but oscillates vertically between the 90/-θ interfaces. A modified version of the specimen with a starter delamination has been proposed (Reference 6.8.6.6.3(b)). Data reduction procedures are discussed in References 6.8.6.6.3(a) and (b). The test has not gained wide acceptance for toughness property determination.

6.8.6.7 Fracture toughness tests for MIL-HDBK-17 data submittal

Data produced by the following test methods (Table 6.8.6.7) are currently being accepted by MIL-HDBK-17 for consideration for inclusion in Volume 2.

TABLE 6.8.6.7 *Fracture toughness test methods for MIL-HDBK-17 data submittal.*

Property	Symbols	Fully Approved, Interim and Screening Data	Screening Data Only
Mode I Toughness	G_{IC}	ASTM D 5528	--
Mode II Toughness	G_{IIC}	ENF	--
Mode III Toughness	G_{IIIC}	--	--
Mixed Mode I, II Fracture	f_c (G_I, G_{II})	--	MMB

6.9 UNIAXIAL FATIGUE TESTING

Static testing of unidirectional composite specimens is useful for material characterization, comparison of materials, and for prediction of application laminate properties through the use of lamination plate theory. In the area of fatigue, however, no generalized methodology has yet been devised to predict laminate behavior from unidirectional specimen data. Hence, the development of fatigue design values becomes a unique problem for each application lay-up. Many studies have been undertaken, and much has been written concerning life prediction for specific laminates under cyclic loading spectra. Even at this level, empirical methods have been favored due to the inadequacy of results obtained from cumulative damage models, fracture mechanics analyses, and other theoretical approaches (References 6.9(a) and (b)).

ASTM Test Method D 3479, "Tension - Tension Fatigue of Oriented Fiber, Resin Matrix Composites", is a generalized coupon testing method (Reference 6.9(c)). However, because composite fatigue is so application dependent, it is important that the laminates represent the application and that the laminates testing account for the service load spectra and environmental conditions. Currently this is accomplished in composite hardware programs through a "building block" test approach involving coupon, element, and component specimens, all representative of full-scale structural details.

It is important to note that, for the majority of current aircraft composite structure, fatigue capability does not become a limiting factor if all static strength concerns have been thoroughly and successfully addressed. Exceptions to this are high-cycle components such as those found in helicopter dynamic systems.

6.10 MULTIAXIAL MECHANICAL PROPERTY TESTING

Multiaxial tests, including biaxial and triaxial loadings, can be performed to experimentally evaluate the effect of combined stress states on composite material response. No standard test methods exist to

guide multiaxial testing, and little data is available. A discussion of multiaxial testing can be found in Volume 3, Section 10.2.3.2.

6.11 VISCOELASTIC PROPERTIES TESTS

6.11.1 Introduction

The time dependence of the properties of organic matrix composites arises primarily from the viscoelastic nature of the polymeric matrix resins, which these materials contain. Although these properties are matrix-dependent, they cannot be simply predicted based upon the viscoelastic properties of the unreinforced matrix. Creep compliance, relaxation modulus and even measured glass transition temperature can vary widely as a function of the content and orientation of reinforcing fibers.

6.11.2 Creep and stress relaxation

Creep is the time-dependent strain exhibited by a material under the action of a constant stress. Creep is characterized as a function of time by measurement of the creep compliance which is determined by dividing the time-dependent strain by the level of constant stress. Similarly, stress relaxation is the time-dependent stress exhibited by a material under the action of a constant strain. The relaxation modulus is determined by dividing the time-dependent stress by the constant applied strain. Creep and stress relaxation are different manifestations of the same underlying mechanisms of molecular mobility. At low levels of applied stress or strain these time-dependent effects may be completely recoverable when the forcing function is removed, but at higher levels irrecoverable deformations may occur under load. Irrecoverable strain, sometimes called permanent set, may be accompanied by time-dependent damage development such as the formation and growth of transverse matrix cracking.

Viscoelastic effects should be considered if the end use involves high stresses in a matrix-dominated direction, high temperature or exposure to a harsh chemical environment. Composite structural designs should be evaluated for potential time-dependent effects if the working load involves significant shear loading. Since high shear loads can be generated near a structural discontinuity, these are areas of potential concern. It should be noted that viscoelastic effects can be beneficial in some of these instances, since stress relaxation in high stress regions can help prevent catastrophic failure. When a thermoplastic matrix is employed, time-dependent behavior may be a problem, especially if the service temperature is at or near T_g. The extent of creep should be smaller in thermoset composites due to cross-linking.

In fiber-reinforced plastics (composites), one can assume that creep will be more important when the composite is loaded in a matrix-dominated manner than in a fiber-dominated manner. For instance, the creep of a unidirectional specimen tensile loaded in the fiber direction is expected to be small and hence only of secondary importance. However, loading a specimen in a matrix-dominated manner is not as straight forward as one would expect. Testing a unidirectional specimen in a transverse tensile manner one would think must essentially load the matrix, and this is not so. There are several explanations. One of the explanations is that loading a transverse specimen puts the matrix in a bidirectional state of stress (tensile) because the fibers prevent the matrix from laterally contracting (i.e., Poisson's effect) and thus the amount of creep response is restricted. Another argument for low creep response in transverse specimens is that the specimens are weak and the strains are small, so the change in strain would also be small. Another way to load the matrix is in shear where the creep response should be large. The most convenient way to load the matrix in shear is to load a [$\pm$45] specimen in tension. Although there is some argument that this test does not produce pure intralaminar shear, it at least produces some shearing and can be thought analogous to loading a unidirectional laminate in shear.

Experience has shown that the resulting creep is significant. Other loadings that would be interesting to examine with respect to creep response would be compression of unidirectional specimens in the fiber direction and three-point bend loading of unidirectional specimens (in both of these methods shear plays a role).

A common experimental procedure is to apply a dead weight tensile load to a [±45] specimen at 5, 10, or 15 ksi (35 MPa, 70 MPa, or 105 MPa) and monitor the strain as a function of time. The strain reading at the first application of the full load is designated as the strain at zero time. Subsequent measurements are timed from that zero time reading. Readings are taken at 1, 2, 3, 10, 20, 30, 60, 100, and 200 minutes, and then as convenience dictates. Strain as a function of time is plotted on semi-log axes and the test continued for at least 30,000 minutes (or 3 weeks). Testing should be done at controlled (constant) humidity and temperature conditions (References 6.11.2(a) and (b)). Generally, specimens are 1 in. wide, 6 in. long, and ~0.04 – 0.06 in. thick (25 mm wide, 150 mm long and ~1-1.5 mm thick). These dimensions are open to question; there is some evidence that wider samples will creep less than narrow samples.

6.12 FORM-SPECIFIC MECHANICAL PROPERTY TESTS

6.12.1 Tests unique to filament winding

6.12.1.1 Overview

The mechanical behavior of filament wound structures is typically different from the behavior of flat laminated structures. Some noted differences result from the type of cure, resin void content, microcracking, and free edge construction. However, filament wound structures require the same mechanical property data for design and analysis as used for general laminated structures. The majority of filament wound structures are used in the rocket motorcase community, and consequently, most of the test specimens are in the form of cylinders or bottles that more closely simulate the geometry of the structures to be designed and analyzed.

6.12.1.2 History

In November 1983, the Joint Army, Navy, NASA, and Air Force (JANNAF) Interagency Propulsion Committee chartered by the Department of Defense formed the Composite Motorcase Subcommittee (CMCS) (Reference 6.12.1.2.(a)). The CMCS was concerned with the application of composite materials in the construction of rocket motorcases for strategic and tactical missiles, space propulsion systems, and cartridge cases for gun propulsion. The CMCS consisted of four working panels two of which were the Testing and Inspection (T&I) panel and the Design and Analysis (D&A) panel.

The T&I panel surveyed industry on test methods which resulted in seventeen different tension tests, seventeen different compression tests and sixteen different shear tests that were being used to obtain mechanical property data. The T&I and D&A panels joined to evaluate the test methods via a JANNAF Workshop (Reference 6.12.1.2(b)). A panel of experts in filament wound composites was selected and tasked to make recommendations for test methods. A joint T&I and D&A Workshop was held in April 1986 to discuss the panel of experts' recommendations and to have an industry selection of JANNAF interim test standards to be used for the determination of uniaxial material properties.

The CMCS conducted a Design Round Robin (DRR) and a Testing Round Robin (TRR) for three of the interim tests: 1) Transverse Tension, 2) Transverse Compression and 3) In-plane Shear of ninety degree filament wound cylindrical specimens. The participants in the DRR and the TRR were paid and were determined through competitive procurement. The manufacturing of the ninety degree filament wound cylinders and strain gaging were also determined through competitive procurement. Each of the test specimens were ultrasonically C-scanned for any anomalies. The TRR was conducted in accordance with ASTM E 691 (Reference 6.12.1.2(c)). The DRR and TRR were successful and resulted in three Military Standards in the fall of 1992. The three Military Specifications were put into ASTM format, run through the balloting phases and approved as ASTM test methods in the fall of 1993. The JANNAF efforts were coordinated with MIL-HDBK-17, ASTM Committee D-30, SACMA, and the DOD Standardization Program for Composites Technology.

6.12.1.3 Tension tests for uniaxial material properties

6.12.1.3.1 Zero degree tension

The test method selected for the zero degree tension test is ASTM D 3039 entitled "Standard Test Method for Tensile Properties of Fiber-Resin Composites" (Reference 6.8.2.2.1(a)). It is recommended that the test specimens be obtained from a filament wound laminate. The JANNAF CMCS initially voted on either the pressurized NOL ring or a pressurized ninety degree filament wound tube. There were several attempts to obtain valid data from each technique but with little repeatable success.

6.12.1.3.2 Transverse tension

The test method selected to determine the uniaxial material properties for transverse tension is ASTM D 5450 entitled "Test Method for Transverse Tensile Properties of Hoop Wound Polymer Matrix Composite Cylinders" (Reference 6.12.1.3.2). This test method was approved as MIL-STD-373 entitled "Transverse Tensile Properties of Unidirectional Fiber/Resin Composite Cylinders" in the fall of 1992, and was subsequently approved as an ASTM test method in the fall of 1993.

6.12.1.4 Compression tests for uniaxial material properties

6.12.1.4.1 Zero degree compression

The test method selected to determine zero degree uniaxial material properties is ASTM D 3410 entitled "Test Method for Compressive Properties of Unidirectional or Crossply Fiber-Resin Composites" (Reference 6.8.3.1(a)). Method B, also known as the IITRI method, is recommended. It is further recommended that the test specimens be obtained from a filament wound laminate.

6.12.1.4.2 Transverse compression

The test method selected to determine the uniaxial material properties for transverse compression is ASTM D 5449 entitled "Test Method for Transverse Compressive Properties of Hoop Wound Polymer Matrix Composite Cylinders" (Reference 6.12.1.4.2). This test method was approved as MIL-STD-374 entitled "Transverse Compressive Properties of Unidirectional Fiber/Resin Composite Cylinders" in the fall of 1992, and was subsequently approved as an ASTM test method in the fall of 1993.

6.12.1.5 Shear tests for uniaxial material properties

6.12.1.5.1 In-plane shear

The test method selected for the determination of in-plane shear properties is a ninety degree, four inch diameter filament wound torsion tube described in ASTM D 5448 entitled "Test Method for In-plane Shear Properties of Hoop Wound Polymer Matrix Composite Cylinders" (Reference 6.12.1.5.1). This test method was approved as MIL-STD-375 entitled "In-plane Shear Properties of Unidirectional Fiber/Resin Composite Cylinders" in the fall of 1992, and was subsequently approved as an ASTM test method in the fall of 1993.

6.12.1.5.2 Transverse shear

The test method selected to determine transverse shear material properties is ASTM D 5379 entitled "Test Method for Shear Properties of Composite Materials by the V-notched Beam Method" (Reference 6.8.4.2.2(a).

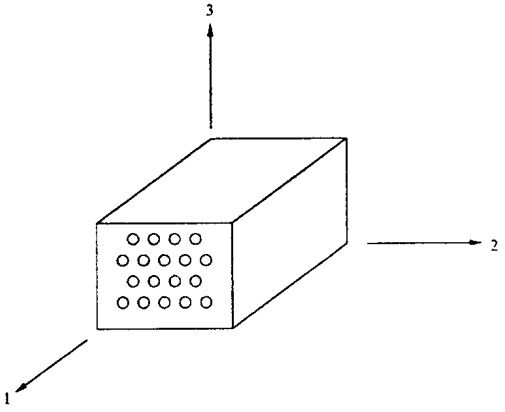
6.12.1.6 Test methods for MIL-HDBK-17 data submittal

Data produced by the following test methods (Table 6.12.1.6) are currently being accepted by MIL-HDBK-17 for consideration for inclusion in Volume 2 (an element of orientation is shown for clarity).

TABLE 6.12.1.6 *Filament wound test methods for MIL-HDBK-17 data submittal.*

Property	Symbols	Fully Approved, Interim, and Screening Data	Screening Data Only
0° Tension*	F_1^{tu} , E_1^t, ν_{12}^t , ε_1^{tu}	ASTM D 3039	---
90° Tension*	F_2^{tu} , E_2^t, ν_{21}^t , ε_2^{tu}	ASTM D 5450	---
0° Compression*	F_1^{cu} , E_1^c, ν_{12}^c , ε_1^{cu}	ASTM D 3410B	---
90° Compression*	F_2^{cu} , E_2^c, ν_{21}^c , ε_2^{cu}	ASTM D 5449	---
In-Plane Shear**	F_{12}^{su} , G_{12} , γ_{12}	ASTM D 5448	---
Transverse Shear***	F_{23}^{isu} G_{23} γ_{23} F_{31}^{isu} G_{31} γ_{31}	ASTM D 5379	---

* Strength, modulus, Poisson's Ratio, and strain
** Strength, modulus, and strain



6.12.2 Tests unique to textiles composites

6.12.2.1 Overview

The physical and mechanical behavior of common textile composites is in many ways similar to that of the unidirectional materials. Many of the same test methods work in the determination of properties of the textile composite. This is only applicable to weaves that are homogenous in nature over a small repeat length and width. A designer must analyze very large coarse weaves for specific situations that repeat over several inches on an individual basis.

In two-dimensional weaves, typical testing concerns are associated with specimen size that defines the representative structure. In most commercially available fabrics, the repeat is over a small square. The use of very coarse or odd weave patterns complicates the simplicity of testing allowed by using unidi-

rectional laminate tests. In these large or irregular patterns, it is required to account for repeat sequence in specimen size selection. Coordinating this within the appropriate test method limitations is sometimes difficult.

In testing of textile composites, a major factor to consider is z-axis reinforcing. Cross-stitching of two dimensional textile composites or interconnection of all axes in 3D weaves accomplishes this reinforcement. The cross-stitching and weaving applications introduce reinforcement in the Z-axis to provide better properties in the out-of-plane direction. In these textile products, testing must address the characterization of the third direction. Although a number of programs are investigating the characterization in the z-direction there is no agreed standard as with the in-plane methods.

For textile processes, the most likely manufacturing defects are associated with porosity and with “dry” regions from insufficient resin flow. Large planes of weak bonds are unlikely. Conventional ultrasonic techniques can detect large areas of porosity. Small areas masked by the inhomogeneous structure of 3D textiles are possible. The dry regions tend to be on the surface and visually detectable.

There is the possibility of microcracks in the resin rich areas near the tow intersections even in toughened systems. The 3D nature of the materials does not allow for contraction in the Z direction as do 2D composites so you put a significant 3D stress state on the resin rich areas, hence the potential for cracks forming.

6.12.2.2 Background

The testing of textile composites is not a new topic. In fact, most of the first composite materials were reinforced with woven materials. There are numerous reviews of the test methods for unidirectional materials, but typically the woven materials are as often part of a design as the unidirectional materials. As discussed previously, test methods for textile composites must account for the pattern associated with woven textiles to be representative of woven structures.

In typical woven materials, the repeat pattern is less than one tenth of an inch. In these cases testing per the standard unidirectional methods yields values that are representative of the bulk properties of the weave. In most instances since the repeating pattern is small, normal testing specimens will accommodate the weaving. When using a standard test method developed for a unidirectional laminate, compare the repeat size to the maximum and typical specimen sizes per the standard test method. If the repeat pattern is 10X smaller than the specimen test length, no problem should result.

For woven textile fabrics, the pattern is part of the style designation. In glass fabrics, the weavers number also assigns yarn properties, refer to Reference 6.12.2.2(a) for definition of style numbers of glass fabric.

In two-dimensional and triaxial braiding this is also true, but the weave comes in the form of a sock. The reinforcement properties of the sock are created by the tows that are used, the mandrel feed speed and the diameter and shape of the mandrel. The specimens from a braiding operation require care to assure the weaving pattern is not changed during the lay-up and impregnation phase of test coupon fabrication.

Tests conducted to evaluate the test specimens used for dry textile preforms and RTM process composites are included in References 6.12.2.2(b) and (c). Section 6.12.2.4 presents discussion of the peculiarities of complex braiding.

6.12.2.3 Fabric and two-dimensional weaves

6.12.2.3.1 Physical property tests

For measuring density and fiber volume fraction of dry composites, the sample should be an order of magnitude larger than the unit cell size to obtain an average density. For textile composites with average

fiber volume fractions of 0.50 - 0.55, the fiber volume fraction within the yarn can be as large as 0.7. Thus, use the actual fiber volume fraction in analyses that model the yarns and resin discretely, not average fiber volume fraction for the textile composite.

Composites made with dry two-dimensional textile preforms used in RTM processes are quasi-laminar in nature. Thus, a cured ply thickness could be calculated and used for scaling strength with thickness. For molded parts, the tool volume controls the thickness. The bulk factor (unrestrained thickness/restrained thickness) for a dry preform should be slightly greater than 1:1 to obtain the desired fiber volume fraction. For bulk factors significantly greater than one, autoclave or press pressures may be required to close the tool, resulting in unacceptable distortion of the preform. For bulk factors less than one, the fiber volume fraction will obviously be less than optimum.

6.12.2.3.2 Mechanical testing

The standard test methods found in the static uniaxial mechanical test section (6.8) provide good results for textile composites made from two-dimensional woven prepreg. However, any testing must take into account the scale affects associated with the use of textile reinforcements. The repeat size of the weave dominates this scale affect. If the test specimen size is small enough to be unbalanced, the results will be unrepresentative of the characterized parts.

As the complexity of the repeat pattern increases, so does the need for larger test specimens. No specific rule of thumb for this is available and the investigator should evaluate this as part of the program to establish mechanical values. There are many studies that the reader is encouraged to review as part of the characterization of the particular weave.

6.12.2.3.3 Impact considerations

The present test methods to measure impact damage tolerance are just as suitable for composite specimens made from dry textile preforms and a RTM process as composites made from prepreg laminates. One must understand impact response to apply the data. A discussion of impact response is in Reference 6.12.2.3.3.

6.12.2.4 Complex braiding considerations

The goal of testing braided specimens is to produce mechanical property data that mimics the performance of that section of the braided structure in a representative manner. Often this is difficult to achieve due to the local contour or details not accurately formed into the test specimens. Therefore, certain assumptions as to the applicability of the specimens to the final part are necessary before the start of testing.

Most specimens assume pristine manufacturing in producing the specimen and this is representative of the final part. This initial coupon may or may not mimic the final product depending upon care and reproducibility in the design of the part. Test specimens for tension, compression, shear, pin bearing, interlaminar shear and interlaminar tension are described in other sections of this handbook. In some cases, the actual shape of the zone may more accurately represent specimen geometry, i.e., a tapered specimen (preferably symmetric) that contains the essential geometry of the actual tapered braid.

In general, the specific mechanical properties of interest are torsional stiffness and strength, shear stiffness and strength, tensile, compressive and flexural moduli and strengths, bearing strength for bolted assemblies, and adhesive bond strength where required. In the event exposure of the braided parts to damage or degradation by the environment is possible, a series of tests to provide a measure of the damage tolerance and environmental effects on braided materials must be included. Damage criteria must include manufacturing defects that are not detectable by non-destructive evaluations.

Several candidate configurations for these types of tests are listed in NASA CR 1092 (Reference 6.12.2.4) and should be considered as part of a design allowables program based upon intended use and environment.

Fatigue and creep tests of braided composites are important considerations whenever the part is a dynamic component. The highly complex courses followed by the braided yarns through the matrix create fiber load paths for which stiffnesses and strengths are difficult to predict. The chance that the matrix is loaded beyond the level predicted by laminated design is high. Therefore, a lower confidence in understanding how a braided part behaves will require a rigorous fatigue and creep test plan to characterize the complex interweave of the braided yarns in the part. Tests designed to determine fatigue or creep effects are part specific and should be conducted on a braided specimen that is identical to the finished product. Scaling effects and the use of similitude in test specimen design should be avoided whenever possible. Otherwise, the results may not mimic a critical defect, which results in a failure of the part before the predicted value.

In general, it is accurate to state that for both two- and three-dimensional braided composites, defects are the single most important factor in the design of the part and the corresponding application of the allowables. This is due to the nature of braided preforms as well as physical limitations in the ability of the yarns to cover an area of rapid change in contour. The good news is that net-shape braided parts do provide a means to build these parts without intensive hand labor. This makes braiding suitable to production environments and fully capable of doing the intended job. This is true when the designer or analyst has made the correct choices in the type of specimens used for determining braided allowables.

6.12.2.4.1 Three-dimensional weave and braids

Three-dimensional textile composites are typically very complex application driven weavings. The testing associated with them determines the specific properties addressed by special weavings or cross-stitching of two-dimensional weaves. Some conventional tests performed on representative sections of the weave determine and characterize the capabilities of these processes. Currently there are many investigations under way to standardize tests in the z-axis but no industry standard has yet been set.

6.12.2.4.2 Through the thickness test methods

Many methods have been developed to characterize the through the thickness properties. Interlaminar test methods are desirable for optimizing the type and amount of through the thickness reinforcement in textile composites; Table 6.12.2.4.2 shows commonly used methods. These test methods must have additional work before recommending them for standardization. None of the in-plane shear test methods was totally acceptable. A review of those investigations is in Reference 6.12.2.3.3.

6.12.2.5 Test methods for submission to MIL-HDBK-17

In general, the methods described in the preceding sections on unidirectional materials should be used to characterize textile composites. These are only applicable to weaves that are homogenous in nature over a small repeat length and width. Very large coarse weave repeats developed for specific situations are beyond the scope of this section and the handbook. Analyze these weaves for testing on an individual basis.

For appropriate test methods for individual test conditions see the following sections:

Tension test methods	6.8.2.4
Compression test methods	6.8.3.4
Shear test methods	6.8.4.4
Fracture toughness tests	6.8.6.7

6.12.3 Tests unique to thick-section composites

No standard test methods exist to guide thick-section testing, and little data is available. Mechanical tests, including uniaxial, biaxial, and triaxial loadings, can be performed to experimentally evaluate the effect of combined stress states on composite material response. A discussion of thick-section testing can be found in Volume 3, Section 7.2.3.

TABLE 6.12.2.4.2 *Proposed in-plane shear tests methods for 3D reinforced composites.*

Test method type	Test Method Specification	Test Method Title	Comments
Shear	ASTM E 143 ASTM D 4255 None	Standard Test Method for Shear Modulus at Room Temperature Standard Guide for Testing In-plane Shear Properties of Composite Laminates Compact Shear	Reference 6.12.2.4.2
Interlaminar tension	ASTM D 6415	Standard Test Method for Measuring the Curved Beam Strength of a Fiber-Reinforced Polymer-Matrix Composite	
Interlaminar fracture toughness	ASTM D 5528	Standard Test Method for Mode I Interlaminar Fracture Toughness of Unidirectional Fiber-Reinforced Polymer Matrix Composites	Reasonable for 2-D braids and stitched uniweaves
Interlaminar fracture toughness	See 6.8.6.4.1	End Notched Flexure (Mode II)	Reasonable for 2-D braids
Interlaminar tension	ASTM C 297	Standard Test Method for Flatwise Tensile Strength of Sandwich Constructions	Reasonable for elastic constants
Interlaminar compression	ASTM D 3410, Procedure B	Standard Test Method for Compressive Properties of Polymer Matrix Composite Materials with Unsupported Gage Section by Shear Loading	Reasonable for elastic constants and strength
Interlaminar shear	None ASTM D 3846	Compact Standard Test Method for In-Plane Shear Strength of Reinforced Plastics	Reference 6.12.2.4.2, Thick composites Thin composites
Interlaminar shear - transverse	ASTM D 2344	Standard Test Method for Short-Beam Strength of Polymer Matrix Composite Materials and Their Laminates	Reasonable for 2-D braids and 3-D weaves

6.13 SPACE ENVIRONMENTAL EFFECTS ON MATERIAL PROPERTIES

This section is reserved for future use.

6.13.1 Introduction

This section is reserved for future use.

6.13.2 Atomic oxygen

This section is reserved for future use.

6.13.3 Micrometeoroid Debris

This section is reserved for future use.

6.13.4 Ultraviolet radiation

This section is reserved for future use.

6.13.5 Charged particles

This section is reserved for future use.

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