1

Introduction

Most of the subject matter in this text has been taught for years to advanced undergraduate and first-year graduate students at the University of Delaware, Florida Atlantic University, and the University of Wyoming. During this time, the authors realized there was no textbook offering a concise treatment of the experimental characterization of composite materials. Most current textbooks deal only with the analysis of composite materials. If the present text appears to emphasize advanced composite materials, it is only because these materials often present the greatest challenges to experimental characterization. These also are the materials most often used in structural applications, where accurate characterization is most important. Interestingly, today, many high-performance designs demand the use of advanced composite materials.

The objective of this textbook is to present processing techniques, specimen preparation, analyses of test methods, test procedures, and data reduction schemes to determine mechanical properties, thermal expansion coefficients, and fracture and strength data for composite materials. Emphasis is placed on practical matters such as preparation and testing of specimens and data reduction methodology. Many of the test methods presented are American Society for Testing and Materials (ASTM) or other national or international standards. Others, although originating within an individual organization and sometimes continuing to be refined in terms of test specimen and fixture geometries, test procedures, and data reduction schemes, are being widely used within the composites testing community.

No attempt is made to present a detailed review of composite mechanics or fracture mechanics. Such a treatment has been presented in many other textbooks to which references are made. Only a brief elementary outline of the theoretical background is provided in Chapter 2. Moreover, no attempt is made to present an overview of all test methods; such reviews are available elsewhere, as will be referenced. The methods presented here are deemed the most appropriate and widely accepted at present. Additional developments can, however, be expected in this evolving field.

This text was prepared for students who have an interest in experimental aspects of composite materials. It will also be useful for engineers in industrial or government laboratories who desire to extend their expertise into experimental characterization of anisotropic materials.

1.1 Background

Composite materials, in the context of high-performance materials for structural applications, have been used increasingly since the early 1960s; although materials such as glass fiber-reinforced polymers were already being studied 20 years earlier. Initially, conventional test methods, originally developed for determining the physical and mechanical properties of metals and other homogeneous and isotropic construction materials, were used. It was soon recognized, however, that these new materials, which are nonhomogeneous and anisotropic (orthotropic), require special consideration for determining physical and mechanical properties.

During this initial period, composite materials technology was developed primarily within the aerospace community. Because composite material test methods were not standardized, each airframe manufacturer tended to develop its own procedures. Although these procedures were not usually proprietary, there was little incentive to adopt common test methods, particularly because few methods had emerged as being clearly superior to others of the same type. The problem was further complicated by the continuous emergence of new materials; e.g., boron and carbon fibers in the mid-1960s and Kevlar® fibers in the early 1970s, along with new epoxies, polyimides, and other matrix materials, including metals. A specific test method, which may have performed reasonably well for the types of composite materials being tested in the past, was not necessarily adequate for the material being evaluated at that time. That is, there was little possibility of standardization. As a result, many diverse test methods were developed for measuring the same properties. Some were easy to use but provided only limited results or data of questionable quality. Others were very complex, operator-dependent, and perhaps also of questionable quality.

In the U.S. the federal government sponsored much of the early development work in composite materials, primarily through agencies such as the National Aeronautics and Space Administration (NASA), the Air Force, and the Navy. The problems associated with the lack of standards were recognized, and attempts were made to identify general test methods, to generate a database for comparison purposes, and to establish standards. These attempts were largely unsuccessful, primarily because newer composite materials did not necessarily behave in the same manner as the prior generation of materials around which the test methods had been established.

Today, almost four decades after these initial attempts, general standards for testing composite materials still do not exist, and perhaps still for the same very practical reasons. That is, as new generations of composite materials are developed, existing test methods have to be modified to accommodate them. Rigid standardization would not permit this. On the other hand, consensus organizations such as ASTM have done much to maintain a degree of uniformity, and an awareness of the general problem of achieving standardization. As additional industries, e.g., automotive, sporting goods, electronics, machine tool, and civil infrastructure, have moved toward the more extensive use of composite materials in their products, this general lack of standardization has become particularly disturbing to them. Most of the more traditional industries are accustomed to following specific design standards, purchasing materials to standards, and testing to standards. Thus, acceptance of the general lack of test method standards has become part of the indoctrination of newer industries into this relatively new technology.

This lack of standardization in composite materials testing is not necessarily a negative aspect, although it may often be inconvenient for the new user, and it should not be unexpected. That is, the term composite material does not define a specific class of fabrication materials, but rather a broad spectrum of materials of widely varying properties. Thus, it can be expected that different test methods will be required for different classes of composite materials. This philosophy is no different than that associated with using a different test method for testing low carbon steel than for testing a ceramic.

With this general background and philosophy in mind, current composite material characterization methods will be discussed and evaluated in the following chapters. Not every known method will be introduced, however. Some methods that were previously popular are now rapidly fading from use. Thus, although these names are familiar to many, and are frequently quoted in the literature, particularly in the older literature, they are becoming obsolete and need not be discussed here. Additional discussion can be found in References 1 and 2.

1.2 Laminate Orientation Code

Typically, the basic building block of a composite material structural component is a unidirectional lamina, i.e., a thin layer consisting of reinforcing fibers all oriented in the same direction and imbedded in a matrix such as a polymer. Alternatively, the reinforcement can be in the form of fibers woven to form a layer of fabric, a thin mat of randomly oriented fibers, or some similar form. All of these laminae are typically characterized experimentally using the test methods described in this text.

However, in the actual structural design process, these individual laminae are stacked and processed together to form a laminate of the desired properties. Such a laminate can be made as complex as required to satisfy the specified design criteria, by adding more and more plies of arbitrary orientations, reinforcement forms, and material types. Until the early 1970s there was no unified system for defining the lay-up patterns of composite laminates. As composite materials moved from the research laboratory to the production shop, the need for a common terminology became obvious.

The Air Force Structural Dynamics Laboratory included a Laminate Orientation Code in the third edition of its *Advanced Composites Design Guide* published in January 1973 [3]. This code, established by general consensus of the aerospace industry at the time, has survived to the present with minimal modification and continues to be used almost universally by the composites community. Thus, it is important for the reader to know at least its general features.

The Laminate Orientation Code, as presented in the 1973 edition of the *Advanced Composites Design Guide*, is summarized in the following sections.

1.2.1 Standard Laminate Code

The Standard Laminate Code, used to describe a specific laminate uniquely, is most simply defined by the following detailed descriptions of its features:

1. The plies are listed in sequence from one laminate face to the other, starting with the first ply laid up, with square brackets used to indicate the beginning and end of the code.
2. A subscript capital T following the closing square bracket should be used to indicate that the total laminate is shown. Although it is not good practice, as will be seen subsequently, the T is sometimes omitted. For a symmetric laminate (see Chapter 2), only the plies on one side of the midplane are shown, and a subscript capital S follows the closing bracket. A subscript capital Q is also defined in the code, to designate an antisymmetric laminate (improperly termed a quasi-symmetric laminate in Reference [3]). However, antisymmetric laminates are not commonly used.
3. Each ply within the laminate is denoted by a number representing its orientation in degrees as measured from the geometric x-axis of the laminate to the lamina principal material coordinate direction (1-axis). Material and geometric coordinate axis systems are described in Chapter 2. Positive angles are defined as clockwise when looking toward the lay-up tool surface. Note that this convention is consistent with the definition of a positive angle in Figure 2.3, because there the view is toward the surface of the laminate, i.e., away from the lay-up tool surface.
4. When two or more plies of identical properties and orientation are adjacent to each other, a single number representing the angular orientation, with a numerical subscript indicating the number of identically oriented adjacent plies, is used.

   For example, a laminate consisting of just three –45° plies would be designated as $[-45_3]_T$. The notation $[-45]_{3T}$ is also acceptable, and in fact is more commonly used.
5. If the angles of otherwise identical adjacent plies are different, or if the angles are the same but the materials are different, the plies are separated in the code by a slash.

For example, a two-ply laminate consisting of a +45°–ply and a –30°–ply of the same material would be expressed as $[45/-30]_T$. Note that the first ply listed in the code is always the first ply to be laid up in the fabrication process. Note also that the plus sign is not used unless omitting it would create an ambiguity.

A six-ply symmetric laminate consisting of identical plies oriented at +45, 0, –30, –30, 0, and +45° would be expressed as $[45/0/-30]_S$.

When a symmetric laminate contains an odd number of plies of the same material, e.g., –30, 90, 45, 90, and –30°, the center ply is designated with an overbar, i.e., $[-30/90/\overline{45}\,]_S$.

6. When adjacent plies are at angles of the same magnitude but of opposite sign, the appropriate use of plus and minus signs is employed. Each plus or minus sign represents one ply and supercedes the use of the numerical subscript, which is used only when the directions are identical (as in item 4, above).

   For example, a four-ply laminate consisting of plies oriented at +20, +20, –30, and +30° would be designated as $[20_2/\mp 30]_T$. Note that ∓ and not ± is used here, to preserve the intended order.

7. Repeating sequences of plies are called sets and are enclosed in parentheses. A set is coded in accordance with the same rules that apply to a single ply.

   For example, a six-ply 45, 0, 90, 45, 0, and 90° laminate would be designated as $[(45/0/90)_2]_T$, or alternatively as $[(45/0/90)]_{2T}$. As in item 4, above, this latter form is no more correct, but is more commonly used.

8. If a laminate contains plies of more than one type of material and/or thickness, a distinguishing subscript (or superscript) is used with each ply angle, to define the characteristics of that ply. For example, $[0_g/90_k/45_c]_S$ for a glass, Kevlar®, and carbon/fiber laminate.

1.2.2 Basic Condensed Code

When the exact number of plies need not be specified (as in preliminary design when the laminate in-plane properties but not the final laminate thickness are needed), the Basic Condensed Code can be used. The plies are written in the order of ascending angle, with only the relative proportions being expressed by whole number subscripts.

For example an actual $[30_2/0_6/-45_2/90_4]_T$ laminate would be expressed using the Basic Condensed Code as $[0_3/30/-45/90_2]$. An actual 30-ply $[90/\pm(0/45)]_{3S}$ laminate would be expressed as $[0_2/\pm 45/90]$. In both examples, the lack of a subscript after the closing bracket indicates that it is a Basic Condensed Code.

1.2.3 Specific Condensed Code

When the total number of plies and their orientations need be preserved, but not their order (stacking sequence) within the laminate, the Specific Condensed Code is used. This code is useful at that point in preliminary design when the laminate is being sized (i.e., when the required total number of plies is being specified). It is also particularly useful to the materials purchasing group because the scrap losses during cutting of the plies, and thus the amount of material that must be ordered, depends on the orientation of each ply in the laminate.

Using the Specific Condensed Code, the actual 30-ply $[90/\pm(0/45)]_{3S}$ laminate used in the previous example would be expressed as $[0_2/\pm45/90]_{6C}$. Note that a full 30-ply laminate is still expressed, the subscript C indicating however that the stacking sequence of the plies has not been retained.

1.2.4 Summary

Although the Laminate Orientation Code may appear complicated at first, it is systematically constructed and is as concise as possible. For simple laminates the code reduces to a simple form, and is easily and quickly written. Yet the most complex laminate can be coded with equal conciseness.

1.3 Influences of Material Orthotropy on Experimental Characterization

The individual lamina (i.e., layer or ply) of a composite material is often the basic building block from which high-performance composite structures are designed, analyzed, and fabricated. Unless stated otherwise, the lamina material is usually assumed to exhibit linearly elastic material response as, for example, in the analyses presented in Chapter 2. In many cases this is a reasonable assumption. However, there are exceptions, particularly in terms of shear response, that sometimes must be accounted for.

1.3.1 Material and Geometric Coordinates

Each composite lamina typically possesses some degree of material symmetry, i.e., principal material coordinate axes can be defined. These lamina are then oriented within a multiple-lamina composite (the laminate) at arbitrary angles with respect to some general geometric coordinate system.

For example, in designing or analyzing the stresses in an automobile, it may be logical to define the x-axis as the forward direction of the vehicle, the y-axis as the lateral direction, and the z-axis as the vertical direction, maintaining a right-handed coordinate system. This coordinate system is termed geometric (or global) because its directions correspond to the geometry of the body to which it is attached.

Because the stiffness and strength properties of a lamina (ply) of composite material are typically not isotropic, e.g., the material is typically orthotropic, it is convenient to define these material properties in terms of directions coinciding with any material symmetries that exist. The corresponding coordinate system is termed a material coordinate system.

For analysis purposes, it is necessary to express the properties of all lamina of the laminate in terms of a common (global) coordinate system, the logical choice being the geometric coordinate system. Thus, it is necessary to transform the material properties of each individual lamina from its own material coordinate system to the global (geometric) coordinate system. These transformation relations (familiar to many as the Mohr's circle transformations for stress and strain) must therefore be developed, and are presented in Chapter 2.

1.3.2 Stress–Strain Relations for Anisotropic Materials

The number of independent material constants relating stresses to strains, or strains to stresses, is dependent on the extent of material symmetry that exists. If the components of stress are expressed in terms of components of strain, these constants are called stiffnesses. If the components of strain are expressed in terms of components of stress, these constants are called compliances. Defying simple logic, the symbol C is customarily used to represent stiffnesses, and the symbol S is used to represent compliances. Literal translations from the non-English language of the original developers account for this confusion. These notations are now seemingly too ingrained to reverse, despite the novice's desire to do so.

In the most general case of a fully anisotropic material (i.e., no material symmetries exist), a total of 21 material constants must be experimentally determined. As material symmetry is introduced, it can be shown that certain of the stiffness terms (and the corresponding compliance terms) become zero, thus reducing the number of independent material constants. Some examples of practical interest are indicated in Table 1.1.

The last entry in Table 1.1, that of isotropic material behavior, is a familiar one. In this case, the material properties are the same in all directions, i.e., an infinite number of planes of symmetry exist, and only two stiffness constants are required to fully define the stress–strain response of the material. Engineers commonly utilize E, ν, and G, termed the Young's modulus, Poisson's ratio, and shear modulus, respectively. However, these three stiffness quantities must mutually satisfy the isotropic relation [5]

$$G = \frac{E}{2(1+\nu)} \tag{1.1}$$

Thus, only two of the three quantities can be independently prescribed.

TABLE 1.1

Number of Independent Material Constants as a Function of Material Symmetry [4]

Type of Symmetry	Number of Independent Material Constants
None (triclinic material)	21
One plane of symmetry (monoclinic)	13
Three planes of symmetry (orthotropic)	9
Transversely isotropic (one plane of isotropy)	5
Infinite planes of symmetry (isotropic)	2

In Chapter 2, an orthotropic material is chosen because it is the material symmetry of major interest. For example, it is representative of a unidirectional composite lamina, as well as many other composite material forms.

1.4 Typical Unidirectional Composite Properties

A very large number of different fiber–matrix combinations have been developed over the years. Nevertheless, the general classes of polymer–matrix composites can be characterized by a few representative materials. In the examples presented in Table 1.2, all properties are normalized to a common fiber volume of 60%.

The columns are ordered from left to right in terms of increasing composite axial stiffness, as primarily dictated by the fiber type. Spectra® is a polyethylene fiber developed by Allied Chemical Corporation, Petersburg, VA. Its relative inability to bond with polymer matrices accounts for the low transverse normal, longitudinal shear, and axial compressive strengths indicated for the unidirectional composite. Note that its highly oriented polymer structure also results in extreme values of coefficients of thermal expansion. Another polymeric fiber is Kevlar 49®, an aramid fiber produced by E.I. du Pont de Nemours and Company, Inc. While the compressive and transverse properties of this composite are generally better than those of the Spectra polyethylene fiber composite, they are still low relative to most of the other composites. AS4, IM6, and GY70 are all carbon fibers, representative of low, medium, and high modulus carbon fibers. The first two were produced by Hercules Corporation, and the third was produced by the Celanese Corporation. In all cases the epoxy matrix indicated is Hercules 3501-6 or a similar polymer. This is a high structural performance, but

TABLE 1.2

Typical Properties of Various Types of Polymer Matrix Unidirectional Composites (Nominal 60 Percent Fiber Volume)

Composite Property	Spectra/ Epoxy	E-Glass/ Epoxy	S2-Glass/ Epoxy	Kevlar 49/ Epoxy	AS4/ PEEK	AS4/ Epoxy	IM6/ Epoxy	BORON/ Epoxy	GY70/ Epoxy
E_1 (GPa)	31	43	52	76	134	138	172	240	325
E_2 (GPa)	3.4	9.7	11.7	5.5	10.1	10.3	10.0	18.6	6.2
G_{12} (GPa)	1.4	6.2	7.6	2.1	5.9	6.9	6.2	6.6	5.2
ν_{12}	0.32	0.26	0.28	0.34	0.28	0.30	0.29	0.23	0.26
X_1^T (MPa)	1100	1070	1590	1380	2140	2275	2760	1590	760
X_2^T (MPa)	8	38	41	30	80	52	50	60	26
X_1^C (MPa)	83	870	1050	275	1105	1590	1540	2930	705
X_2^C (MPa)	48	185	234	138	200	207	152	200	70
S_6 (MPa)	24	72	90	43	120	131	124	108	27
α_1(10^{-6}/°C)	–11.0	6.4	6.2	–2.0	–0.1	–0.1	–0.4	4.5	–0.5
α_2(10^{-6}/°C)	120	16	16	57	29	18	18	20	18
β_1 (10^{-4}/%M)	1.0	1.3	1.1	1.9	0.5	0.4	0.3	0.2	0.2
β_2 (10^{-3}/%M)	3.2	3.0	3.0	3.5	3.2	3.1	3.1	3.2	3.2
ρ (g/cm^3)	1.13	2.00	2.00	1.38	1.57	1.55	1.60	2.02	1.59

brittle, epoxy, resulting in strong but not highly impact resistant composites. The PEEK matrix is polyetheretherketone, a high-temperature thermoplastic. It is included here along with the brittle epoxy matrix to permit, in particular, a direct comparison with the AS4/epoxy composite system.

The rows in Table 1.2 indicate unidirectional composite in-plane material properties, i.e., stiffnesses, strengths, and hygrothermal properties. As will be discussed in greater detail in Chapter 2, the subscript 1 indicates the axial direction (the fiber direction), and subscript 2 indicates the in-plane transverse direction. Standard symbols are used and are defined as follows:

E_1	Axial stiffness
E_2	Transverse stiffness
G_{12}	In-plane shear stiffness
ν_{12}	Major Poisson's ratio
X_1^T	Axial tensile strength
X_2^T	Transverse tensile strength
X_1^C	Axial compressive strength
X_2^C	Transverse compressive strength
S_6	In-plane shear strength
α_1	Axial coefficient of thermal expansion
α_2	Transverse coefficient of thermal expansion
β_1	Axial coefficient of moisture expansion
β_2	Transverse coefficient of moisture expansion
ρ	Density

It is immediately obvious from Table 1.2 that the various unidirectional composite materials are highly orthotropic in terms of all of their mechanical and physical properties. The axial stiffness varies by an order of magnitude. Many have negative axial coefficients of thermal expansion, some much more negative than others. Yet, all of the materials have positive transverse coefficients of thermal expansion, although some are almost an order of magnitude higher than others. Many similar observations can be made by studying this table.

Overall, the use of composite materials offers the designer tremendous design flexibility and potential. However, because the strengths are also highly orthotropic, being very low in transverse tension and compression, and in shear, special care must be taken to design properly with them. The next chapter summarizes the analysis procedures required.

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