Handbook of Ceramics and Composites

Volume 1

Synthesis and Properties

Edited by: Nicholas P. Cheremisinoff

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SYNTHESIS AND PROPERTIES

edited by

Nicholas P. Cheremisinoff

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Preface

The *Handbook of Ceramics and Composites* is devoted to composite and ceramic materials of engineering importance. Coverage of composites includes both metallic and nonmetallic structures. This first volume contains overview chapters concerning the structural and physical properties of important classes of materials. The first chapter provides an overview of techniques for the strain characterization of composites and ceramic materials. Chapter 2 deals with the alumina-silica system, which is one of the most important binary oxide systems in science and technology as a high-temperature material, a basic ingredient in glasses, and others. Chapter 3 covers the properties of zirconia-toughened mullite ceramics, an important candidate for high-temperature structural applications.

Chapter 4 concerns compaction and mechanical characteristics of ceramic powders. An understanding of this subject matter is necessary in order to characterize raw ceramic powders and to analyze the microstructure of compacted powders during pressure-forming processes. Chapter 5 discusses the use of silicon-based ceramics in severe services in industrial systems such as heat engines and heat exchangers. Epoxy resin networks are treated in Chapter 6, with emphasis given to the technique of dynamic mechanical spectroscopy. These resins are highlighted because of their extensive use in reinforced matrixes. Chapters 7 through 11 are devoted to various characteristics, properties and end-use performances of fiber-reinforced composite materials. Chapter 7 covers planar-random fiber composites; Chapter 8 provides engineering modeling discussions on porous glass wool composite strength. Chapter 9 is devoted to stress corrosion cracking of fiberglass-reinforced plastics (FRP).

Chapter 10 discusses the fracture toughness of fiber-reinforced plastics, and Chapter 11 concerns the crush properties of FRP, which is of particular interest in automobile and aircraft construction. In Chapter 12, a general review of classes of composites is given with emphasis on polymer-based lignocellulosic fiber composites. The low-temperature thermochemical expansion behavior of polymer composites is treated in Chapter 13. Chapter 14 deals with engineering structural theory in reinforced plastics, and Chapter 15 is devoted to the important fabrication operation of laser cutting.

Chapter 16 makes an important departure at this point in the volume, devoting discussions to the preparation of high-density ferrites. These materials are magnetic oxides containing iron as a major metallic component. Their importance is in telecommunication and various microwave devices. Chapter 17 concerns metal matrix composites (MMC), which have principal space and aircraft applications. Continuing discussions are given in

Chapter 18, which covers synthesis by casting and solidification routes. Since many metal fabrication techniques involve liquid metallurgy, Chapter 19 serves as an introduction to this subject by treating binary diffusion in molten metal.

This volume represents the time, efforts, and opinions of over 40 experts. Each author is to be viewed as responsible for the statements and recommendations made in respective chapters. The editor extends heartfelt gratitude for their devotion in this work. Gratitude is also extended to Marcel Dekker, Inc., for the fine production of this series.

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Techniques for Strain Characterization of Materials

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FIBROUS COMPOSITE MATERIALS

Introduction

The strength and modulus of a single unidirectional ply of fiber/resin composite, in the direction of the fibers, are given by the law of mixtures as

$$\sigma_{CA} = V_F\sigma_{FA} + (1 - V_F)\sigma_{MA} \quad \text{and} \quad E_{CA} = V_F E_{FA} + (1 - V_F)E_M$$

(Refer to the Notation section for definitions.) At elevated temperature or simply under prolonged loading, the matrix resin will relax, owing to creep or cracking. The composite strength and modulus then reduce to the minimum values

$$\sigma_{CA} = V_F\sigma_{FA} \quad \text{and} \quad E_{CA} = V_F E_{FA}$$

These minimum-value equations depict the situation in which the load is carried by the fibers, and the matrix plays the secondary role of transferring load between fibers and across breaks at low stress levels.

Perpendicular to the fibers, stress is transmitted alternately by fibers and resin in series, like a chain whose strength is limited to that of its weakest link. In this case, the strength of the composite transverse to the fiber is essentially the strength of the resin

$$\sigma_{CT} \sim \sigma_M$$

The composite transverse modulus is a complex function of fiber and matrix moduli and of the fiber-packing configuration and density. It is usually closer to the matrix modulus than to the fiber modulus. Relaxation of the resin reduces both the transverse strength and effective long-term transverse modulus.

Composite shear strength and modulus parallel and transverse to the fibers are also limited by the resin. Fibers can move axially with respect to their neighbors with only local matrix distortion. Thus relaxation or creep of the resin reduces the shear strength and effective long-term shear modulus.

High-performance composites require maximum possible fiber content. This requires that the fibers be laid parallel to one another without crossovers. Any other packing traps more resin and thus lowers performance.

Stress in more than one direction must be handled by fibers laid in each direction, because a fiber composite can only sustain significant stress parallel to the fiber. Any number of stress directions can be accommodated by a laminate of plies, provided that those directions are restricted to a single plane. Shear stress must be carried as direct tension and compression by fibers laid at 45° to the shear axes. Otherwise, as noted above, the resin properties become limiting.

It is impossible to arrange fiber in a three-dimensional array and retain a high fiber content. Three-dimensional loading may be carried by a structure of two-dimensional laminates. Chopped fiber or complex weaves can handle stress in three dimensions, but these are not high-performance composites because of the unavoidably high proportion of resin. A further category of composite materials is fiber-reinforced plastics. As the name implies, they primarily depend on the matrix properties and are best considered as plastics or as whatever other material is used for the matrix.

This summary of the properties of high-performance fiber composite materials illustrates a distinction which simplifies understanding. Strain characterization of high-performance composites can be simply described. The complications introduced for composites which directly depend on resin properties can then be separately considered. Such materials are found in situations in which the load directions cannot be clearly defined and quasi-isotropy is desired, where for any reason a high proportion of resin cannot be avoided, or simply as a result of poor design.

An example of established poor design was the construction of pressure vessels and tanks by filament winding at an angle of 55° from the axis. This had the irresistible attraction of a lay-up ideally suited to filament winding, and it was justified by netting analysis. Because the axes of principal stress, the loads, are in the hoop and axial directions, the 55° lay-up depended on shear stress and the vessels were limited by resin properties. The axial and hoop lay-up of the cylindrical portion of such vessels, as required for high performance, is more difficult, but it can be done. High-performance lay-up of the domed ends, where loads are hoop and radial, is excluded by geometry. Such ends are best made from a truly isotropic elastic material such as a metal.

Strain Characterization

Structures and components are built to withstand specified conditions of load. This usually means that the strain response to a given load must be acceptable. Collapse or fracture is only rarely the indication of failure. Strain-at-fracture is set as an ultimate value which may only be approached but never attained.

Strain-at-failure may be estimated in design, but can only be fixed from failure tests of prototypes. Such tests allow an informed decision in setting the design strain. It is important to know the margin of safety between design strain and strain-at-failure, as well as the degree of linearity of the strain at its design level.

It is convenient to speak of strain but the term *strain response* is better in some ways. It suggests that more than one strain may be important. It also emphasizes that strain, or unit deflection, is a response to load and should be thought of primarily as an indication of the stress induced by applied loads.

It is difficult to measure stress directly. Most methods of indicating stress are primarily indications of strain or deflection. Thus strain response is a way of indicating the distribution of stress in the structure or component. It also enables the worst or most demanding combination of load or stress to be defined for the design.

Against this background, it is important to know the strain response of composite materials. They cannot be characterized out of context because they are tailored to each structure. It is not just that different component materials may be used in different proportions and laid in different directions; it is also that the geometry of the structure determines the method by which the component materials may be laid up, and the method imposes limits on the proportions of and properties attained by, the component materials. The strain responses of the component materials may be characterized out of context, but the strain responses of composites of those components can only properly be defined in relation to a given structure and lay-up method. As an example of this, the wound ring cannot be characterized from the properties of flat laminate for reasons outlined under ''ring tests'' in the section ''Methods for Applying Strain'' at page 14.

Strain characterization of composite materials will then be considered in relation to the process of designing, prototyping, manufacturing, and monitoring a structure or component. It will be discussed initially in relation to high-performance composite materials and extended, where appropriate, to include the complications of matrix-dependent materials.

Strain Characterization Testing

Strain characterization must involve testing, but a simple listing of all tests used could be misleading. This section identifies tests without detail and puts them into a logical scheme for the development of a structure or component made from composite materials. As already said, it is primarily for high-performance composites. The extensions necessary to deal with lower-performance materials where matrix properties have a direct role are indicated.

The logical scheme for the development of a structure or component using composite materials is outlined in Table 1. It is here expanded and discussed.

Design Characterization Tests

Design characterization tests are all those tests necessary to build up the data and methods required for the design of a structure or component. A considerable body of design data is now established, but other tests are required from time to time as more component materials become available, methods improve, and understanding increases.

Component materials characterization. Component materials are characterized by simple tests which are described in the next section. The requirements of these tests are listed here.

1. Fiber. Single-fiber tension test; diameter, density, etc., are also measured. Fiber tow (resin impregnated and cured) tension test; short-term characterization and long-term stress rupture are required.

Table 1 Strain Characterization Testing Scheme

Design characterization tests
Component materials characterization
Component materials compatibility
Laminate and typical structural element tests
Prototype testing
Component materials characterization
Component materials compatibility
Load/strain tests of complete prototype
Acceptance and quality control test specification
Quality control tests
Acceptance tests
Service monitoring

2. Resin (or other matrix). Cast samples tension tests to give, at temperature, the stress-strain curves, creep, and stress rupture. Resin viscosity, density, pot life, etc., are also needed.

Component materials compatibility tests. Not all reinforcement and matrix materials can be used together. Where they can, it is necessary to know their suitability for, and the limits to, their combination. Tests to indicate this are listed here.

1. Fiber wetting in the chosen resin: fiber sink rates in the resin; composite porosity measured for cast samples.
2. Fiber-resin bonding: interlaminar shear tests; both short beam and ring and other tests.
3. Fiber packing in resin: molded samples, to check the packing properties of the tow; ring-winding tests, to check packing properties of the hoop layer and axial or 45° layer thickness under a hoop winding.

Laminate and typical structural element tests. The basic element of fiber composite materials is the unidirectional lamina. It is first necessary to relate its properties to those of its constituents and their proportions. Next comes the laminate made from several laminae laid at different angles to, and in proportions necessitated by, the applied loads. It would be ideal if, once laminate properties are known, they could be applied to deduce the properties of simple structural elements. Unfortunately, laminate theory can only give an indication of the properties of such elements, and it must be substantiated for all the different typical geometries and their associated lay-up methods. The geometry determines the lay-up method, and the two together govern the matrix/fiber distributions, residual stresses, deflections, stability, etc.

Laminate properties must be known with respect to temperature and time. The following list shows the different types of tests, which are detailed in the next section.

- Tension test in fiber direction (to confirm rule-of-mixtures)
  - Of flat lamina
  - Of a hoop wound ring
- Tension test at 0° of a 0°/90° cross-ply
  - Flat laminate and/or cross-ply ring as above

To confirm rule-of-mixtures for the 0° ply
Acoustic emission and microscopy to check that resin does not crack in the 90° layer
Creep tests to demonstrate relaxation of the 90° layer (or 45° layer)
At-temperature tests to show upper limit for 0° layer and lower limit for 90° layer
Note that ring tests overcome end grip problems; must be pressure loaded; essential if product is tube
Thick ring test for E_A and E_T, and ν_{TA}/ν_{AT}
Compression test—columns
Bending test—long beam
Disk tests—spin tests, static deflection
Plate tests

Laminate tests for low-performance composites. Low-performance laminates require the same tests as high-performance composites except that loads at a range of angles to the fiber directions must be included. Because resin properties are governing in the presence of shear stress, time and temperature dependency tests become important. Tests to examine failure modes and to establish the necessary large factors of safety (ignorance) become necessary.

Prototype Testing

The data obtained and the methods developed from the design characterization tests described above or from the literature or new work are used to design the required structure or component. For a new design, prototype testing is then essential. There are too many variables to permit immediate production unless the product is completely noncritical.

Component materials characterization and component material compatibility tests. These tests are precisely the same as described for design characterization tests. If such tests have already been done, repeating them at this stage would only be a quality control measure. As will be shown later, the number of tests essential for quality control is small.

Load/deflection tests of prototype. Essentially this test loads the component to its operating level and holds the load for an appropriate time. Ideally it should be kept under load for its design lifetime, but usually this is impractical and it is held at load until there is no change of deflection (strain response). The most severe combination of conditions is maintained at this time (e.g., maximum operating temperature, wetness, wind or other secondary loads, etc.).

If deflections (strain responses) are measured at lower loads, nonlinearity of the deflection/load curve could be sufficient to reject the design as likely to fail due to continued increase in deflection. Increase of the load above its design level to reach nonlinearity, fracture, or excessive deflection will indicate the margin of safety.

Strain under test can be measured by an extensometer built onto the test machine, by calibrated telescope or microscope, or by proximity probe. Strain patterns can be shown by photoelastic coatings, by brittle surface lacquers, or by interferometry.

Acoustic emission monitoring during load increase is valuable to ensure that there is negligible resin cracking or fiber breakage. Monitoring the increases of emission above the design load is also of assistance in assessing the margin of safety.

Inspection for damage after load testing at both low and high levels can be instructive. Ultrasonic, x-ray, or γ-ray searches for cracks or delaminations are possible methods.

Surface cracks may be located by dye penetration. Selected small areas can be cut out, polished, and examined by microscope. Change of vibration response is an easy and reliable indication of the presence of damage.

Acceptance tests and quality control test specification. As the prototype is progressively loaded, the chosen acceptance test can be regularly applied to check its response to increasing load and deflection. This enables the acceptance criteria to be set and shows whether the chosen test is affected by damage.

As an example, if the component were a pressure tube, its weight, density, fiber fraction, and transverse resonance frequency would be good checks that there was no change in the product. As damage accumulated from overload conditions, the resonance frequency would drop. This suggests the possible periodic use of the resonance test during the life of the item, to check for the onset of failure, thereby permitting operation closer to the failure level.

Quality Control Tests

After successful prototype testing comes production. Quality control must ensure that all the items manufactured would pass the same tests as the prototype by discovering and removing any that would not. The subject of quality control is not at issue here, but clearly there would have to be sufficient testing of the composite material components to give confidence that they remained within specifications. There is then the vexed question of intermediate tests along the production path, but, as will be seen, the process of manufacturing fiber composite materials is to some degree an inherent inspection process. It is assumed that the acceptance test of the structure or component would be the final quality control test.

Fiber. Inspection on receipt, observation during processing (breakages, fuzz, dryness, etc.), and the tensioning and bending applied during lay-up (e.g., by filament winding) are constant and effective quality controls. In addition, it may be wise to wet-out and cure occasional samples of tow and check their breaking strain. If there are tube off-cuts, it is much simpler and better to pressure burst one of them.

Resin. Inspection on receipt, observation during processing (viscosity when mixing, color, smell (with care for the health hazard), pot life, gel time) and inspection of the finished component (correct resin runoff, full cure, color) are constant and effective quality controls. In addition, it is wise to make the occasional set of "dog bone" specimens (ASTM [D 638]*) for tensile load/deflection testing at room, operating, and above-operating temperatures to be sure that resin quality is maintained.

The composite component. If an acceptance test is sensitive to composite modulus (e.g., measurement of resonance vibration frequency), it will also indicate variations of resin and fiber properties. If such a test is sufficiently developed, fiber and resin quality testing, apart from observation, may only be needed retrospectively to identify problems in rejected components. Informed visual inspection, supplemented by measurements of critical dimensions, is the most fundamental and important quality control test.

*ASTM references in the text give the number of the relevant standard of the American Society for Testing and Materials.

Acceptance Tests

Acceptance tests are devised to ensure that all items manufactured would pass the tests that were applied to the prototype and to satisfy potential users or statutory authorities that the structure or component is satisfactory for its duty. Acceptance tests should be nondestructive and quickly performed. They should have been applied to the prototypes, and their relationship to the prototype tests should have been demonstrated.

Samples cannot in general be used for acceptance test purposes unless they were made by precisely the same process as the production item. An off-cut from the production item would be acceptable and could obviate the need to test the item itself. An example of this would be an off-cut ring from a pressure tube, which could reasonably be accepted as representative of the whole tube. Weight, density, fiber fraction, color, and other simply checked properties are obvious acceptance checks.

Resonance vibration frequency is recommended as an acceptance test. It subjects the whole item to strain and is sensitive to variations of density, modulus, shape, etc. It is very quick, sensitive, and nondamaging. Other forms of acceptance tests could be ultrasonics or radiography.

Statutory requirements may still require a repetition of the prototype test. A prime example of this is the overpressure test required on a pressure vessel. It has always been argued that such tests unnecessarily damage the vessel and may thereby hasten its failure, but it is not reasonable to expect such an emotionally reassuring test to be eliminated. All that can be said here is that damaging acceptance tests should be avoided where possible. High-performance composite pressure vessels (i.e., having fiber laid in the directions of principal stress) would be little affected by a 50% overpressure because they would be fully elastic up to fracture, whereas a composite vessel with the "balanced" 55° lay-up could be significantly damaged.

Service Monitoring

In certain cases, such as large structures, it is not feasible to construct a prototype. Transducers may be incorporated in the structure to allow monitoring of the response to service loads. The data from these allow refinement of the design codes. Monitoring the degradation of stiffness to determine the time for retirement from service is also possible and can be desirable whether or not a prototype was tested.

Techniques for Strain Characterization

In this section, the techniques used to measure or observe strain are described. Each technique may be applicable to several of the categories of strain characterization testing described in the preceding section.

For strength testing it is desirable to subject a substantial quantity of material to an applied uniform strain. In this way the presence of representative strain-concentrating flaws can be ensured. The concept of "gauge length" arises from this consideration as well as from the need to have an extension or deflection sufficiently large to permit precise measurement. In such a test it is deflection or extension which is observed, and from it average strain is calculated.

In practice the strain in a component is not usually uniform, and failure is likely to initiate in areas of concentrated strain. Techniques are needed then to survey the full field of strain and indicate where it is most concentrated. It is then necessary to be able to measure it at that location. The idea of strain at a point is carried to its ultimate with microscopic