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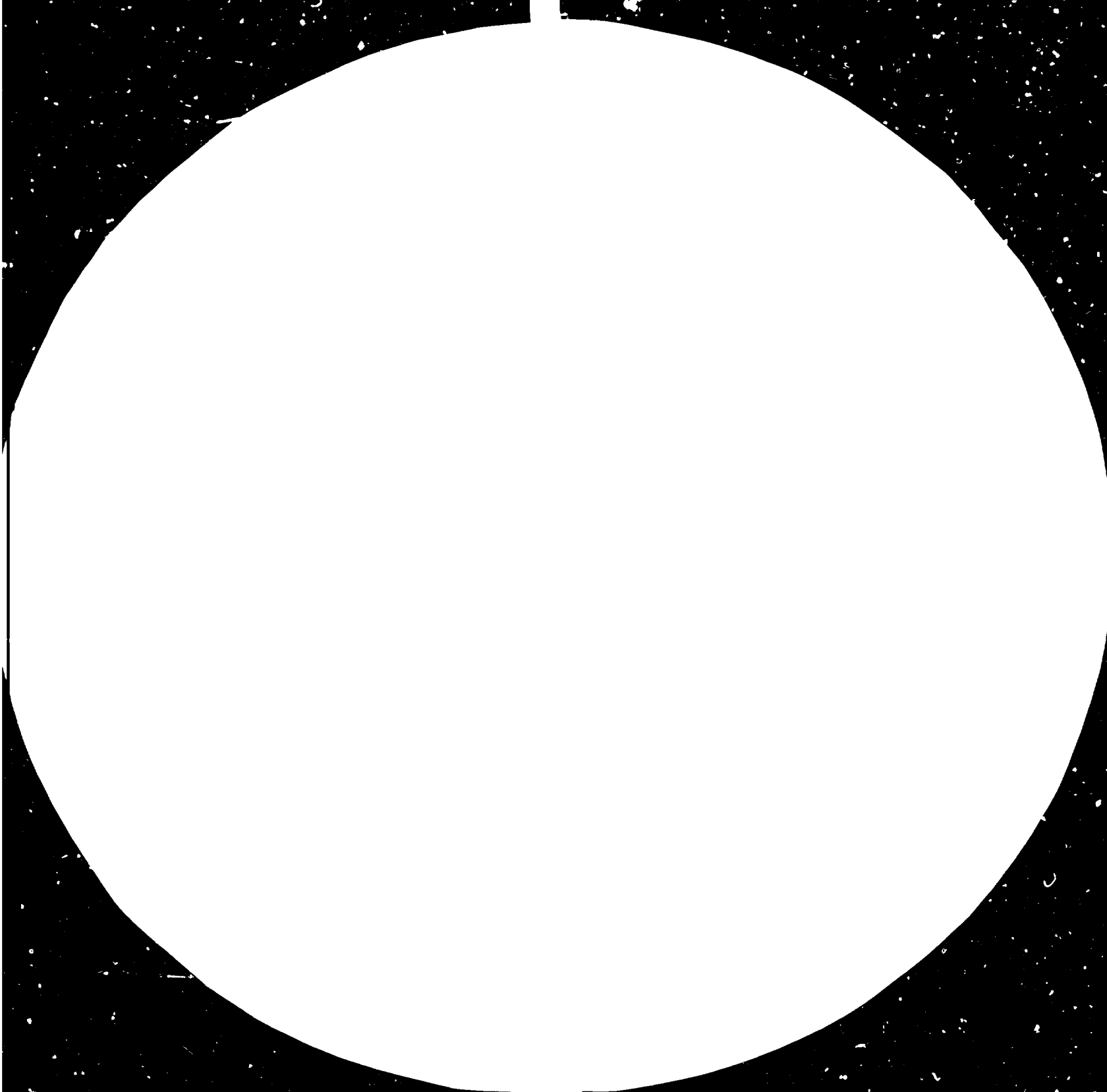

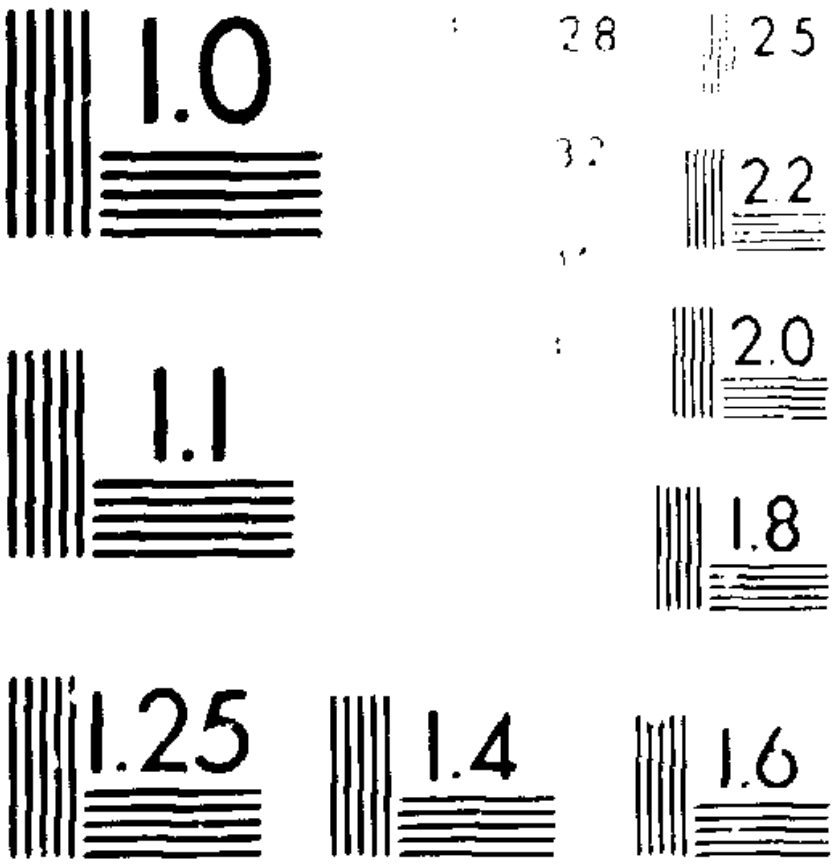

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CARBON FIBRE-REINFORCED PLASTICS TESTING AND PROPERTIES OPTIMIZATION*

by

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Introduction

The proper testing of composite materials has been a topic of continuing emphasis for more than 25 years. Early work was with glass fiber reinforcements; and the problem was not altered significantly when the high modulus fibers such a boron and graphite were introduced in the mid-1960's. The principal problems continue to be the ability to introduce the load into the highly anisotropic composite material in a uniform manner, and to accurately measure the small strains developed. Both problems do become somewhat more difficult to overcome when testing carbon fiber-reinforced plastics (CFRP), because of the relatively low shear strengths of these composites relative to their axial tensile strengths, and the fact that a higher stiffness for a given tensile strength translates into an even smaller axial strain to be measured.

An additional complication when testing CFRP is associated with the negative coefficient of thermal expansion of the carbon fiber itself. When these fibers are combined with a polymer matrix material of limited stiffness but very high thermal expansion, a composite of extremely low thermal expansion can result. While this is often a very attractive attribute of CFRP, e.g., in its use in dimensionally stable structures, the adequate measurement of these very small strains creates special problems.

Since most structural polymers absorb considerable amounts of moisture (5 to 10 percent by weight) and swell in the process, while carbon fibers do not, it is also necessary to characterize the moisture expansion properties of CFRP. Unlike thermal expansion, which occurs rapidly with temperature change and quickly stabilizes, moisture expansion is associated with the very slow diffusion of water molecules

through the polymer. Even in a relatively thin composite, e.g., 1 mm thick, this process can take many weeks. Since very accurate measurements must be made over long periods of time, special data acquisition problems are encountered.

A variety of other unique measurement problems also exist when testing CFRP, associated with their low thermal and electrical conductivities (which are also anisotropic), high damping properties, low impact resistance, low delamination resistance, etc.

Thus, it is not difficult to understand why CFRP testing continues to be a topic of major interest within the composite materials community.

Test Methods

Test methods covers a broad range of technologies. Particular emphasis will therefore be concentrated here on test methods primarily measuring static mechanical properties. In terms of most properties of composite materials, these properties are most difficult to measure for unidirectionally-reinforced materials, since the degree of anisotropy is highest in this configuration. Unidirectional composite, or lamina or ply, properties are of primary importance, of course, since the unidirectional ply is the building block of most actual composite structures. Thus, test methods developments are usually focused on unidirectional composites.

To characterize a unidirectional composite, it is desired to measure the strength and stiffness both parallel and perpendicular to the direction of fiber reinforcement, i.e., both axial and transverse properties. Since different failure mechanisms govern, both tensile and compressive properties must be measured. Shear properties parallel to

the fibers must also be determined. Shear properties perpendicular to the fibers, i.e., through the thickness of the unidirectional ply, have not historically been of primary interest, and are not usually easy to measure. However, with increasing concern for free edge (delamination) effects associated with induced interlaminar stresses (both normal and shear), these stresses can no longer be automatically neglected.

In summary, primary static structural properties of the unidirectional ply include:

σ_1^{yt}, σ_1^{yc} - axial yield strengths in tension and compression

σ_1^{ut}, σ_1^{uc} - axial ultimate strengths in tension and compression

σ_2^{yt}, σ_2^{yc} - transverse yield strengths

σ_2^{ut}, σ_2^{uc} - transverse ultimate strengths

τ_{12}^{y}, τ_{12}^{u} - longitudinal shear strengths, yield and ultimate

E_{11}^{t}, E_{11}^{c} - axial modulus, tension and compression

E_{22}^{t}, E_{22}^{c} - transverse modulus, tension and compression

ν_{12}^{t}, ν_{12}^{c} - major Poisson's ratio, tension and compression

G_{12} - longitudinal shear modulus

α_{11}, α_{22} - coefficients of thermal expansion, axial and transverse

β_{11}, β_{22} - coefficients of moisture expansion, axial and transverse

It is recognized that α and β are physical rather than mechanical properties, but they are typically also required for complete analysis. Often the compressive stiffness properties E_{11}^{c} and E_{22}^{c}, and the major Poisson's ratio in compression ν_{12}^{c}, are not measured, being assumed equal to the corresponding tensile properties.

The properties listed above are typically adequate for basic design purposes, where linearly elastic material response is assumed. However, it is often desirable to perform a nonlinear analysis, in which case complete stress-strain curves must be documented. Unfortunately, these data are often not collected. If temperature and moisture absorption are also design parameters, then all of the above properties must be generated over a range of temperatures and moisture contents. This could result in a prohibitively large test matrix (prohibitive in terms of cost, and calendar time required). Thus, often only room temperature, dry and elevated temperature, wet conditions are selected, "elevated temperature" implying the maximum use temperature expected in the application, and "wet" corresponding to the quasi-equilibrium moisture content to be achieved. (A value of approximately one percent by weight in CFRP in a typical environment is representative.) If practical, tests at all nine combinations of three temperatures and three moisture contents should be performed, as a minimum.

Although not specifically addressed in this brief paper, many other properties besides those static properties listed and discussed here must often be evaluated in completing a specific design. These include fatigue, impact, creep, relaxation, multiaxial loading, and high rate loading, to name just a few. The proper determination of these material characteristics is often more difficult than static properties evaluations, and cannot be minimized, although outside the scope of the present discussion.

Having defined the static properties to be measured, test methods for measuring them can now be discussed.

Tensile Properties (σ_1^{yt}, σ_1^{ut}, σ_2^{yt}, σ_2^{ut}, E_{11}^{t}, E_{22}^{t}, ν_{12}^{t})

The most suitable tensile test specimen has evolved into a relatively simple form over the years. Various dog-bone shapes have given away to a simple, straight-sided specimen of constant width and thickness. The key is in the tab design, and the adhesive used to bond the tabs to the CFRP specimen. Metal tabs (steel or aluminum) have been replaced by glass fabric/epoxy tabs (printed circuit board material often being used, as being readily available and inexpensive). The tabs are generously tapered on one end, to provide a smooth transition of the applied load into the CFRP specimen. An adhesive which has a very high shear strength, and which forms a thick bond line, is desirable. The requirement of high shear strength may require an elevated temperature cure under some clamping pressure. Of course axial tensile testing is much more difficult than transverse tensile testing, because of the much higher forces to be transferred from tabs to specimen.

Either strain gages or extensometers are usually used to measure the strains. A strain measurement in the direction of loading is required to measure E, and also in a direction normal to the loading if ν is to be determined also. In a transverse tensile test, the strain normal to the loading direction is often not measured (needed to measure the minor Poisson's ratio ν_{21}^{t}), since its value is typically small and hence difficult to measure accurately. The reciprocal relation [1], i.e.,

$$\frac{\nu_{21}}{E_{22}} = \frac{\nu_{12}}{E_{11}} \tag{1}$$

states that if ν_{12} and the moduli E_{11} and E_{22} are measured, ν_{21} can be calculated. The relation also indicates that ν_{21} will be smaller than

ν_{12}, in the ratio E_{22}/E_{11}. Hence, for a highly anisotropic material such as CFRP, where E_{22}/E_{11} is typically on the order of 0.1, ν_{21} will be on the order of 0.1 ν_{12}. While ν_{21} is not listed as a property to be normally measured, if it is, Eq. (1) can be used as a check of the mutual consistency of the data set.

Commercially available extensometers [usually strain gage instrumented, but occasionally Linearly Variable Differential Transformer (LVDT) instrumented] are expensive (currently about US $1000 each), but are reusable and easily installed. Strain gages cost on the order of US $5 per single-element gage, but are not reusable, and require more labor to install. Problems can also be encountered when strain gages must be used at high temperatures (they tend to reinforce the softened matrix surface of the specimen, giving low strain readings) and moist environments (in addition to the problem of a softened matrix, the moisture may weaken the adhesive bond, causing the gage to become partially, or even completely, debonded). Hence the present author prefers to use extensometers whenever possible.

Compressive Properties (σ_1^{yc}, σ_1^{uc}, σ_2^{yc}, σ_2^{uc} E_{11}^{c}, E_{22}^{c}, ν_{12}^{c})

Although not commonly recognized as such, compressive testing might very well be the most difficult to perform properly. The problem is that compressive strength is not a uniquely defined property. The mode of compressive failure can smoothly transition from gross buckling, to local instability, to fiber microbuckling, to shear failure, to end crushing (brooming). Unfortunately, any one of these failure modes may be meaningful in an actual application, and no one measurement is a fundamental material property. That is, each depends on the "structure" of the composite. Since there may be as much as an order of magnitude

difference in measured "strength" from one extreme to the other, which failure mode a particular test method induces is obviously important. Often the test method is selected which is believed to best represent the failure mode of the structure it is to be used in, obviously a difficult task.

As a compromise, the Celanese [2] or ITTRI [3] test methods have been somewhat standardized for use. By using a common test method, the failure mode tends to be uniform from one material to the next (although not necessarily), and hence at best relative comparisons can be made. However, the use of this type of compression test is by no means universal today, and with good reason, as stated above.

Strains in compression are measured using the same devices previously described for tensile testing, with the same limitations. Although perhaps poor practice, compressive strains are often not even measured. The values of E and ν in compression are simply assumed to be equal to those in tension. Of course, this also means that complete stress-strain curves will not be available.

Shear Properties (τ_{12}^{y}, τ_{12}^{u}, G_{12})

The determination of the shear properties of composite materials has perhaps received more attention than any other over the years [4]. The problem is a combination of having many different test methods to choose from, and the fact that many of these tests are either difficult to use, or provide only limited data. For example, thin-walled tubes are relatively expensive to fabricate, and somewhat delicate to handle. Solid rods are an excellent compromise, being easily fabricated and very rugged, but do not produce a uniform state of shear stress. Strength and modulus can both be measured, and a complete shear stress-shear

strain curve can be generated, if a strain gage or rotometer is used.

In contrast, many of the commonly used shear tests permit the measurement of either strength or modulus, but not both. For example, the short beam shear or interlaminar shear test, and the notched tensile test, only provide strength, while the plate twist test only provides modulus. Others, such as the 2- and 3-rail shear tests, or the picture frame test, while claiming to provide both strength and stiffness, are suspect in strength determination because of induced stress concentrations at the attachment points.

The Iosipescu shear test has emerged during the past few years as a very promising shear test for composites [4,5]. The specimen is compact, easily fabricated and easily tested, providing both strength and modulus, for any one of the three possible shear loadings, i.e., the in-plane and two interlaminar shear components. Much study of this test method is currently underway, both analytical and experimental.

Coefficients of Thermal and Moisture Expansion (α_{11}, α_{22}, β_{11}, β_{22})

Until the past 6-8 years, most applications of CFRP were in ambient temperature environments. Also, moisture absorption effects, while known to be a potential problem, were largely ignored. Thus, there had been little concern with measuring mechanical properties over a range of hygrothermal conditions, and even less concern with measuring the coefficients of thermal and moisture expansion of the various composites. This has now changed. Elevated temperatures and moist environments are now an integral part of the service conditions of composite hardware, and must be accounted for in design and stress analysis. This is particularly true as performance demands increase and the margins of safety decrease. For example, even the curing-induced

thermal residual stress can no longer be ignored, as once was the case. To predict these stresses, and to account for them in the design process, the coefficients of thermal expansion, both parallel to the fiber reinforcement, α_{11}, and perpendicular to the reinforcement, α_{22}, must be known. The same can be said for the coefficients of moisture expansion, β_{11}, and β_{22}, when humidity is present. Since β has the units of strain per unit moisture absorption (usually expressed as weight percent moisture relative to the dry composite), it is also necessary to know the moisture absorption rate (moisture diffusivity) and moisture saturation level of the composite.

As previously noted, the axial coefficient of thermal expansion α_{11} of CFRP is difficult to determine since the values are typically very small (on the order of 10^{-7}/°C, or less). Standard quartz tube dilatometers are not sufficiently sensitive, and some type of interferometer is usually required. Thus, the cost of determining α_{11} goes up. On the other hand, α_{22} is usually readily measured using a dilatometer, since values may be in the 10^{-5}/°C range. One distinct advantage of thermal expansion testing is that the specimen can be brought to thermal equilibrium relatively quickly. Thus, testing is rapid.

In contrast, moisture equilibrium can be a very slow process; it may require one to two months for even a relatively thin (1 mm) specimen to reach equilibrium. Thus, testing is slow, and potentially expensive. Also, with limited equipment of this specialized nature available to most laboratories, the volume of data generated to date is still very limited [6]. Although the magnitudes of β_{11} are small relative to β_{22}, the problem of measuring β_{11} is not quite as severe as when measuring

α_{11}. Nevertheless, a standard quartz tube dilatometer (equipped, of course, for humidity control) may be marginally adequate.

Often it is feasible to measure the α and β of the neat polymer matrix itself (more easily achieved since these values are typically large), and then use a micromechanics analysis to predict values of α_{11} and β_{11} (see for example, References [6,7]).

Data Reduction Techniques

The days of reading dials and recording data on clipboards have now passed for the most part. Chart recorders have become the primary mode of data acquisition. Data reduction must still be done manually in this mode, however. Material modulus is the estimated best-fit slope of the stress-strain plot (usually a force-strain plot, suitably converted), while yield and ultimate strengths are points picked off the chart. Having only a single hard copy analog plot of the raw data, the tendency is to digitize the data manually into these few points of information. Thus, much potentially useful information is lost to future users.

The current trend is to develop computerized data acquisition systems. That is, the raw data are stored in digital form on magnetic tape or disk, along with information as to specimen identification, specimen dimensions, test conditions, etc. While the raw data can be immediately reduced to usable forms, e.g., moduli, Poisson's ratios, strengths, and stress-strain curves, and displayed on a CRT, in essentially real time as the test progresses, this is not the sole, or even the primary, benefit of computerization. The most important advantage is that the data are permanently on file, available for immediate recall at any time. Thus, if at some future time (days,

weeks, or even years later), it is desirable to restudy the data, or reduce them in another manner, or compare them with more recently acquired data, this can be done almost instantly. No information is lost during the lapse of time. This obviously increases the value of testing.

Such computerized data acquisition and reduction systems are currently expensive, however, and demand a considerable number of user hours to get familiar with them and their capabilities. Thus, a testing laboratory must be willing and able to expend considerable funds to get started (perhaps on the order of US $100,000), and designate at least one dedicated user. Presently, such systems are somewhat unique; in another decade they will probably be commonplace, and considerably less expensive.

Property Prediction Techniques

As emphasized in previous sections, the complete, or even reasonably complete, static material properties characterization of a unidirectional CFRP material is a major undertaking, both in terms of calendar time and cost. While excessive cost is always a strong consideration, calendar time may often be also. If several weeks are required to fabricate and test a laminate, little can be done to speed the process. If a month or two are required to moisture precondition a specimen, the time scale is set. Even when time is not a governing factor, the fabrication, instrumentation and testing of hundreds of specimens, with sufficient numbers of replicates to ensure a reliable data base, is a very labor intensive process.

During the past 20 years, work has proceeded steadily toward the development of so-called micromechanics analyses for predicting unidirectional composite properties based upon known constituent

material (fiber and matrix) properties and the composite geometry (fiber volume fraction, fiber shape and packing array, interface bond performance, and thermal and moisture history). While early models were crude, and often very empirical in nature (e.g., rule-of-mixtures, Halpin-Tsai equations, etc.), subsequent models have become very rigorous in their representation. In particular, the finite element method of analysis, which has come into common use during this same time period, and the growth of large scale digital computers, have combined to revolutionize micromechanics analyses.

It is now possible to model arbitrary constituent material stress-strain response, i.e., both time-independent and time-dependent material nonlinearities, crack initiation and propagation, interface debonding, temperature- and moisture-dependent material properties, and anisotropic material response, using either a 2D or 3D analysis [8-13]. Limited analytical/experimental correlations available to date [6,8,9,14] indicate that bulk properties (E, ν, G, α, β) can be predicted very accurately using current methods. Work is now proceeding toward the improvement of methods to predict point properties (strengths), and good progress is being made.

The realistic goal of micromechanics analyses is not to replace composites testing, but rather to supplement it. That is, the analyses would be used to predict those properties not measured experimentally as part of a limited test program, using correlations with measured values as verification of the predictive ability. In this manner, complete data tables could be made available at a fraction of the cost of a fully experimental program, since a computer simulation is much less expensive than an actual experiment.

An important secondary advantage of a demonstrated micromechanics analysis capability is in optimizing existing composites, and developing new systems. A hypothesized combination of existing or proposed fiber and matrix can be analytically "tested" to establish its potential. Only the most promising systems then need be subjected to the more costly and time consuming laboratory testing. This capability currently exists; it may be in widespread use in another decade.

Brasil's Potential Role in CFRP Testing

With the possible exception of computerized data acquisition, which does require sophisticated equipment and extensive resources, much of the needed development of composites test methods and properties optimization primarily involves careful thought, attention to detail, and considerable personal effort. Thus, any composites group is a potential contributor to the technology.

Certainly Centro Tecnico Aerospacial (CTA) in San Jose dos Campos is an excellent example of an organization in Brasil which has the facilities to do this type of work. Perhaps the weak element at present is the lack of background knowledge of what has already been done elsewhere. Thus, selected personnel must be provided with current literature, and have the opportunity to visit other laboratories, and talk with other investigators. International conferences such as the present one are an excellent source of such contacts.

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