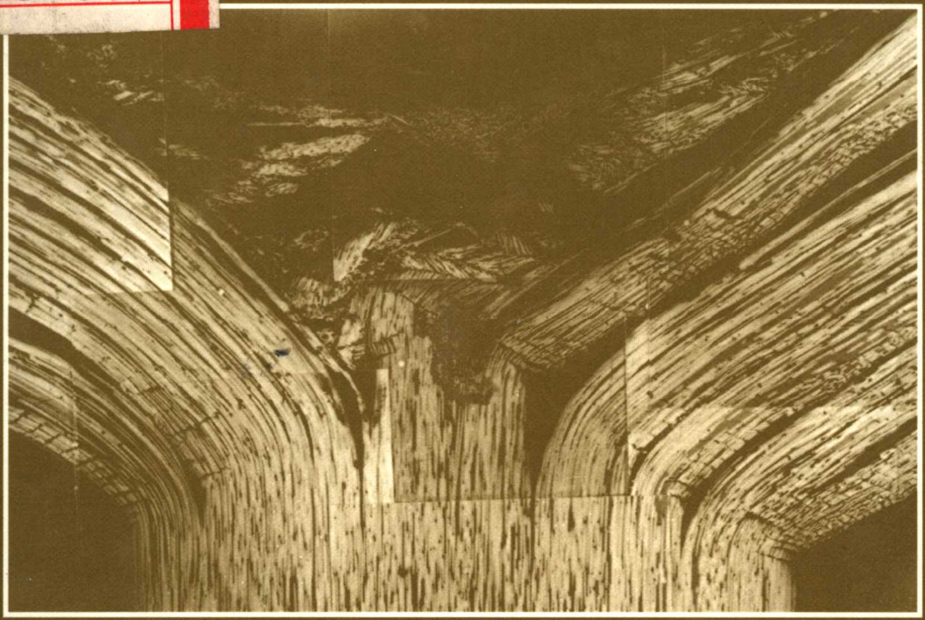

COMPOSITE MATERIALS

Testing and Design

TWELFTH VOLUME

Ravi B. Deo and Charles R. Saff, editors

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The quality of the papers in this publication reflects not only the obvious efforts of the authors and the technical editor(s), but also the work of these peer reviewers. The ASTM Committee on Publications acknowledges with appreciation their dedication and contribution to time and effort on behalf of ASTM.

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Foreword

The symposium on Composite Materials: Testing and Design (Twelfth Volume) was held 16–17 May 1994 in Montreal, Canada. ASTM Committee D30 on High Modulus Fibers and Their Composites sponsored the symposium. Ravi B. Deo, Northrop Corporation, and Charles R. Saff, McDonnell Douglas Aerospace Corporation, presided as symposium cochairmen and are editors of this publication.

Contents

Introduction

Ravi Deo and I originally intended this symposium to focus on how industry has solved some of the problems associated with implementation of composite materials in products and hardware. As papers were submitted and reviewed for presentation, it became apparent that we were receiving papers with a greater emphasis on general solutions to the testing and design problems encountered by industry than on specific examples of solutions developed by industrial firms. While this may seem at cross purposes to our original plan, the results may ultimately be more useful to the technical community than a collection of specific examples would have been.

Because this volume contains more general examples of test and analysis methods applied to composite materials and structures, we believe it will serve two purposes; one to present new test and analysis techniques and the other to present the trends of the technology development in composite materials in this decade. The focus of the work presented herein is toward applications in more aggressive environments and reducing the cost of composite applications and testing to ensure economic viability.

This volume, containing 21 papers, should be valuable to both researchers and to those involved in the design and analysis of structures using advanced composite material systems. The papers cover four primary topics ranging from evaluation of the effects of environmental aging and physical damage to evaluation of new product forms and the analysis and design of structures implementing these new product forms.

Environmental Effects Testing

Papers in this category reflect the emphasis in the research environment toward long term applications of composites in higher temperature structures. The effects of environmental aging include physical as well as chemical changes in the material which affect the mechanical response of the materials. Short term test techniques that can identify these behaviors and quantify their effects are valuable tools for determining what matrices are most useful for these long term applications. The fiber-matrix interface, or interphase material, if it exists, can often determine the life of the composite system even when the matrix material is capable of long term exposure without degradation. Either by wicking moisture of corrosion products into the system, or by directly attacking the integrity of the fiber, the interphase (or interface) degradation can cause sudden catastrophic failures not predicted by tests of fiber or matrix alone. Much more work needs to be done in this area. NDE methods must be developed to determine when composites have been damaged by heat to the point that their mechanical properties have been degraded. Environmental stress cracking and hygrothermal cracking are additional degradation mechanisms that must be understood so that short term tests can identify those systems which will survive and perform in aggressive environments.

Design Allowables and Damage Tolerance Testing

Test techniques for damage tolerance testing continue to be a source of new ideas for tests that can be interpreted rationally for structural applications. Two new experimental techniques are evaluated herein: one on the use of static indentation to mimic impact damage in composites and the other on the use of a torsion specimen to eliminate specimen end effects on delamination fracture testing. While both methods show promise, static indentation test methods still require a rigorous test of their limitations, and application of the Mode III delamination results to realistic structures remains unproven. Of particular interest to structural designers and analysts will be the papers included in this section dealing with skin stiffener debonding in composite panels, mechanical characterization of syntactic foam core panels, and comparison of static indentation versus impact damage in composite tubes. These papers address a common problem in design of these structures in interpreting simple test results in the analysis of complex, realistic structural geometries.

Textile and Other Advanced Composites

Papers in this section address two widely disparate systems: textile composites and metal matrix composites. Textile composites have long been a source of improved damage tolerance for both impact and manufacturing damage. However, as industry has accepted their use into cocured structures, evaluation through the thickness strength has become a concern. In a related vein, analysis to predict the properties of the wide variety of possible textile product forms has become a requirement, since no one can afford to test all the possibilities for a given application. Much more needs to be done here to assess all of the properties required for design purposes, but the analyses presented provide a start toward this development.

Metal matrix composite (MMC) materials have been relegated to the high temperature end of the composite family as resin matrix composite capabilities for intermediate temperatures expand. However, the analysis and design of structures under the high temperature loading conditions of these extreme environments becomes a complex study. But, even though the materials differ, the effects of creep at high temperatures, coupled with variable amplitude loadings, is a study as relevant to thermoplastic matrix systems at intermediate temperatures as it is to MMCs. This is likewise true of the study presented on inelastic behavior of MMC under compressive loadings.

Design, Analysis, and Test Techniques

These papers examine the interpretation of test data from a variety of mechanical test techniques, ranging from micromechanical tests of creep in high temperature composites throught the testing extension-twist-coupled composite laminates to the development of design allowables using regression models. These papers focus on the need in composite materials to link the analysis of the test result to the specimen and its loading to develop useful data for evaluation of real structures. From micromechanical models to overall structural models, analysis of the failure modes, loadings, and processes is vital to the proper application of the data to design and analysis of composite hardware.

The papers contained herein represent a broad spectrum of the applications being examined for composite materials today and a look toward the potential application of the future. While few specific hardware applications are presented, knowledgeable reviewers will see reflections of the direction of composite applications of the future in the data presented herein. And the test techniques presented herein point in the directions of simplified tests, greater depth of analytical interpretation, and ease in application to real structures. These are the directions of composite technology for the foreseeable future.

We want to acknowledge the efforts of the authors, presenters, reviewers, and the ASTM staff for making this volume possible.

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Environmental Effects Testing

Thomas S. Gates[1] *and Mark Feldman*[2]

Effects of Physical Aging at Elevated Temperatures on the Viscoelastic Creep of IM7/K3B

REFERENCE: Gates, T. S. and Feldman, M., **"Effects of Physical Aging at Elevated Temperatures on the Viscoelastic Creep of IM7/K3B,"** *Composite Materials: Testing and Design (Twelfth Volume), ASTM STP 1274,* R. B. Deo and C. R. Saff, Eds., American Society for Testing and Materials, 1996, pp. 7–36.

ABSTRACT: Physical aging at elevated temperatures of the advanced composite IM7/K3B was investigated through the use of creep compliance tests. Testing consisted of short-term isothermal, creep/recovery with the creep segments performed at constant load. The matrix-dominated transverse tensile and in-plane shear behavior were measured at temperatures ranging from 200 to 230°C. Through the use of time-based shifting procedures, the aging shift factors, shift rates, and momentary master curve parameters were found at each temperature. These material parameters were used as input to a predictive methodology that was based upon effective time theory and linear viscoelasticity combined with classical lamination theory. Long-term creep compliance test data was compared to predictions to verify the method. The model was then used to predict the long-term creep behavior for several general laminates.

KEYWORDS: composite materials, testing, design, polymeric composites, physical aging, creep (materials), viscoelasticity

Strength, stiffness, and weight considerations make polymer matrix composites (PMCs) desirable for structural materials in such diverse applications as aircraft, civil infrastructure, and biomedical implants. One common aspect shared by structural designers and material developers for all of these PMC applications is the concern for long-term durability. When considering durability, adverse environmental conditions, high loads, elevated temperature, and fatigue may all play a role in determining the useful lifetime of a PMC structure. To address these problems, accurate analytical predictive methods and comprehensive material property characterization techniques are essential.

One material behavior that is ubiquitous to all polymeric materials is physical aging. This type of aging is a thermoreversible process that occurs due to the evolution of the free volume in a polymer toward thermodynamic equilibrium after cooling below glass transition temperature (T_g). This evolutionary process may be affected by many parameters including temperature, moisture, and stress. Struik [*1*] showed that it was possible to isolate the physical aging process in polymers from other behaviors by performing isothermal creep compliance tests and using superposition techniques to establish the aging-related material constants. Linear viscoelasticity was used to characterize the creep, and the "effective time" theory was developed to aid in predicting the long-term creep compliance from short-term test data.

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It is the intent of this paper to utilize the test techniques and analysis tools given by Struik [1] and adapt them to the elevated temperature characterization of physical aging in an advanced graphite/thermoplastic PMC. For the purposes of this research, short term was defined as tests spanning approximately three decades of time whereas long term was defined as tests spanning approximately five decades. Material property testing consisted of short-term, isothermal, creep/recovery with the creep segments performed at constant load. Both transverse tensile and in-plane shear behavior were measured. Through the use of time-based shifting procedures, the aging shift factors, shift rates, and momentary master curve parameters were found at each temperature.

These material parameters were used as input to a predictive methodology given by Brinson and Gates [2] that was based upon effective time theory and linear viscoelasticity combined with classical lamination theory. Long-term creep compliance test data were compared to predictions to verify the method. The model was then used to predict the long-term behavior for several general laminates.

Traditionally, time/temperature superposition (TTSP) provides a method by which long-term behavior can be predicted from short-term tests, thereby providing an accelerated test scheme. As described by Griffith et al. [3], TTSP has a long history of use in viscoelastic characterization of materials and a number of procedures have been proposed to arrive at the TTSP master curve. However, for long-term creep, Struik's [1] work with polymers and more recently tests on PMCs by Sullivan et al. [4,5] and Feldman and Gates [6,7] have shown that TTSP by itself is not sufficient to account for the long-term effects of physical aging. This has led to the development of the effective time theory as a way of predicting the effects of physical aging.

Material System

The PMC material used in this study was a continuous carbon fiber reinforced, amorphous thermoplastic polyimide with 62% fiber volume fraction. The material was fabricated by DuPont and designated IM7/K3B. The as-received T_g was 240°C as measured by dynamic mechanical analyzer (DMA) G″ peak. Chemical aging of the K3B polymer was not expected to occur. For this study, it was therefore assumed that the T_g of IM7/K3B would remain constant over the duration of the material characterization tests used in the current study. All test panels had excellent initial quality as revealed by post-fabrication C-scan.

All methods verification and material property tests utilized a rectangular specimen of approximately 241 by 25 mm with a 12-or 8-ply thickness. Per ply thickness was approximately 0.00135 mm. The transverse and shear creep compliance data came from $[90]_{12}$ and $[\pm 45]_{2s}$ specimen layups, respectively. Three to four replicates were used at each test temperature. Although all the specimens came from the same material lot, many of the replicate specimens were cut from different panels.

Test Procedures

Testing was performed to understand material behavior, to develop material constants for the analytical model, and to provide verification of the predictive model. This section will highlight some of the important test procedures. Specific procedures and techniques relating to testing may also be found in Feldman and Gates [6].

Short-Term Testing

The test temperatures selected for the study were 200, 208, 215, 225, and 230°C. These temperatures were selected to ensure that measurable aging occurred within the test period.

Prior to testing, the specimen was heated to 250°C (10° above T_g) for 20 min. By rapidly quenching the specimen from above T_g to the test temperature, it was ensured that all test specimens started the test sequence in the same unaged condition. This procedure was based upon work by Struik [1] and others who showed physical aging is thermoreversible and the excursion above T_g prior to quenching effectively rejuvenates the material.

A well-documented [1] technique to explore physical aging is a series of sequenced creep (constant load) and recovery tests as shown in Fig. 1. The basic procedure is that while the specimen isothermally ages, periodic creep tests are performed using a constant applied load. For this study, the duration of each creep test was 1/10th the duration of the prior total aging time. These creep tests are termed momentary tests. The aging times (time after quench) selected for starting each creep segment were 2, 4, 10, 24, 48, 72, and 96 h. Figure 2 shows creep compliance data from a typical sequenced test. The curves in Fig. 2 have a similar shape on the double log plot and are shifted horizontally relative to the initial (2-h) curve. This shift is an indication that physical aging affects creep compliance. The effects of sequencing on the test results were examined by Gates and Feldman [7] where it was determined that the sequencing procedure had little or no effect on the data. Damage to the specimens along the free edges due to thermomechanical loading was inspected for with a high power optical microscope. Only those specimens without damage (that is, microcracks) were used in the final study.

All creep tests were performed in convection ovens. Thermocouples monitoring the test temperature and providing feedback for the oven controller were placed near the test section. Load was applied through a dead-weight cantilever arm arrangement. Strain in the gage section was measured with high-temperature foil strain gages applied back to back at the center of the specimen.

Between the creep segments of the sequenced test, creep recovery took place. To facilitate recovery, the specimen remained in the oven at temperature while being completely unloaded.

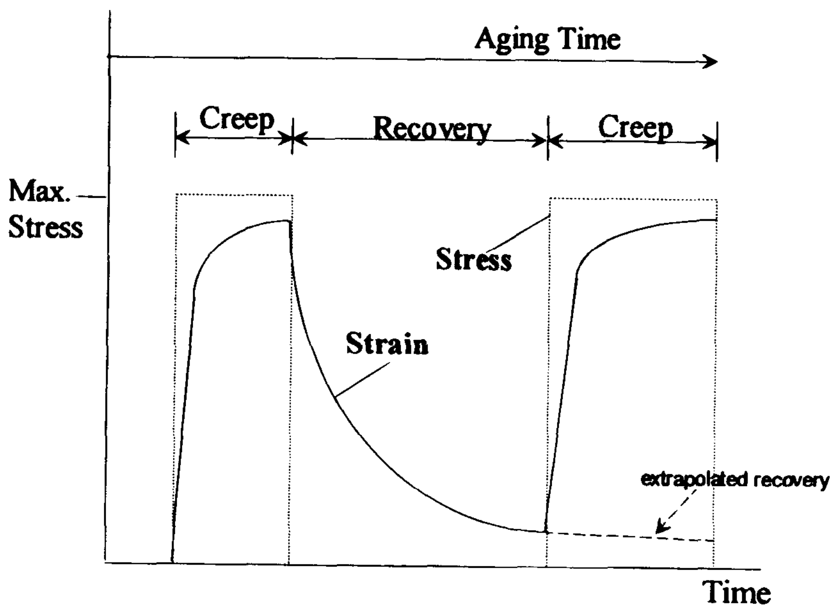

FIG. 1—*Schematic illustrating sequenced creep/recovery tests.*

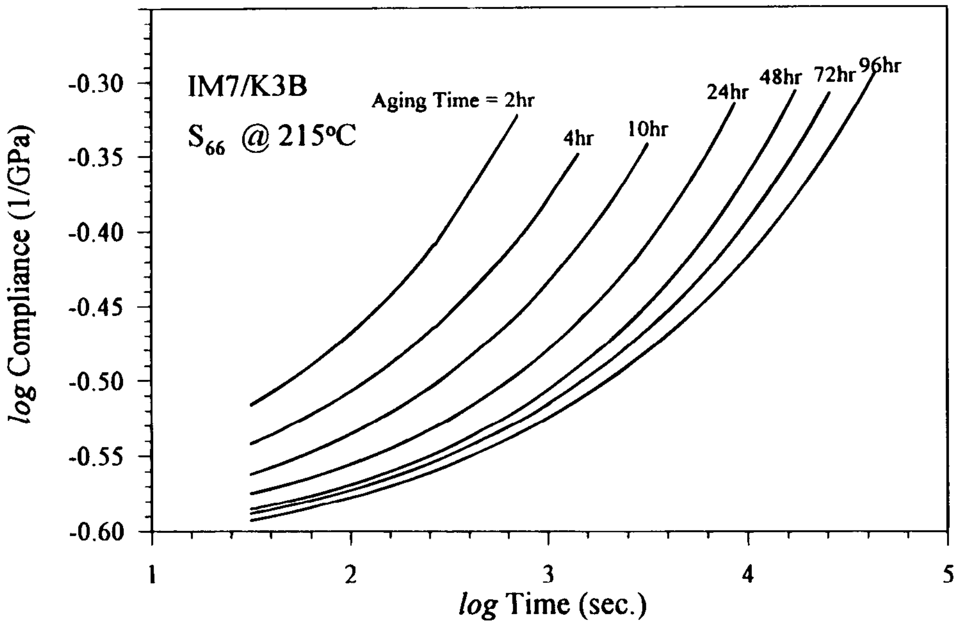

FIG. 2—*Typical shear compliance momentary curves.*

Strain data were taken during both the creep and recovery segments. Residual strain due to lack of complete recovery was accounted for fitting the final part of the recovery curve with a linear function and subtracting the extrapolated recovery strain from the subsequent creep strain. Figure 1 illustrates this extrapolated recovery strain. Strain variations during the course of the test due to thermal apparent strain were accounted for through the use of the thermal apparent strain correction technique [*8*].

Linearity—The choice of stress levels for the creep tests were based on linearity checks at the highest test temperature for a given layup. For linear viscoelastic behavior, it was assumed that superposition and proportionality conditions would be met. Given an initial state of stress, σ', applied for a time, t, and an additional stress, σ'', applied at a time, t_1, Boltzman's superposition principle [*9*] states that

$$\epsilon[\sigma'(t) + \sigma''(t - t_1)] = \epsilon[\sigma'(t)] + \epsilon[\sigma''(t - t_1)] \tag{1}$$

Therefore, Eq 1 implies that given creep data, superposition would allow the exact prediction of the subsequent recovery period so that

$$\epsilon(t^*) = \sigma\{S(t) - S(t - t_1)\} \tag{2}$$

where σ is the constant stress, t_1 is the time of load removal, t^* is the time during strain recovery, and $S(t)$ is the creep compliance function. Creep and creep/recovery data provided data for checking superposition.

Proportionality states that for an applied stress, σ, the strain in a material at any other stress state is found using

$$\epsilon[c\sigma(t)] = c\epsilon[\sigma(t)] \tag{3}$$

where c = constant.

Proportionality was checked by plotting isothermal creep compliance versus test time for a specimen that was repeatedly rejuvenated, quenched, and loaded at various stress levels. The supposed transition from linear to nonlinear behavior would be evident by the vertical separation of the compliance curves with increasing stress. These checks for proportionality were made at the lowest and highest test temperatures thereby ensuring that the effects of applied stress were minimized for all temperatures and a linear assumption could be used in the model with assurance of reasonable accuracy.

Long-Term Testing

For a time scale measured in "log" seconds, the short-term test data required by the analytical model spanned approximately 2.5 to 3 decades. To verify the analytical predictions of long-term creep compliance, long-term tests were run with data taken over approximately five decades of time. Test procedures prior to initial loading were the same as those described for the short-term tests. The initial unloaded aging time was 2 h. After load application, the specimen remained at a constant load for the remaining duration of the test period. Therefore, the total loaded test time proceeded for approximately the same time as aging. It was expected that the effects of aging on the creep compliance would become apparent after the total aging time exceeded the time span used in the momentary tests.

Analytical Model

An analytical model was developed [2] to predict the long-term creep compliance given the material properties developed from short-term tests as input. Defining "long term" as any time greater than the time scale of the material property tests, it was expected that the model would provide insights into the effects of physical aging on the long-term viscoelastic behavior of advanced polymeric composites.

Linear Viscoelastic Creep Compliance

The ability to satisfy Boltzman superposition and proportionality requirements were the conditions necessary for linear viscoelastic behavior. In general, the time-dependent linear creep compliance was modeled with a three-parameter expression given by

$$S(t) = S^0 e^{(t/\tau)^\beta} \tag{4}$$

where S^0, τ, and β are the initial compliance, retardation time, and shape parameter, respectively.

Time-Based Superposition—Time/aging-time superposition of the short-term creep compliance test data provided the means for the data to be collapsed into a single momentary master curve (MMC) at each test temperature. As demonstrated by Struik [*1*] and illustrated in Fig. 2, horizontal separation of the sequenced creep compliance curves is due to aging and can be characterized by the aging shift factor (-log a). This shift factor is simply defined as the horizontal distance required to shift a compliance curve to coincide with a reference compliance