A Comparison of Tension and Compression Creep in a Polymeric Composite and the Effects of Physical Aging on Creep

Thomas S. Gates
Langley Research Center, Hampton, Virginia

David R. Veazie
Clark Atlanta University, Atlanta, Georgia

L. Catherine Brinson
Northwestern University, Evanston, Illinois

August 1996

Abstract

Experimental and analytical methods were used to investigate the similarities and differences of the effects of physical aging on creep compliance of IM7/K3B composite loaded in tension and compression. Two matrix dominated loading modes, shear and transverse, were investigated for two load cases, tension and compression. The tests, run over a range of sub-glass transition temperatures, provided material constants, material master curves and aging related parameters.

Comparing results from the short-term data indicated that although trends in the data with respect to aging time and aging temperature are similar, differences exist due to load direction and mode. The analytical model used for predicting long-term behavior using short-term data as input worked equally as well for the tension or compression loaded cases. Comparison of the loading modes indicated that the predictive model provided more accurate long term predictions for the shear mode as compared to the transverse mode. Parametric studies showed the usefulness of the predictive model as a tool for investigating long-term performance and compliance acceleration due to temperature.

1

Introduction

Long term mechanical behavior of advanced polymeric composites (PMC's) are a critical issue for many modern engineering structural applications such as those found in the biomedical, civil infrastructure, and aerospace disciplines. Three primary concerns exist when the engineer addresses long term performance of PMC's: 1) screening for final materials selection, 2) acquiring the data base of critical engineering properties that extend over the projected life-time of the structure, and 3) developing the combined analysis and test methods that provide a means of predicting durability and performance. The intent of this research is to present the results of recent work that applies to all three of these concerns.

The three basic constituents of advanced PMC's are fiber, interphase, and matrix. In many circumstances the polymeric matrix can be the major constituent that contributes to degradation or changes in durability of PMC's. Changes in composite stiffness, strength and fatigue life can all be related to changes in the mechanical properties of the polymer matrix. As shown in Bank et al. [1] the matrix was found to be the key constituent in the durability of PMC's subjected to long term exposure at elevated temperatures. Their report also demonstrated that individual test methods combined in an integrated scheme will provide an accurate method for understanding the different contributions of various degradation mechanisms to durability. Therefore, to develop an understanding of the degradation factors, the methods pursued in this work concentrated on matrix dominated behavior.

With this background established, the current work has utilized elevated temperature creep tests to determine the effects of physical aging on the long term viscoelastic compliance of an advanced PMC. Selection of creep to asses viscoelastic behavior in PMC's was based upon the previous work by many separate investigators. A recent review of this topic by Scott et al. [2] found that viscoelastic models (both linear and nonlinear) should take into account the effects of shearing deformations to ensure accurate predictions. In addition it was found that temperature was a primary factor in creep behavior and that the use of time-temperature based superposition principles provided the type of parameters necessary for making accurate long-term predictions. Other investigators such as Struik [3] and McKenna et al. [4] have used creep tests of neat polymers to determine the effects of physical aging on long term creep. In these studies investigations were made into the concepts of effective time theory and relationships between aging and free volume evolution. Combining creep tests, concepts of linear viscoelasticity, and mechanics of composite materials has been done more recently by investigators such as Hastie and Morris [5], Sullivan [6], and Wang et al. [7] utilizing high performance PMC's tested below the glass transition (T_g) temperature.

For tension loaded materials, the authors recently used the procedures and models found in the literature as a foundation for expanding the analytical and test methodologies. Brinson and Gates [8] determined that the physical aging shift rate was the most critical

parameter in calculating the magnitude of agings' effect on long term performance. Results from Gates and Feldman [9] and Veazie and Gates [10] showed that the sequenced creep testing procedures produced repeatable test data for IM7/K3B laminates loaded in tension and compression. Time/temperature and time/aging-time superposition techniques provided the material properties required to make long-term predictions.

This paper extends these investigations by comparing the effects of physical aging in tension versus compression for both shear and transverse loading modes. To establish the background for the analytical methodology, a brief summary is given on linear viscoelasticity and physical aging in composites. The properties of the test material, IM7/K3B, are given and the experimental equipment and procedures are described. The methods of data reduction are explained including the use of superposition techniques. Results from the short and long term tests are discussed. Direct comparisons between tension and compression behavior for shear and transverse loading modes are made by comparing aging shift rates, material constants, material master curves, long term data and predictions for a variety of laminates.

Viscoelasticity and Aging

An analytical model as provided in Brinson and Gates [8] was used to predict the long term tension creep compliance using as input the material properties developed from short term tests. A "long term" test was defined as a test time at least 10 times greater than the time of the 'short term" material property tests. Using this definition 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. For the purposes of this discussion, the model is briefly recounted for tension loading only. Apart from the obvious sign differences between tension and compression loading the tension based model was not modified for analysis of compression loading.

Linear Viscoelastic Creep Compliance

The time dependent linear creep compliance was modeled with a three parameter expression given by

$$S(t) = S^o e^{(t/\tau)^\beta}$$

(1)

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

Time Based Superposition

As depicted in figure 1, the material parameters required by the model are found from sequenced short-term creep and recovery tests. Time/aging-time superposition of the short term creep compliance test data provided the means for the sequenced, short-term data to be collapsed into a single momentary master curve (MMC) at each test temperature. As demonstrated by Struik [3] and illustrated in the log-log plot of figure 2, horizontal separation of the sequenced creep compliance curves is due to aging and can be

3

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 curve. A linear fit of all the shift factors versus the logarithmic aging time (*log* t_e) for each MMC, (figure 3) gave the aging shift rate

$$\mu = \frac{-d\log a}{d\log t_e}$$ (2)

where t_e is the aging time. The reference compliance curve could be any of the sequenced curves, but for convenience of data manipulation, the longest (96 hour) compliance curve was selected as the reference during formation of the MMC. However, to facilitate data reporting, all MMC parameters were subsequently referenced to the shortest (2 hour) compliance curve. For a horizontal (time) translation of a compliance curve, only the retardation time parameter needs to be recalculated. Given the aging shift rate and reference curve parameters, the translation from one aging time to another was accomplished through the use of

$$\tau_e = \tau_{ref}\left(\frac{t_e}{t_{eref}}\right)^{\mu}$$ (3)

where t_{eref} is the reference aging time (Brinson and Gates [8]).

Equation 2 implies a linear relation between *log* a and *log* t_e. Figure 3 shows this relationship for one of the replicate tests used to establish the MMC's. The shift factors for the data on figure 3 were found from the curves on figure 2 and are representative of all the IM7/K3B MMC data.

To facilitate the collapse of the shifted data for the MMC, vertical (compliance) shifts were also utilized. This use of small vertical shifts in reduction of PMC creep compliance/aging data was also reported by Sullivan [6] and Hastie and Morris [5]. The vertical shifts for all data sets were small in comparison to the magnitude of the corresponding horizontal (time) shifts. No clear trends existed in these vertical shifts and analysis of vertical shift factors versus aging time did not lend itself to developing a constant vertical shift rate. Figure 4 illustrates how a typical data set is collapsed through the use of horizontal and vertical shifts.

Composite Physical Aging

The analytical model used to make long term creep compliance predictions for PMC's required a time dependent form of laminated plate theory. As given in Jones [11], for a single lamina under plane stress conditions, the compliance matrix referenced to the material coordinate axis is,

4

$$[S] = \begin{bmatrix} S_{11} & S_{12} & 0 \\ S_{12} & S_{22} & 0 \\ 0 & 0 & S_{66} \end{bmatrix} \qquad (4)$$

where subscripts 1,2 are the material coordinates referenced to the directions along and transverse to the fiber respectively and the subscript 66 refers to shear.

As demonstrated by previous investigations, the only time dependent compliance terms in equation 4 that play a role in creep are the transverse (S_{22}) and shear (S_{66}). Therefore, using equation 1 and 2, the two time dependent terms are given as

$$S_{22}(t) = f(S_{22}^0, \beta_{22}, \tau_{22}(t_{eref}), \mu_{22}; t) \qquad (5)$$

$$S_{66}(t) = f(S_{66}^0, \beta_{66}, \tau_{66}(t_{eref}), \mu_{66}; t) \qquad (6)$$

where the numerical subscripts reference properties in the material coordinate system and S^0, β, τ, and μ are defined in equation 1 and 2. The term t_{eref} is the reference aging time.

Long Term Compliance

For test periods that exceed the time required to collect the short term (momentary) data, the response can be expected to be influenced by the ongoing aging process. Struik [3] proposed an effective time that could be used to replace time such that the compliance in equation 1 would be written as

$$S(t) = S^o e^{(\lambda / \tau(t_e^o))^\beta} \qquad (7)$$

where λ is the effective time and is calculated according to

$$\lambda = t_e^0 \ln(\tfrac{t}{t_e^0} + 1) \quad for \quad \mu = 1$$

$$\lambda = \frac{t_e^0}{1 - \mu} \left[(1 + \tfrac{t}{t_e^0})^{1-\mu} - 1 \right] \quad for \quad \mu \neq 1 \qquad (8)$$

where t_e^o is the aging time prior to loading and at some time later the total aging time is $t + t_e^o$ where t is the creep test time. Brinson and Gates [8] demonstrated that the effective time theory is self-consistent and shift rates exceeding unity are both physically and mathematically permissible. Use of the effective time expressions in the laminated plate model allowed for the prediction of long term creep compliance. Input to the model was the material parameters measured from short term tests.

5

Time/Temperature Superposition

The use of time/temperature superposition (TTSP) (Findley et al. [12]) requires that creep compliance to be a function of temperature (T) and time (t) such that

$$S = S(T, t) \qquad (9)$$

and that

$$S(T, t) = S(T_o, \varsigma) \qquad (10)$$

$$\varsigma = \frac{t}{a_T(T)} \qquad (11)$$

where ς is the reduced time that is related to the real time t by the temperature shift factor $a_T(T)$ and T_o is the reference temperature.

The collection of individual MMC's for each loading direction and loading mode can be collapsed into single material master curves using TTSP. The collapse is made using a single reference curve and horizontal (time) shifts only. Characterizing these master curves with an expression like equation 1 along with the reference aging time and reference temperature allows the investigator to calculate the individual creep compliance curve for any test condition.

Acceleration

Acceleration of aging implies that short term tests can be used to determine the equivalent state of degradation experienced in a long term test. In this study, elevated temperature was used as the primary accelerator of the physical aging process. Aging shift rate was found as a function of the test temperature and the predictive model was used to investigate the effects of elevated temperature on creep compliance.

Test Materials and Specimen Configuration

The material system chosen for this study was a continuous carbon fiber reinforced thermoplastic polyimide fabricated by DuPont and designated IM7/K3B. The fiber, IM7, was an intermediate modulus carbon fiber manufactured by Hercules. The unaged T_g in the composite as measured by Dynamic Mechanical Analyzer (DMA) G'' peak was $240°C$. Change in the T_g from the unaged condition over extended aging times was measured by industrial studies and found to remain within $3°C$ for 10,000 hours of isothermal aging at $180°C$. For this study, it was therefore assumed that chemical aging of the composite would not occur and the T_g would remain constant over the duration of the tests.

6

Rectangular test specimens similar to those described in ASTM Specification D3039-76 measuring 24.1 cm. by 2.54 cm. for tension specimens, and 20.32 cm. by 2.54 cm. for compression specimens, were cut from laminated panels. All specimens consisted of 12 or 8 plys where each ply measured approximately 0.0135 cm. thick. To reduce experimental errors at least three replicates were tested at each test temperature. Although all the specimens came from the same material lot, many of the replicate specimens were cut from different panels. Prior to testing, all specimens were dried for at least 24 hours at 110°C in a convection oven.

The in-plane transverse (S_{22}) and in-plane shear (S_{66}) creep compliance data came from unidirectional 12-ply $[90]_{12}$ and angle-ply 8-ply $[\pm 45]_{2s}$ specimens, respectively. The use of a $[\pm 45]_{2s}$ for shear characterization requires that the shear be induced in the laminate through uniaxial loading. To account for any measured differences due to the applied loading direction, shear behavior was measured from 'tension induced shear" and "compression induced shear."

Test Equipment

Testing was performed to understand the material behavior, develop material constants for the analytical model, and provide verification of the predictive model. This section will highlight some of the important test equipment and procedures. Specific procedures and techniques relating to the tensile and compressive testing may also be found in Gates and Feldman [9] and Veazie and Gates [10].

Review papers on static compression testing in composite materials by Berg and Adams [13] and Schoeppner and Sierakowski [14] revealed that many features of compression test apparatus and procedures can affect the ultimate compressive strength of PMC's. However, very little work has been presented in the literature on compressive creep of PMC's. References found in the literature showed most creep tests were performed using relatively thick test specimens. Among these, Tuttle and Graesser [15] used short (152mm) specimens with a center hole loaded in a high capacity load frame. In another investigation, Irion and Adams [16] used variations of the standard fixtures developed for static compression tests to study compressive creep at room temperature. In a study of physical aging of polyetheretherketone at elevated temperatures, Nguyen and Ogale [17] also performed compressive creep tests.

The current study used the long, thin specimens described previously. A uniaxial constant load was applied through a dead-weight cantilever arm system. These tests were performed in convection ovens equipped with digital controllers. For the tensile tests, mechanical wedge grips held the specimen during the loaded or creep segments. High temperature tabs were attached to the specimen ends to prevent slipping. For compression testing, a unique apparatus (Veazie and Gates [10]) was constructed to allow a tensile creep test frame to be used for application of a compressive load. The compressive creep apparatus consisted of two rigid frames connected by steel rods running through linear

7

bearings. To ensure stable compression, the specimen was supported from column buckling by lightweight knife edge guides. Column buckling was checked during loading by longitudinally aligned back-to-back strain gages, that would show a lack of parity in strain if simple bending occurred.

During the unloaded or recovery segments in the tension tests, the lower grip was released using a remote cable and pulley system. For recovery in the compression tests, the two inner rigid fixtures of the rigid frames were separated from the specimen by applying a slight force from the creep frame lever arm. These unloading mechanisms provided for virtually unconstrained recovery while allowing the test chamber to remain closed during the entire test sequence. (See Veazie and Gates [10] for details).

Strain in the specimen gage section was measured with high temperature foil strain gages applied in the center of the specimen. Proper selection of the gage type and adhesive gave coefficient of thermal expansion (CTE) match and stability at elevated temperatures. Thermal apparent strain was corrected for by using the compensating gage technique (Murry [18]). Laminate damage in the form of matrix cracks can alter the strain measurements therefore after each test sequence the specimens were inspected with an optical microscope for matrix cracks along their exposed edges. These inspections revealed no apparent damage after the sequenced tests.

Experimental Procedures and Data Reduction

To explore the effects of physical aging on the creep properties, a well-documented technique that measures the creep compliance as described in Struik [3] was used for all tests. This procedure consisted of a sequence of creep and recovery tests using a constant applied load while the specimen isothermally ages. The test temperatures selected for the study were 200°, 208°, 215°, 220°, 225°, and 230°C. These test temperatures were selected to ensure that measurable aging occurred within the test period.

<u>Linearity</u>

For all the creep tests, a single applied stress level was chosen for each layup and subsequently used at all temperatures. To provide compatibility, the same stress levels were used in the tension and compression tests. Determination of the stress level necessary to stay within the linear viscoelastic range was made by checking that proportionality condition and Boltzman's superposition (Findley et al. [12]) would be satisfied. Creep and creep/recovery tests provided data for checking superposition. Proportionality checks were performed by plotting isothermal, creep compliance versus test time for a specimen that was repeatedly rejuvenated (described below), 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 linearity checks were made at the lowest and highest test temperatures thereby minimizing the effects of

applied stress. This process also allowed a linear assumption to be used with assurance of reasonable accuracy.

Short-term Tests

To provide for the test condition that all specimens start the test sequence in the same unaged condition, a means of rejuvenating the specimen was required. Rejuvenation was accomplished by a procedure based upon work by Struik [3] and others who showed that physical aging is thermoreversible and the excursion above T_g prior to quenching effectively rejuvenates the material. In the current tests, the gaged specimen was heated to 250°C (10°C above T_g) for 30 minutes immediately before the start of any physical aging test sequence. High-pressure air was used to quench the specimen from above T_g to the aging temperature. Relaxation of thermal residual stresses during a tensile test after using this type of quenching procedure was investigated by Allen et al. [19]using data from a resin similar to K3B. Although thermal residual stresses may play a role when trying to compare compressive and tensile static loading, Allen concluded that during creep the effect of apparent aging due to residual stress was much weaker than the effect of physical aging itself.

The duration of each creep segment was 1/10th the duration of the prior total aging time. The aging times (time after quench) selected for starting each creep segment were 2, 4, 10, 24, 48, 72 and 96 hours. After each creep segment, the specimen was unloaded and allowed to recover until the start of the next creep test. To account for any remaining residual strain due to a lack of complete recovery, the strain measured in the creep segment was corrected by subtracting the extrapolated recovery strain from the prior creep curve as illustrated in figure 1.

The momentary (short-term) sequenced creep/aging curves were collapsed into MMC's through a horizontal (time) shift using the longest aging time curve as the reference curve. In some cases, small (as compared to the horizontal shifts) vertical (compliance) shifts were also used in reduction of the IM7/K3B data.

An individual MMC was found for both transverse and shear modes at each individual test temperature. These MMC's are given in figure 5 for tension and figure 6 for compression loading. All of these curves represent the best fit to collapsed data from all replicates used in the test program. The three curve fit parameters used to characterize each of the MMC's are given in table 1. The parameters from this fit were termed the momentary master curve parameters for a given temperature.

Material Parameters

Aging shift rates (μ) were calculated for all cases using the sets of master curves. These calculated values are given in table 1 and plotted against test temperature in figure 7. The error bars on figure 7 represent the high-low scatter in the replicate testing. Each set of master curves can be further reduced using the time/temperature superposition

principle (TTSP). Curves of creep compliance versus time resulting from the TTSP operation are given in figure 8.

Results and Discussion

Master Curves

The momentary master curves that resulted from time/aging-time superposition demonstrated that IM7/K3B varied with temperature as expected but somewhat differently than expected with regards to loading directions and modes. Creep compliance in both the tension and compression case was a strong function of test temperature with an increase in temperature resulting in an increase in compliance and associated creep rate. For all cases, TTSP provided a means for collapsing the sets of momentary master curves into single material master curves. These curves, shown in figure 8, reveal that not only do differences exist between the transverse and shear modes but differences between tension and compression loading exists as well. The repeatability of the testing and the consistency of the data indicates that although these differences may be slight, they do reflect real material behavior.

Aging Shift Rate

Based upon the known effects of physical aging in amorphous polymers, it was expected that aging shift rate would be a function of test temperature. This function would show a decrease in shift rate as the temperature approached T_g. Figure 7 in general reflects these expected trends. The study by Nguyen and Ogale [20] showed that polyetheretherketone exhibited a shift rate that was a function of loading mode. However, it was not known prior to testing how the differences due to loading direction and mode would be reflected in the IM7/K3B composite data. The differences in the four sets of data in figure 7 and the sensitivity of the long term predictions to aging shift rate imply that to obtain the highest degree of accuracy in the predictions one must account for loading mode and loading direction.

Note that in all except the 225°C case, μ_{22} is less than μ_{66}. In linear shear deformations there is no global volume change, whereas in tension there is typically a small increase in volume on deformation. Since an increase in volume would provide enhanced molecular mobility, one could correlate the smaller aging rate in the transverse tests to this volumetric difference of the type of loading in the two tests. By the same logic, since compression loading causes a small decrease in volume, one might expect that in compression loading μ_{22} would be greater than in the equivalent tension case (to represent accelerated aging due to decreased mobility). However, the results are the opposite of this scenario: in fact, μ_{22} in compression is even less than that in tension. Consequently, it appears that the volume changes provided by the loading mode and direction do not noticeably affect the aging of the material. The differences in the shift rate for different loading modes and directions must be due to some other mechanism, not due to the volumetric changes induced by loading. This postulate is supported by data of

10

Santore et al. [21] which show that for nonlinear torsional deformations, the volume increase due to the application of the load is recovered quickly upon release of the load and does not affect the baseline volumetric densification of the material due to aging, which occurs over a much larger time scale. The effect of the volumetric changes due to loading in long term test, where loading is sustained, is unknown.

Long Term Test versus Prediction

Two temperatures (215, 225°C) were selected for both the tension and compression long term tests. The results for the transverse and shear loading modes at these temperatures are given in figures 9 - 12. Predictions of long term behavior were made using material constants given in table 1. All of the long term test times lasted approximately 11 to 12 times the short term tests.

Comparing loading modes, the results indicated that prediction of shear creep compliance is more accurate than predictions of transverse creep compliance. Expanding the comparison to include loading direction indicated that only the transverse compression case gave predictions that may diverge appreciatively from the test data for long loading times. Overall, three cases over-predicted the measured data with the other five cases showing slight under-predictions of the test data. For creep compliance, an over-prediction can be considered conservative.

For the tension case, two additional layup configurations ($[30]_{12}$ and $[15]_{12}$) were tested at the 215°C temperature level. The off-axis fiber angle produced a combined state of stress with the ratio of transverse to shear stress in the specimen determined by the fiber angle relative to the load direction. These two long term, off-axis test specimens were tested in a manner identical to all the other tension tests. The test data from these off-axis specimens and the corresponding long term predictions are given in figure 12. The accuracy of the predictions for these off-axis specimens was consistent with the other cases examined above.

Long Term Behavior: Parametric Studies

Material constants from table 1 were used along with the predictive model to perform parametric studies on the long term creep behavior at elevated temperatures. Although no verification data of extremely long term behavior exists, the predictions were run out to approximately 3.2 years to provide comparisons of long term performance.

For the complete range of temperatures, figures 14 and 15 presents the long-term predictions of the tension and compression loading for both the transverse and shear modes. Examination of these results shows that temperature has a similar effect on tension and compression loading with the compression loading showing somewhat less uniformity than the tension loading cases. In addition, the compression loading produced more exponential long-term behavior as compared to the tension cases.

11

Figures 16 -18 present a direct comparison between long-term creep compliance for both loading directions and modes at 208°, 215°, and 225°C respectively. Examination of these figures reveals that initially the creep curves for the compression loaded cases were shifted lower on the compliance axis than the corresponding tension loaded cases. However, examination of the long-term predictions reveals that the compression loaded cases ended up with greater compliance compared to tension for longer times. These figures also illustrate how increased temperature will significantly alter the long term creep compliance.

Concluding Remarks

Experiments were performed to determine the effects of physical aging on creep compliance of IM7/K3B composite laminates loaded in tension or compression. Experimental results and established analytical methods were used to investigate the similarities and differences of tension and compression for both the shear and transverse loading modes.

The short term (96 hour) tests, run over a range of sub-T_g temperatures provided material constants, material master curves and aging related parameters. The test data was consistent and repeatable over the entire range of test temperatures. Comparing results from the short term behavior indicated that although trends in the data with respect to aging time and aging temperature are similar, differences exist due to load direction and mode. Temperature has a similar effect on tension and compression loading with the compression loading producing more exponential long term behavior as compared to the tension cases.

The long term (1500+ hour) predictions compared favorably to the long term test data with the model demonstrating more accuracy in the shear mode as compared to the transverse mode. The sensitivity of the long term predictions to aging shift rate imply that the predictive model must account for both loading mode and loading direction. This implication would also hold true when considering development of accelerated test methods based upon the changes in physical aging due to temperature.

References

[1] Bank, L. C., Gentry, T. R. and Barkatt, A., "Accelerated Test Methods to Determine the Long-Term Behavior of FRP Composite Structures: Environmental Effects," *Journal of Reinforced Plastics and Composites,* Vol. 14, June, 1995, pp. 559-587.

[2] Scott, D. W., Lai, J. S. and Zureick, A.-H., "Creep Behavior of Fiber Reinforced Polymeric Composites: A Review of the Technical Literature," *Journal of Reinforced Plastics and Composites,* Vol. 14, 1995, June, pp. 588-617.

[3] Struik, L. C. E., *Physical Aging in Amorphous Polymers and Other Materials,* Elsevier Scientific Publishing Company, New York, 1978.

[4] McKenna, G. B., Leterrier, Y. and Schultheisz, C. R., "The Evolution of Material Properties During Physical Aging," *Use of Plastics and Plastic Composites: Materials and Mechanics Issues,* MD -46, Y. K. Stokes Eds., The American Society of Mechanical Engineers, 1993,

[5] Hastie, R. L. and Morris, D. H., "The Effect of Physical Aging on the Creep Response of a Thermoplastic Composite," *High Temperature and Environmental Effects in Polymer Matrix Composites,* ASTM STP 1174, C. Harris and T. Gates Eds., American Society for Testing and Materials, Philadelphia 1992, pp. 163-185.

[6] Sullivan, J. L., "Creep and Physical Aging of Composites," *Composites Science and Technology,* Vol. 39, 1990, pp. 207-232.

[7] Wang, J. Z., Parvatareddy, H., Chang, T., Iyengar, N., Dillard, D. A. and Reifsnider, K. L., "Physical Aging Behavior of High-Performance Composites," *Composites Science and Technology,* Vol. 54, 1995, pp. 405-415.

[8] Brinson, L. C. and Gates, T. S., "Effects of Physical Aging on Long Term Creep of Polymers and Polymer Matrix Composites," *International Journal of Solids and Structures,* Vol. 32, 6/7, 1995, pp. 827-846.

[9] Gates, T. S. and Feldman, M., "The Effects of Physical Aging at Elevated Temperatues on the Viscoelastic Creep of IM7/K3B," NASA TM-109114, June, 1994.

[10] Veazie, D. R. and Gates, T. S., "Physical Aging Effects on the Compressive Linear Viscoelastic Creep of IM7/K3B Composite," NASA, TM - 110224, December, 1995.

[11] Jones, R. M., *Mechanics of Composite Materials,* Scripta Book Company, Washington, D.C., 1975.

[12] Findley, W. N., Lai, J. S. and Onaran, K., *Creep and Relaxation of Nonlinear Viscoelastic Materials,* North-Holland Publishing Company, Toronto, 1976.

[13] Berg, J. S. and Adams, D. F., "An Evaluation of Composite Material Compression Test Methods," *Journal of Composites Technology & Research,* Vol. 11, No. 2, 1989, pp. 41-46.

[14] Schoeppner, G. A. and Sierakowski, R. L., "A Review of Compression Test Methods for Organic Matrix Composites," *Journal of Composites Technology & Research,* Vol. 12, No. 1, 1990, pp. 3-12.

[15] Tuttle, M. E. and Graesser, D. L., "Compression Creep of Graphite/Epoxy Laminates Monitored Using Moire Interferometry," *Optics and Lasers in Engineering,* Vol. 90, 1990, pp. 151-171.

[16] Irion, M. N. and Adams, D. F., "Compression Creep Testing of Unidirectional Composite Materials," *Composites,* 1981, pp. 117-123.

13

[17] Nguyen, D. H. and Ogale, A. A., "Physical Aging of Polyetheretherketone: Characterized by Creep Tests in Compressive and Flexural Modes," *Journal of Thermoplastic Composite Materials*, Vol. 4, January, 1991, pp. 72-82.

[18] Murry, W. M., and Miller, W. R., *The Bonded Electrical Resistance Strain Gage*, Oxford University Press, New York, 1992.

[19] Allen, D. H., Zocher, M. A. and Groves, S. E., "Apparent Physical Aging of Polymeric Composites," ASME Winter Annual Meeting, November, 1995,

[20] Nguyen, D. H. and Ogale, A. A., "Compressive and Flexural Creep Behavior of Carbon Fiber/PEEK Composites," *Journal of Thermoplastic Composite Materials*, Vol. 4, January, 1991, pp. 83-99.

[21] Santore, M. M., Duran, R. S. and McKenna, G. B., "Volume Recovery in Epoxy Glasses Subjected to Torsional Deformations: the question of rejuvenation," *Polymer*, Vol. 32(13), 1991, pp. 2377-2381.

Table 1. Momentary master curve parameters for both the
transverse and shear creep compliance.

Compliance Terms	T (°C)	S^o (1/GPa)	τ (sec.)	β	μ	$\Delta\mu$ (Std. Dev.)
Tension						
S_{22}	200	0.133	1.56E+5	0.423	0.864	0.139
S_{22}	208	0.130	1.23E+5	0.366	0.927	0.016
S_{22}	215	0.127	7.69E+4	0.315	0.999	0.030
S_{22}	225	0.134	1.76E+4	0.297	0.876	0.092
S_{22}	230	0.118	2.83E+3	0.231	0.482	0.068
S_{66}	200	0.238	5.56E+4	0.383	1.051	0.030
S_{66}	208	0.236	2.89E+4	0.362	1.070	0.030
S_{66}	215	0.239	9.19E+3	0.382	1.017	0.056
S_{66}	225	0.227	2.40E+3	0.301	0.928	0.082
Compression						
S_{22}	208	0.0937	7.76E+4	0.468	0.808	0.0827
S_{22}	215	0.0933	2.43E+4	0.559	0.742	0.0327
S_{22}	220	0.0914	1.53E+4	0.470	0.751	0.0370
S_{22}	225	0.0923	3378.87	0.316	0.644	0.0579
S_{22}	230	0.0589	43.617	0.155	0.587	0.0155
S_{66}	208	0.1521	2.21E+4	0.433	0.871	0.0089
S_{66}	215	0.1452	1.39E+4	0.368	0.853	0.0071
S_{66}	220	0.1402	4595.7	0.356	0.791	0.0510
S_{66}	225	0.1297	485.60	0.252	0.574	0.0603
S_{66}	225	0.1297	485.60	0.252	0.574	0.0603
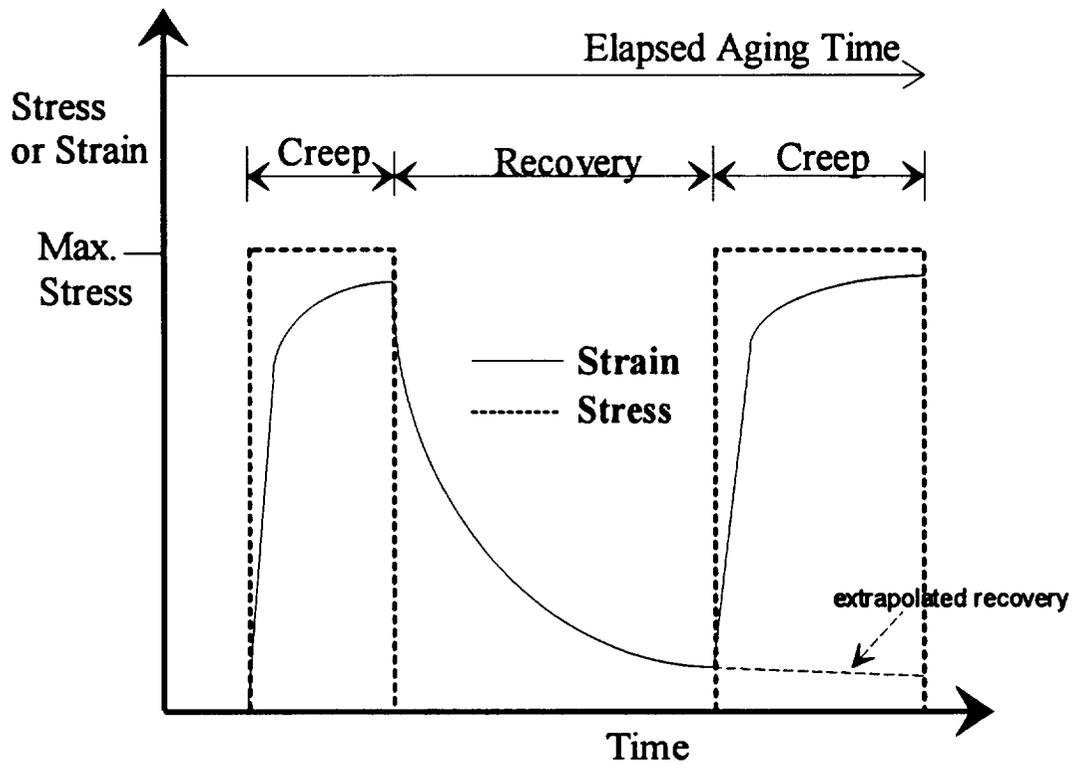
Fig. 1. Sequenced creep compliance test procedures.
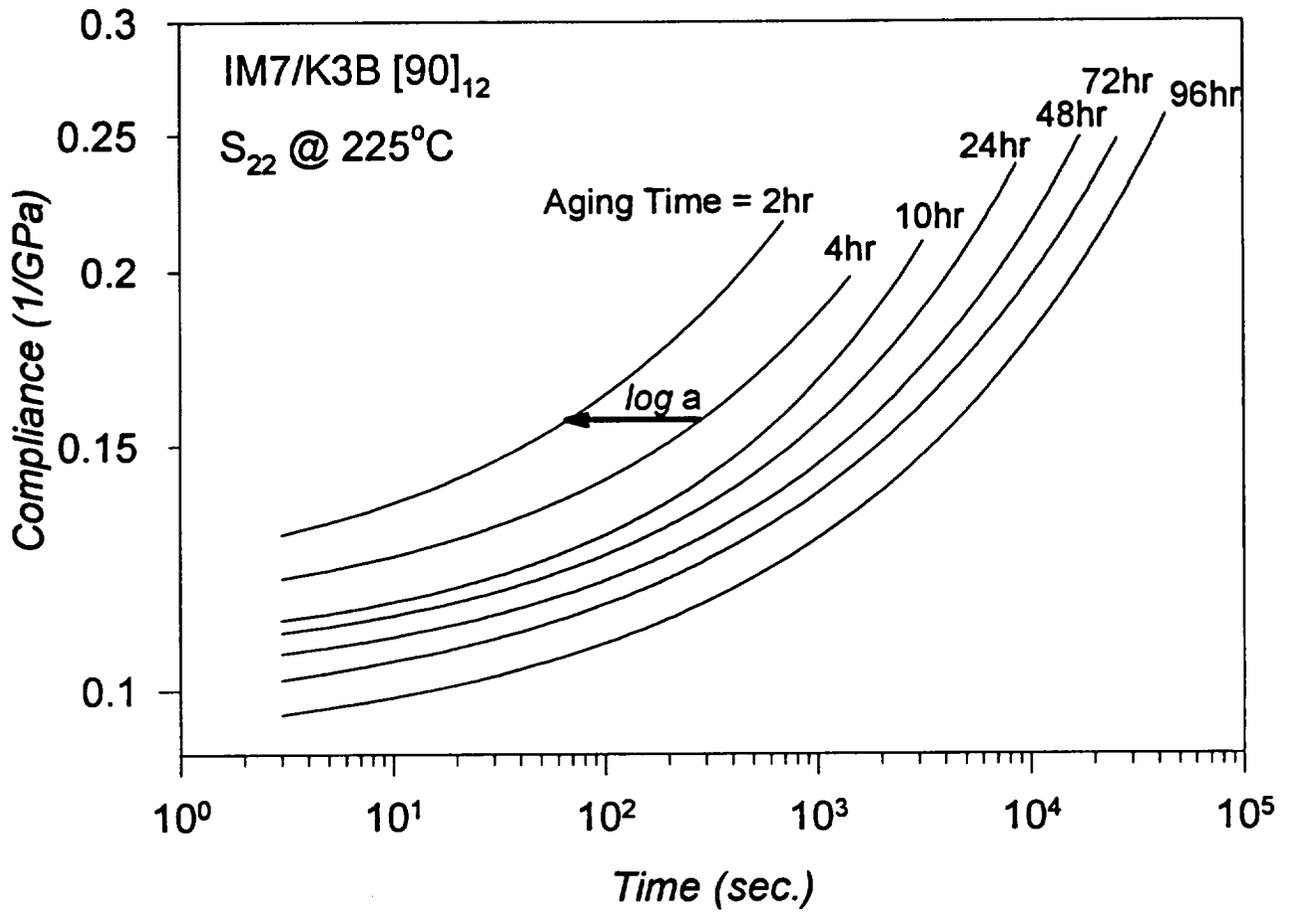
Fig. 2. Typical transverse compression creep compliance momentary curves.
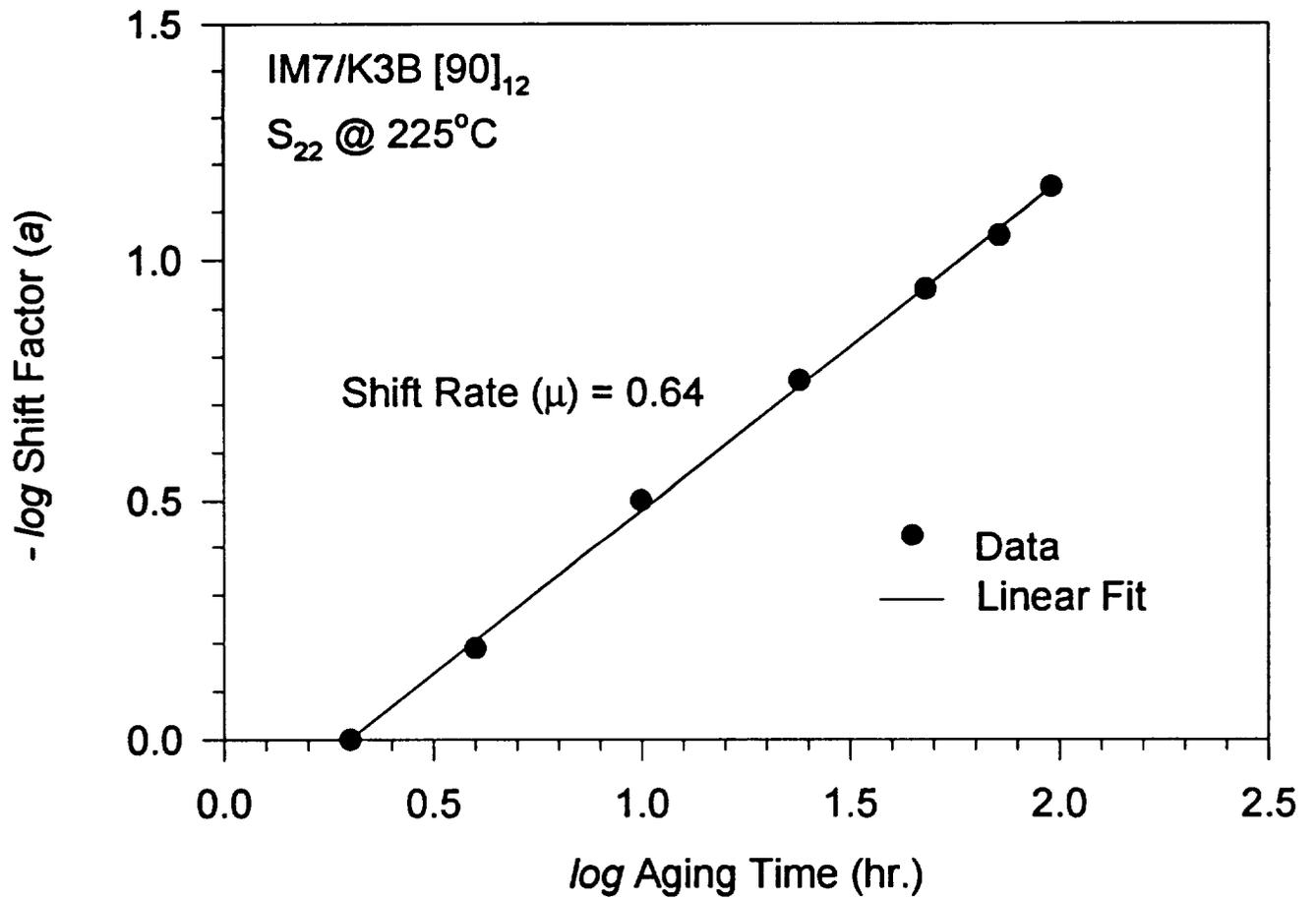
Fig. 3. Typical compression aging shift factor as a function of aging time.
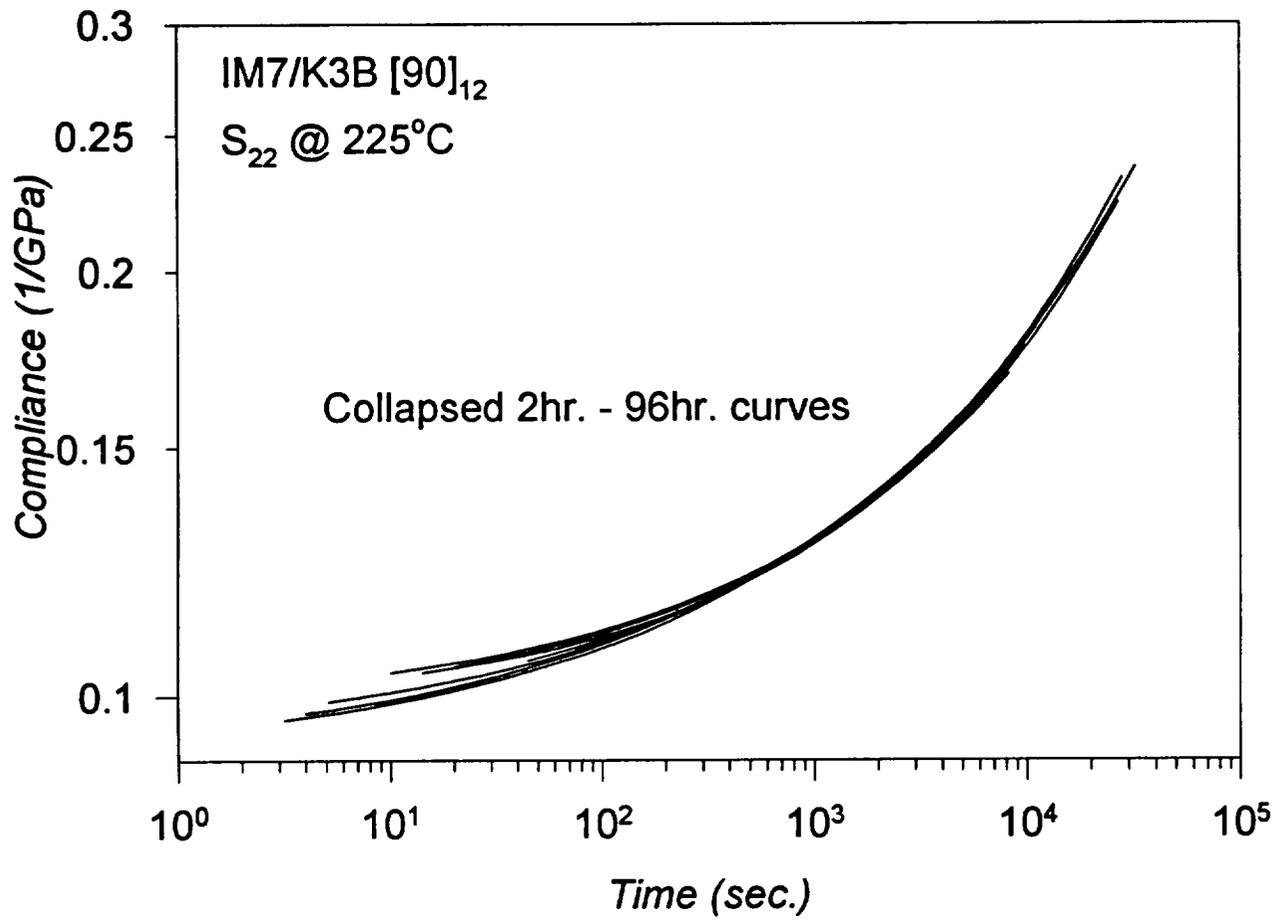
Fig. 4. Typical momentary compression creep compliance during aging.
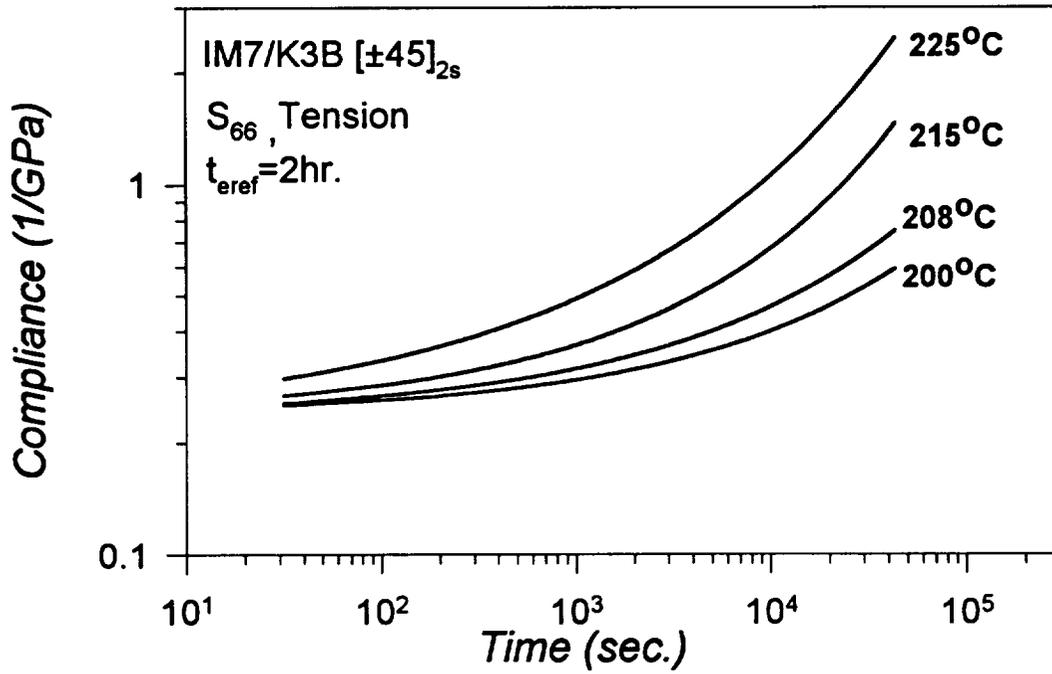
(a) Shear mode master curves.
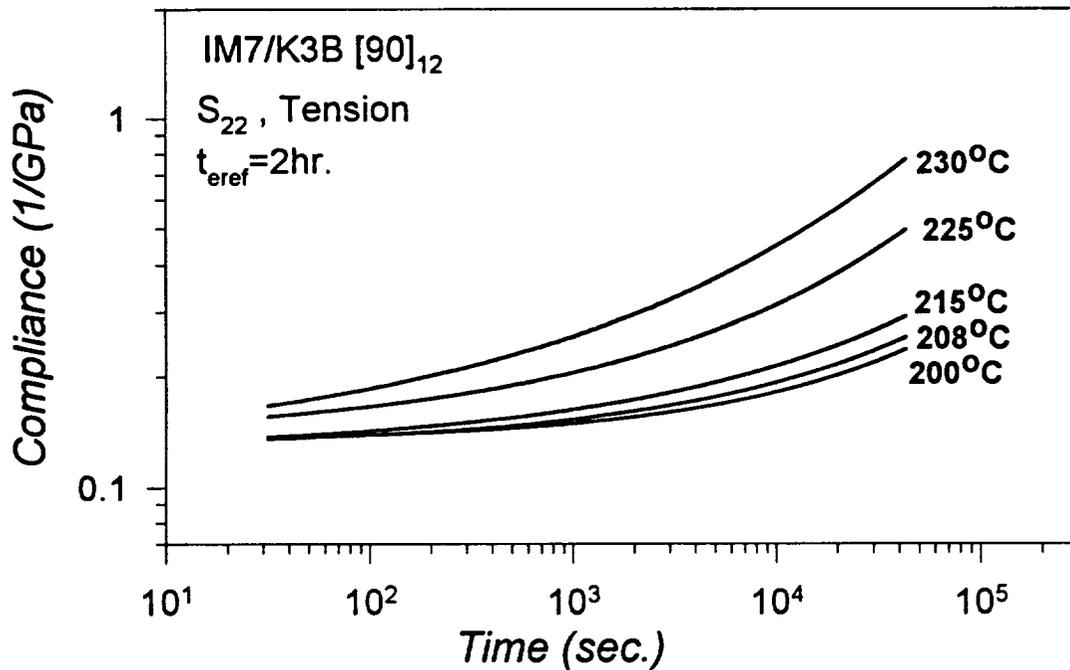
(b) Transverse mode master curves.

Fig. 5. Isothermal momentary master curves for tension loading.
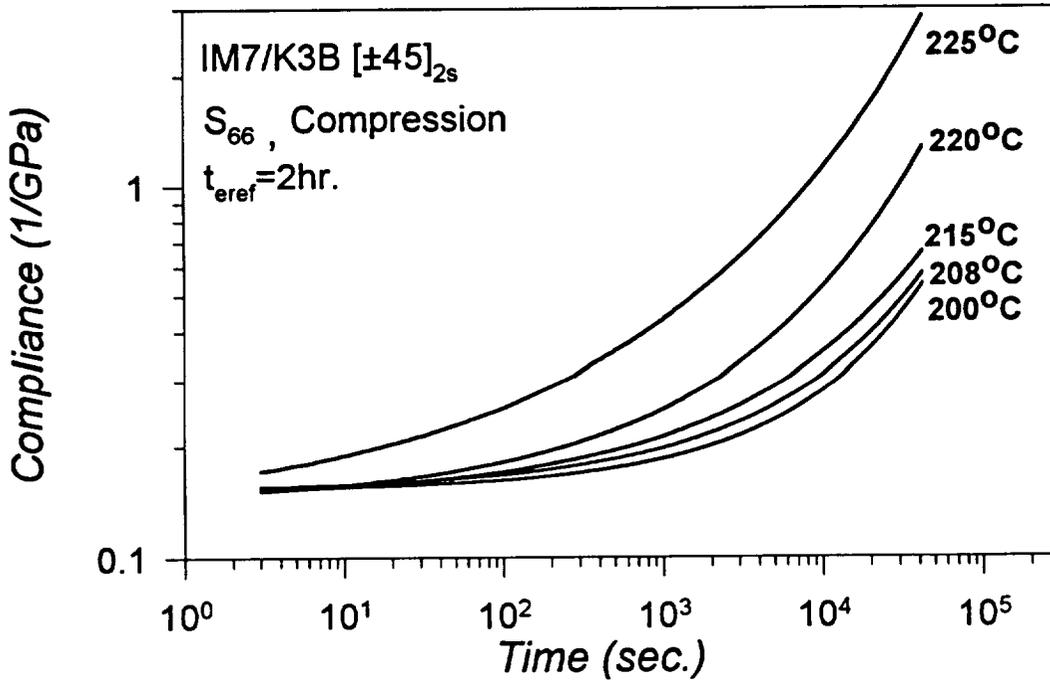
(a) Shear mode master curves
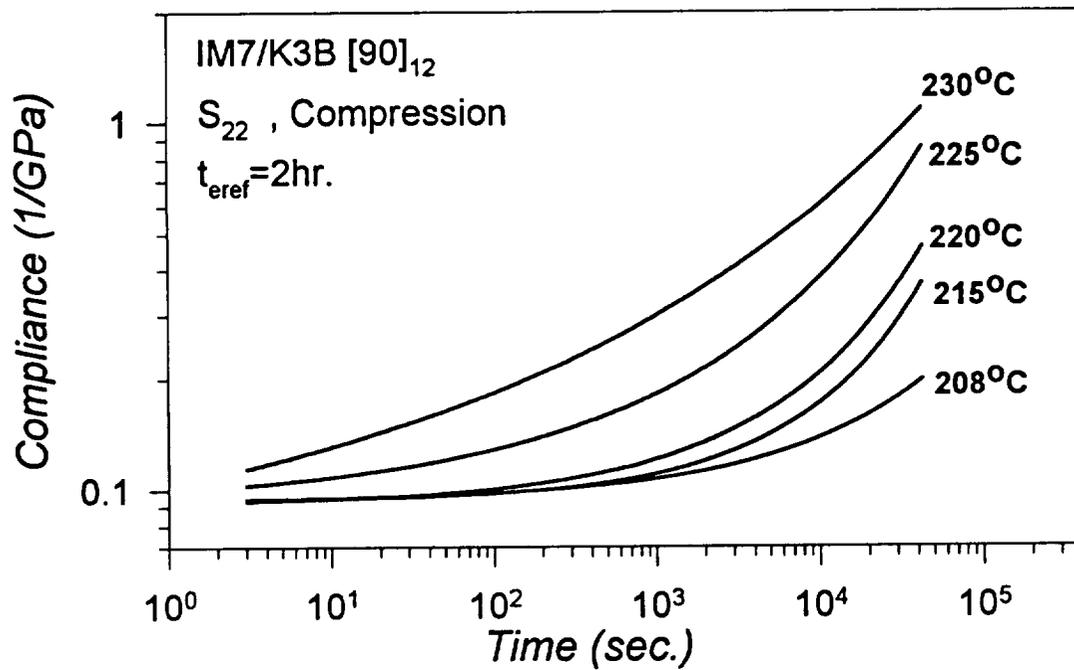
(b) Transverse mode master curves

Fig. 6. Isothermal momentary master curves for the compression loading.

21
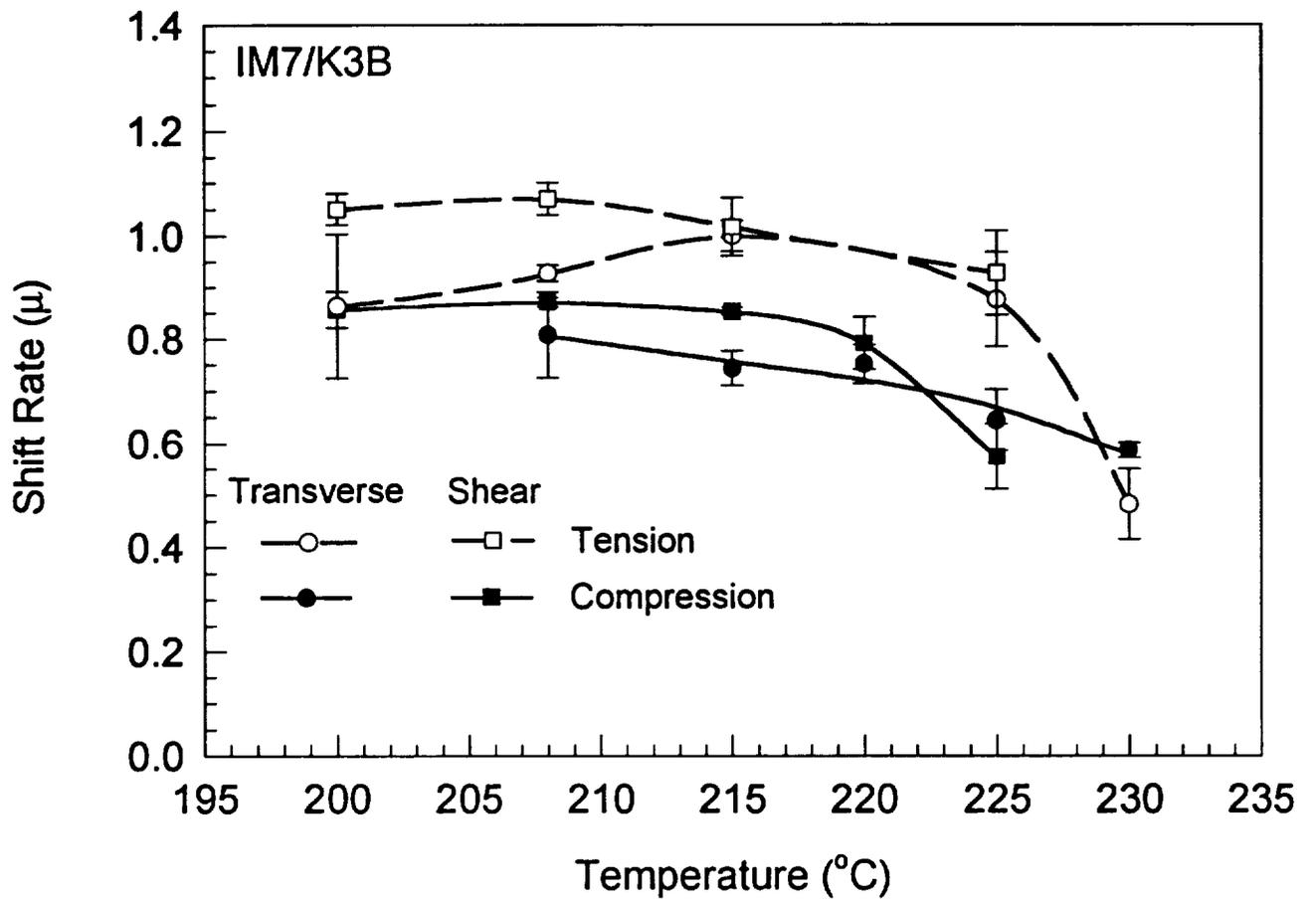
Fig. 7. Transverse and shear shift rates as a function of temperature.
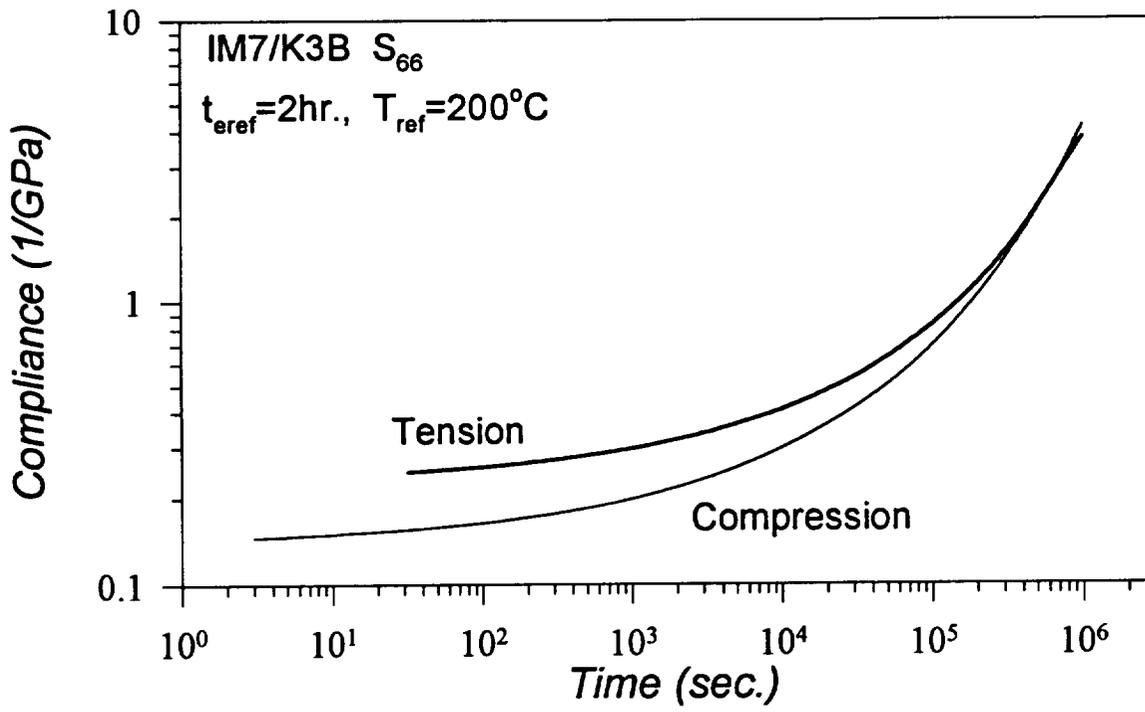
(a) Shear creep compliance TTSP master curve.
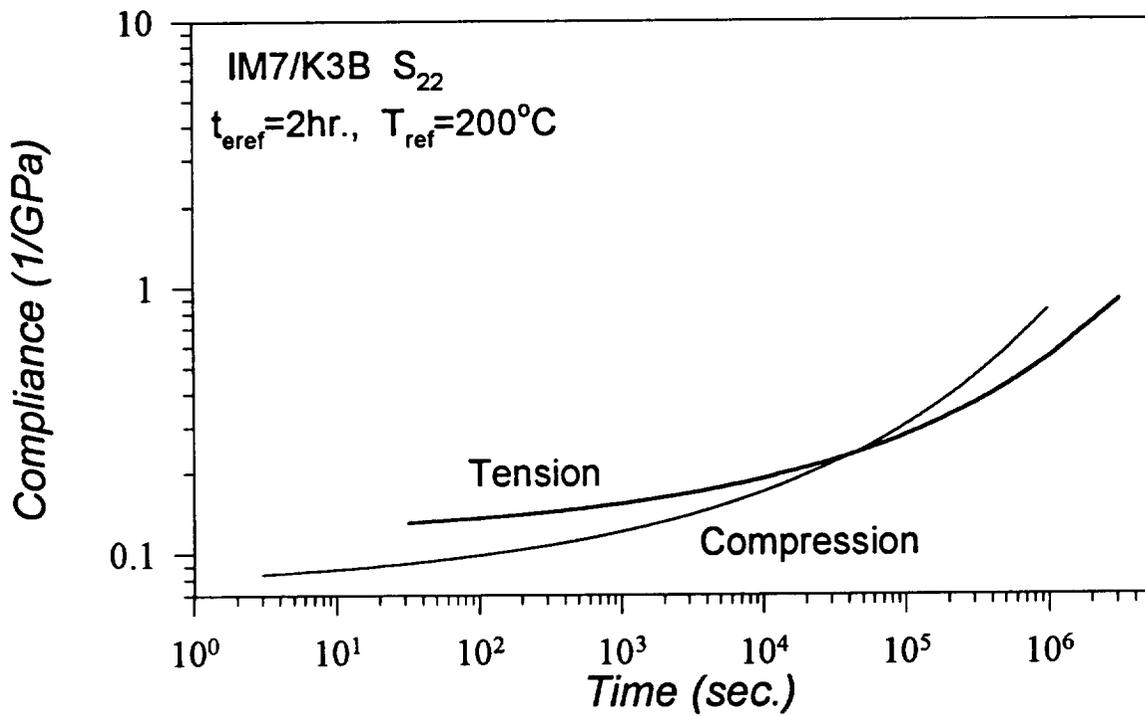
(b) Transverse creep compliance TTSP master curve.

Fig. 8. Time-temperature superposition master curves for the transverse and shear modes.

23
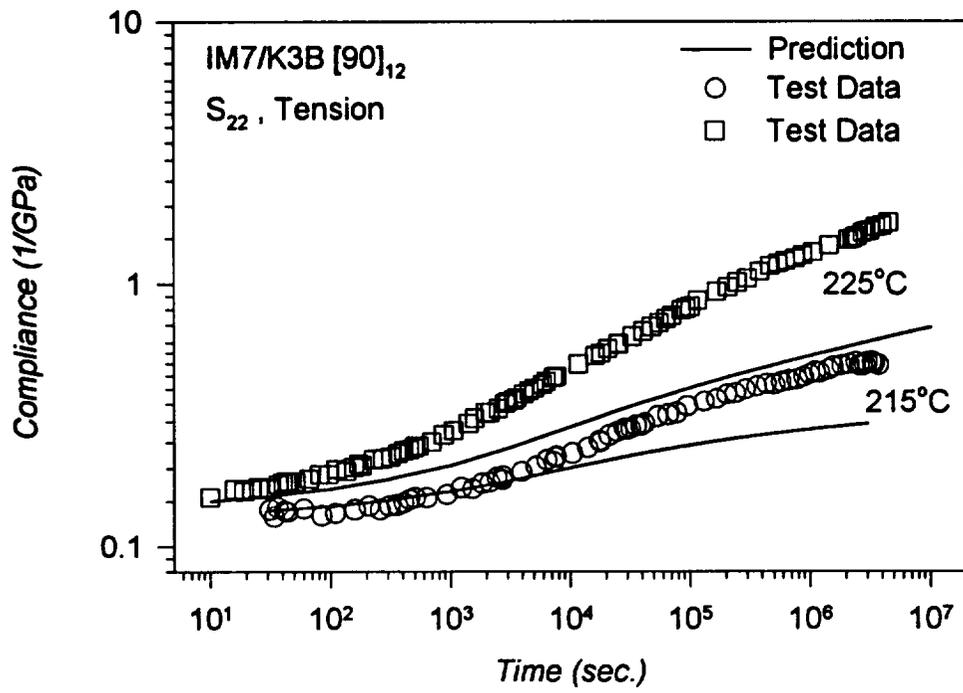
Fig. 9. Test versus prediction for long-term, transverse tension creep tests.
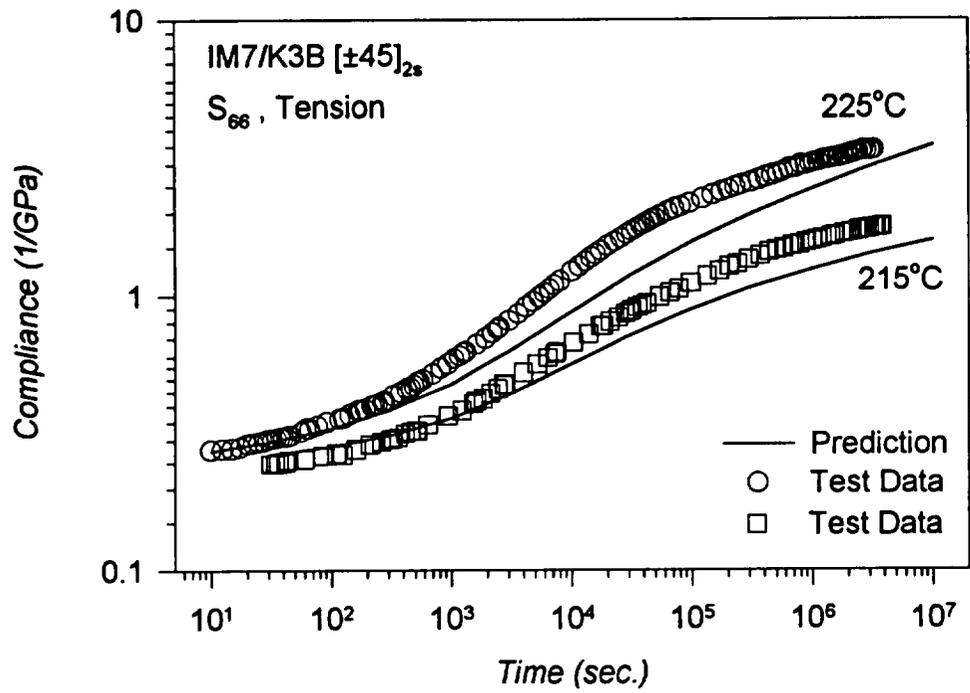
Fig. 10. Test versus prediction for long-term, shear, tension creep tests.
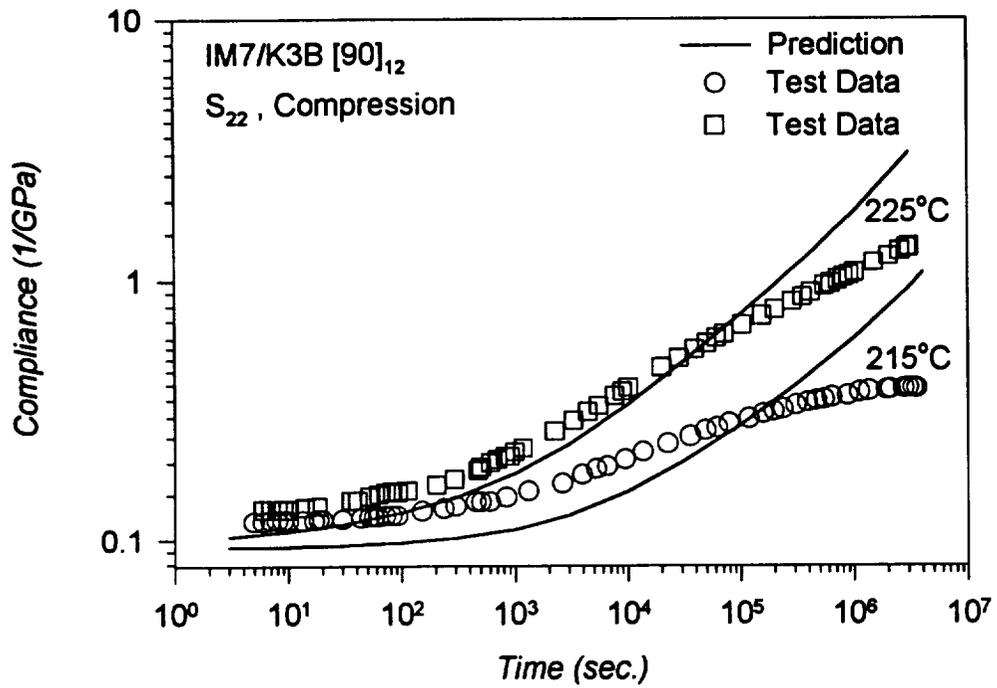
Fig. 11. Test versus prediction for long-term, transverse compression creep tests.
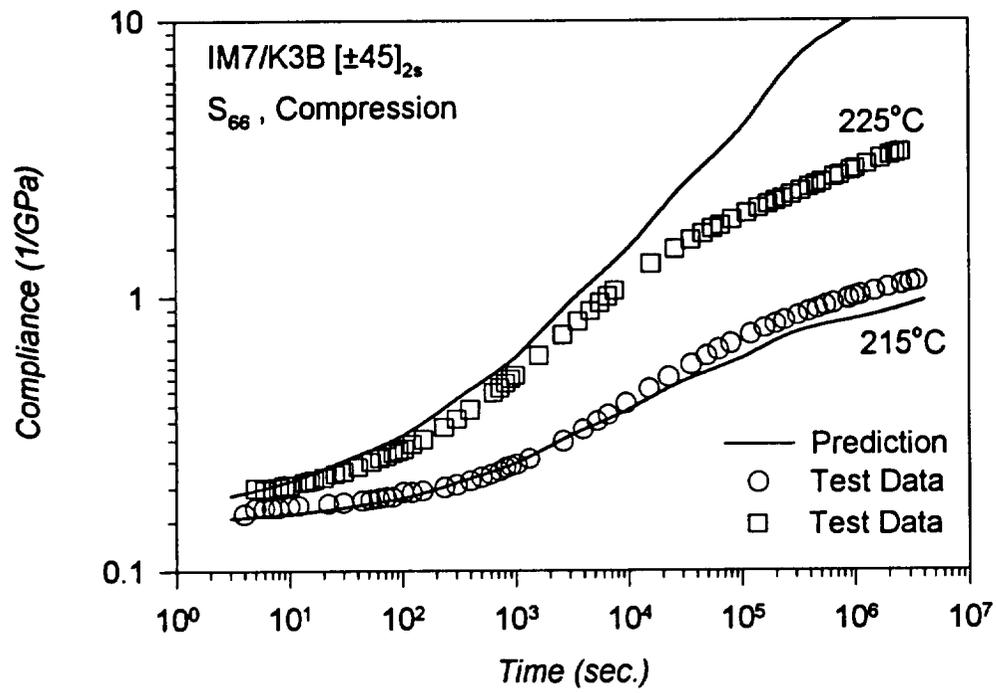
Fig. 12. Test versus prediction for long-term, shear compression creep tests.
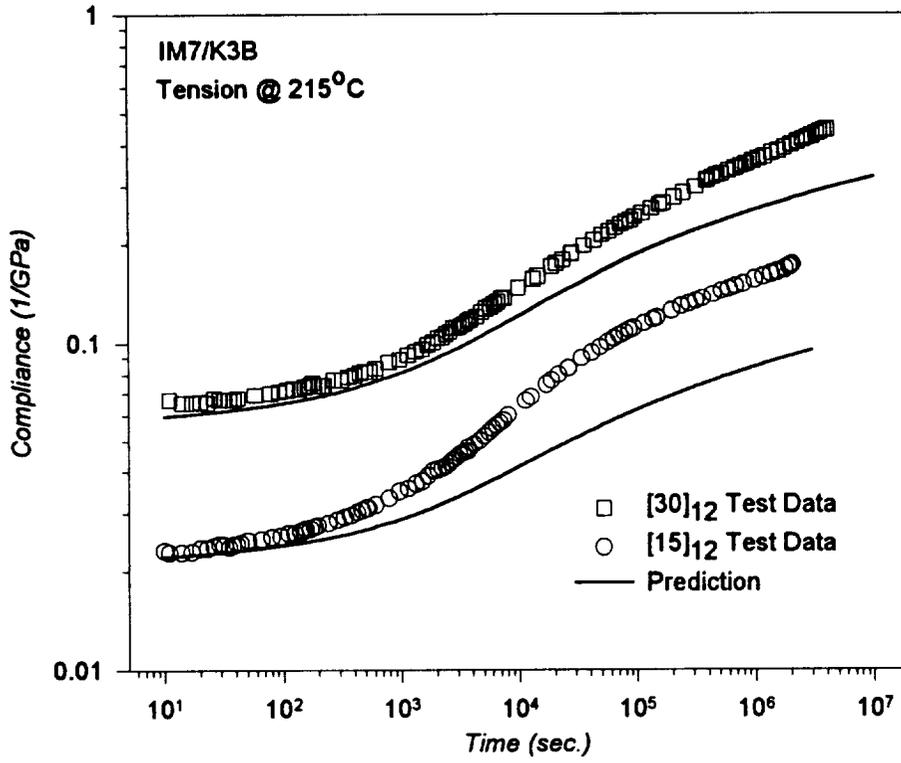
Fig. 13. Test versus prediction for long-term, off-axis tension creep tests.
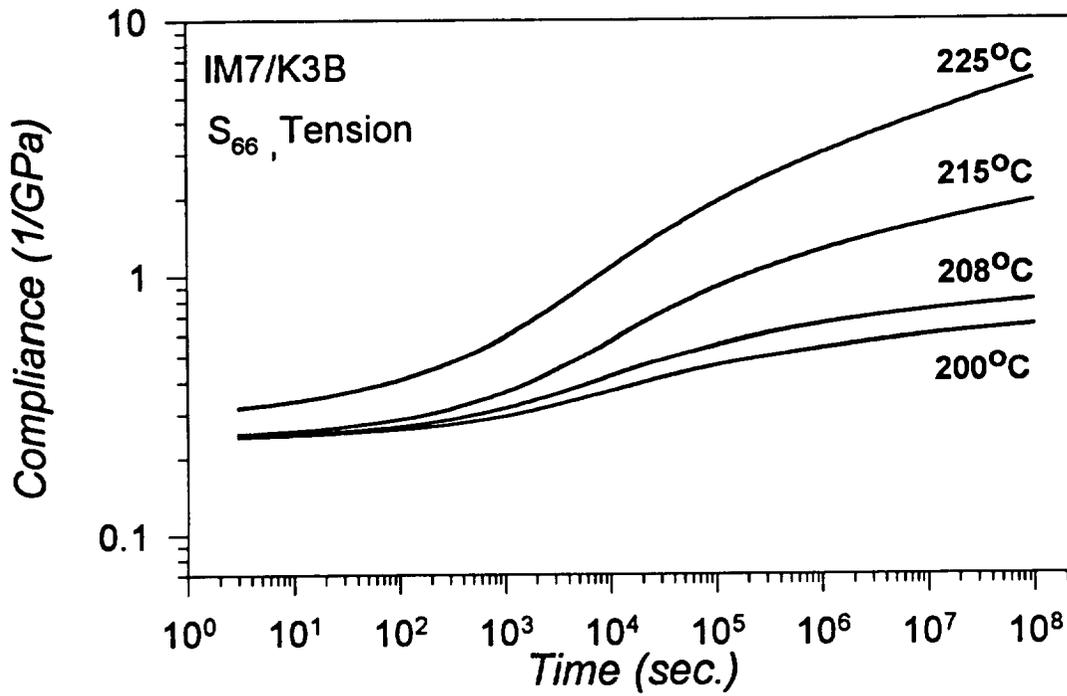
(a) Shear mode, tension loading.
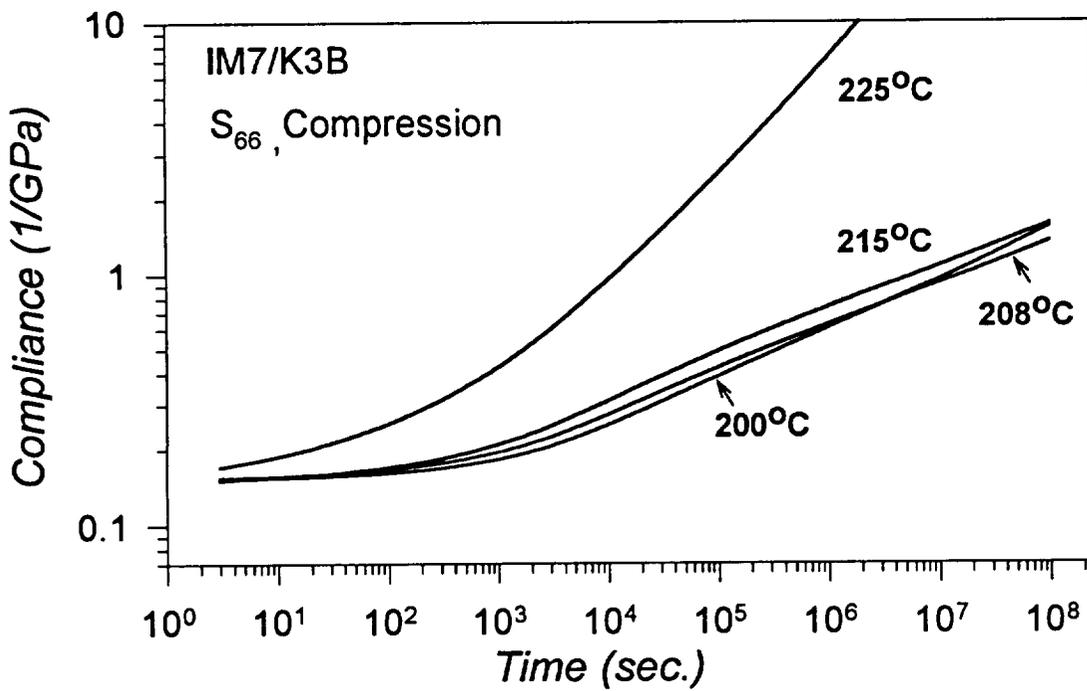
(b) Shear mode, compression loading.

Fig. 14. Predicted long-term shear creep compliance for tension and compression.
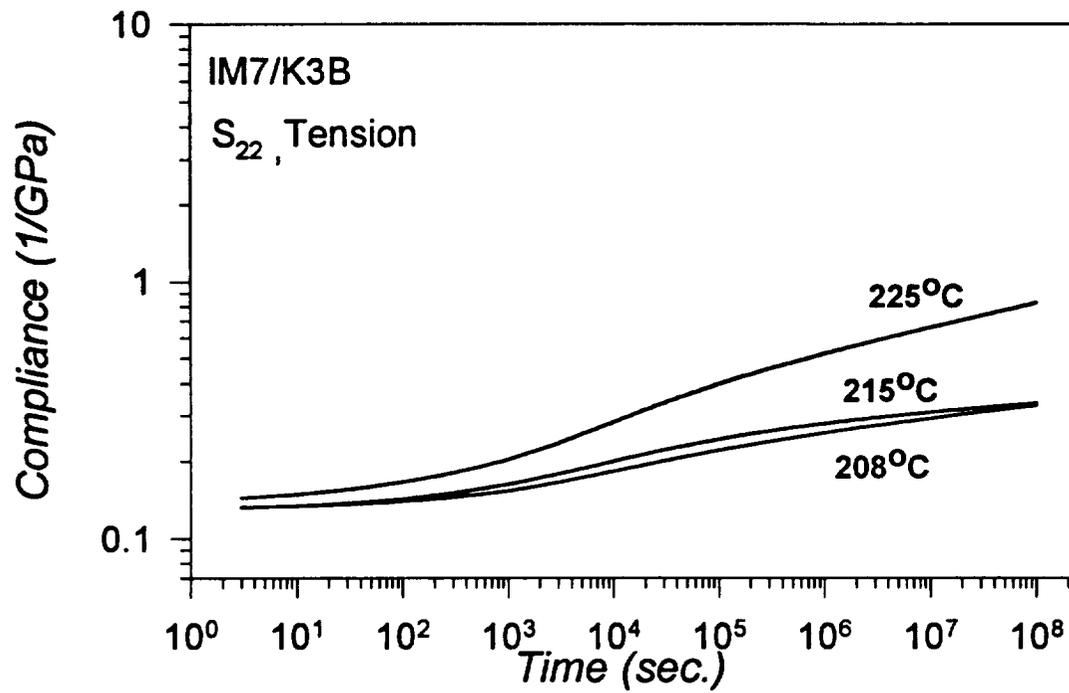
(a) Transverse mode, tension loading.
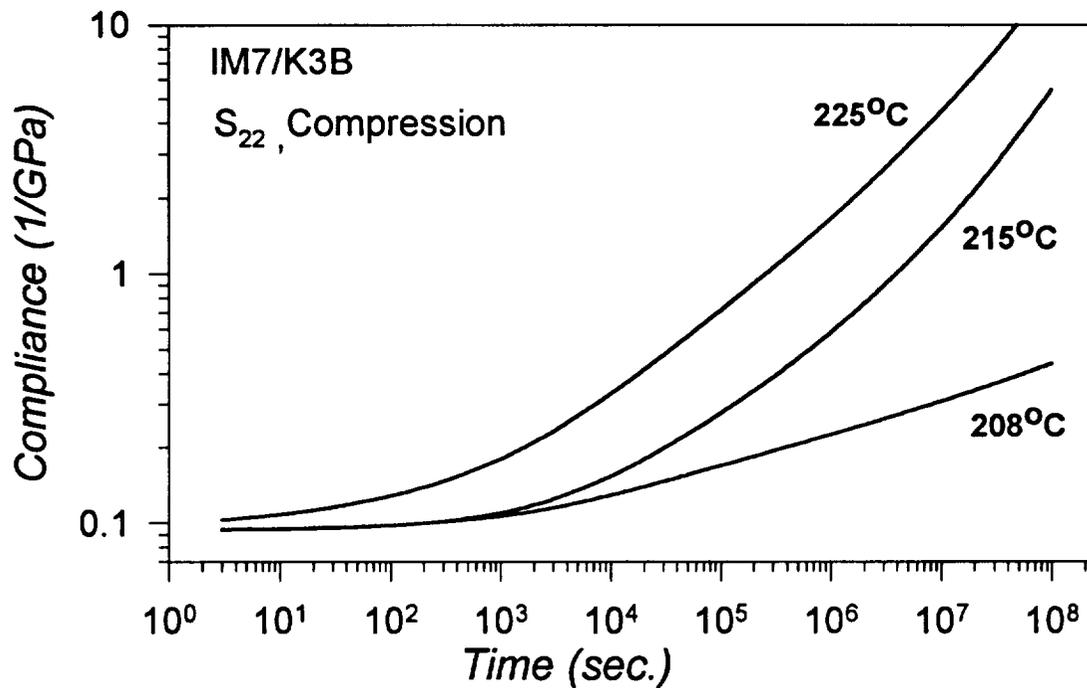
(b) Transverse mode, compression loading.

Fig. 15. Predicted transverse creep compliance for both tension and compression.
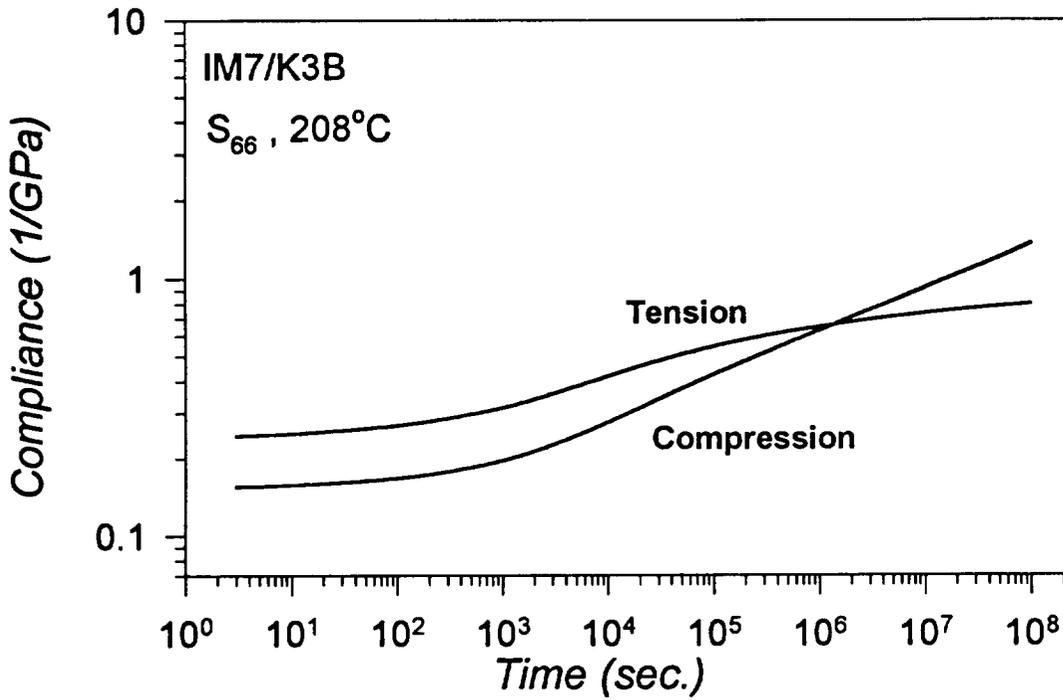
(a) Shear mode compliance.
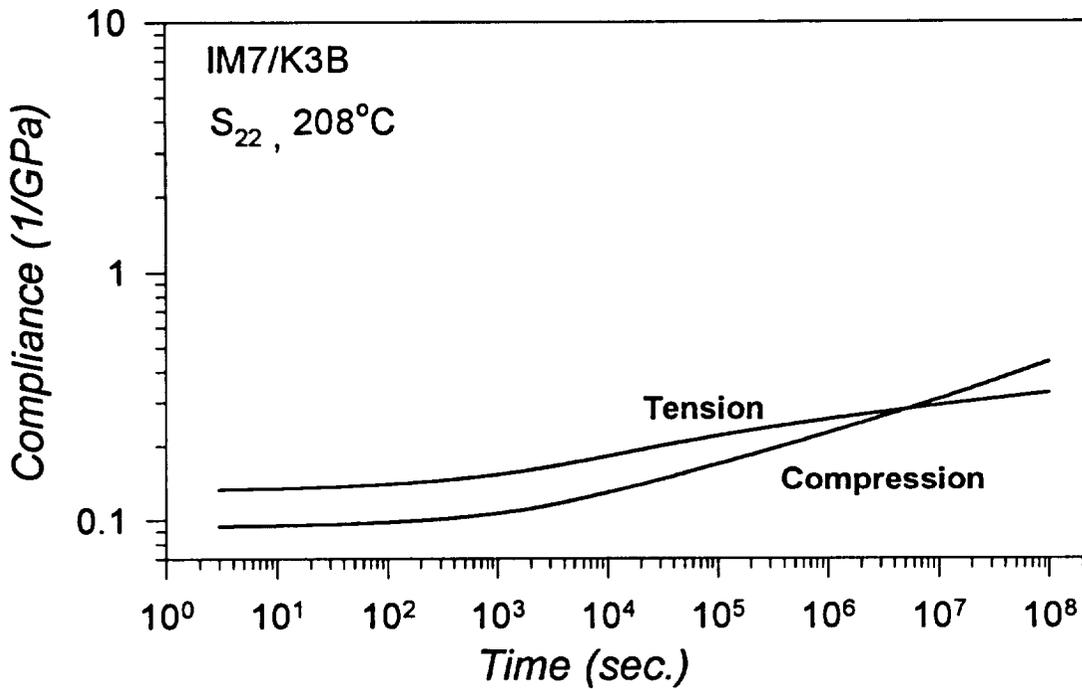
(b) Transverse mode compliance.

Fig. 16. Predictions of long-term creep compliance at 208°C showing differences between tension and compression.
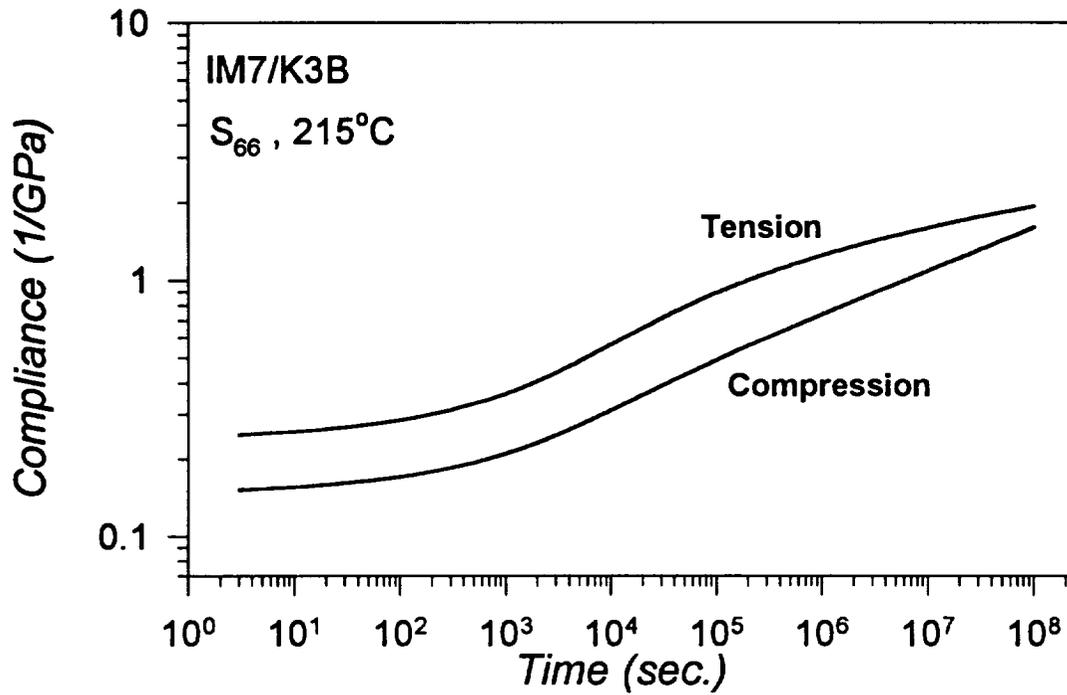
(a) Shear mode compliance.
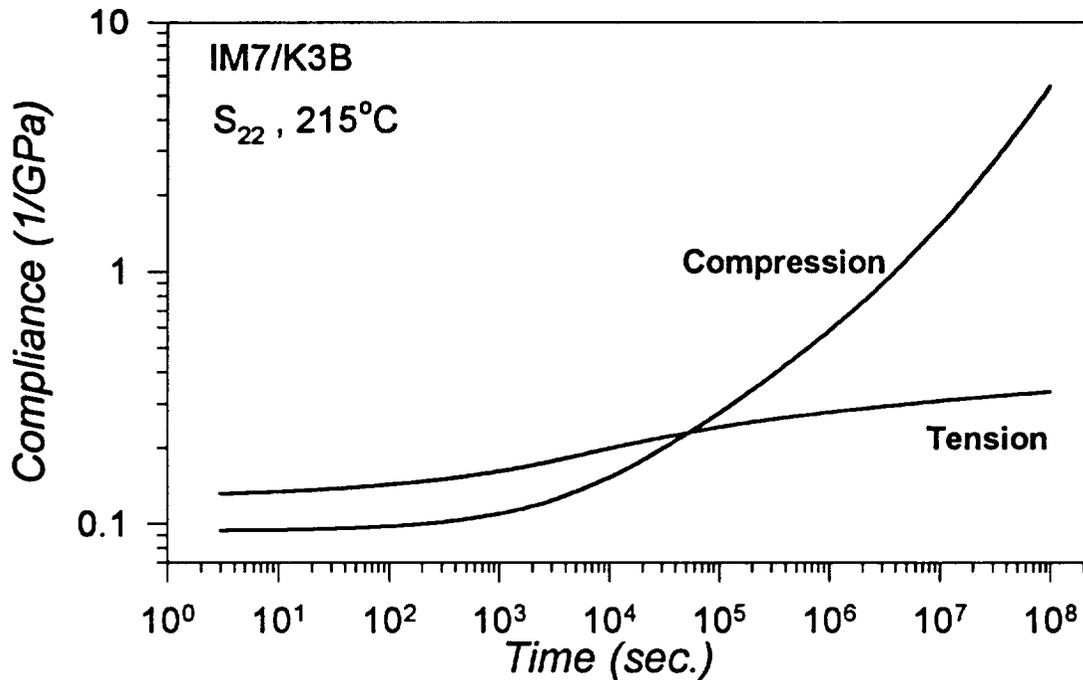
(b) Transverse mode compliance.

Fig. 17. Predictions of long-term creep compliance at $215^{\circ}C$ showing differences between tension and compression.
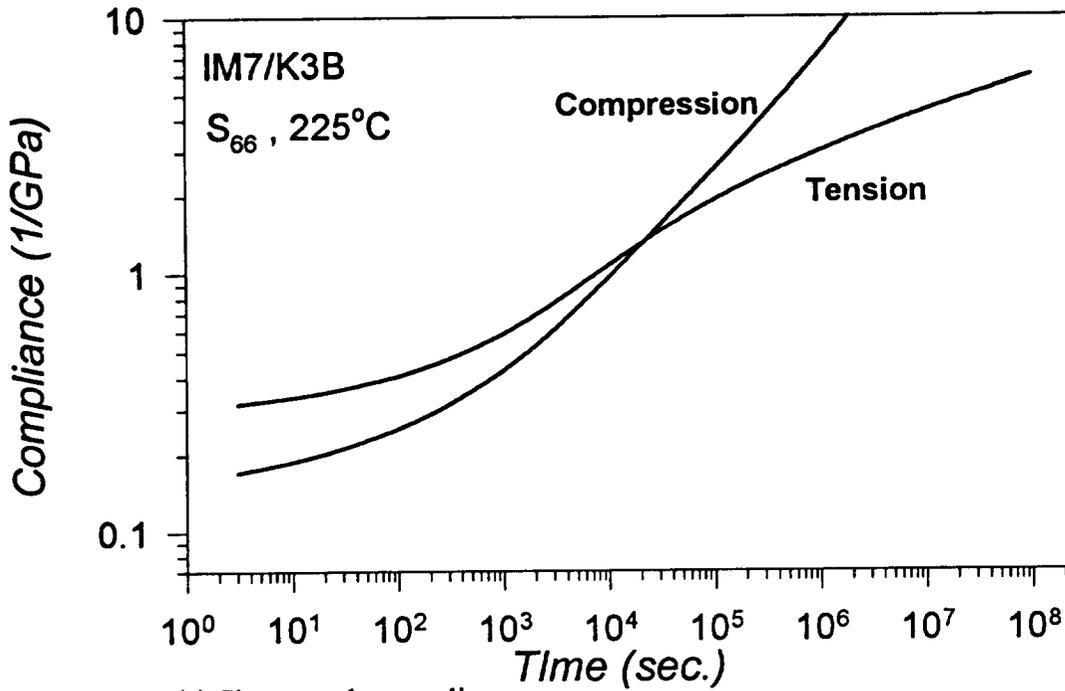
(a) Shear mode compliance.
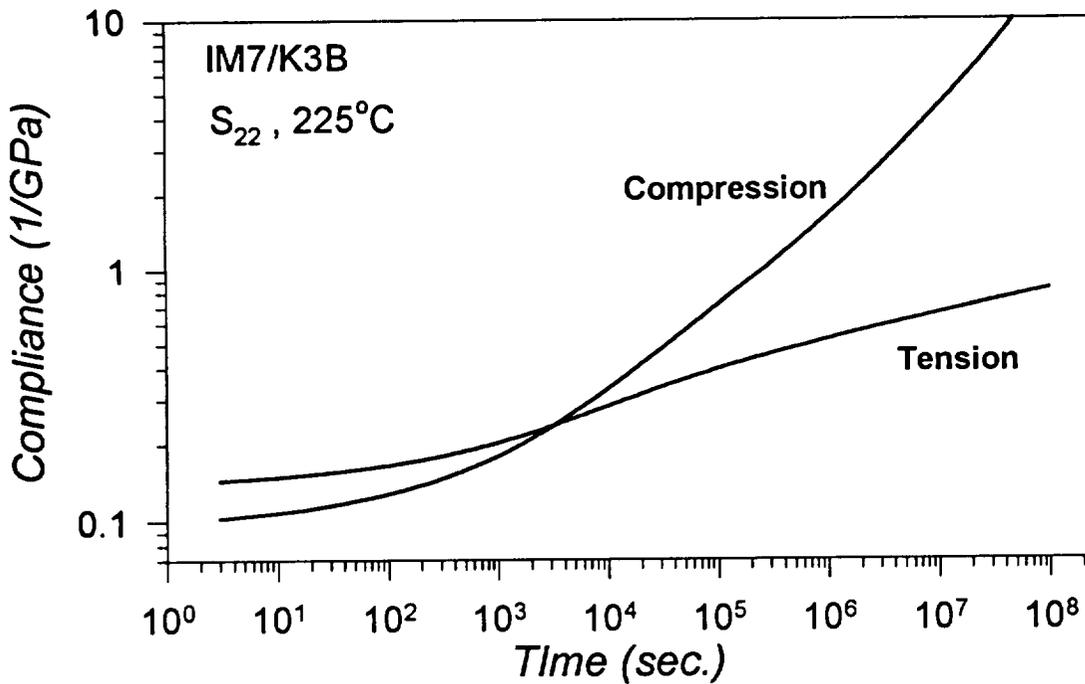
(b) Transverse mode compliance.

Fig. 18. Predictions of long-term creep compliance at 225°C
showing differences between tension and compression.

REPORT DOCUMENTATION PAGE

Public reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden, to Washington Headquarters Services, Directorate for Information Operations and Reports, 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302, and to the Office of Management and Budget, Paperwork Reduction Project (0704-0188), Washington, DC 20503.

1. AGENCY USE ONLY (Leave blank)	2. REPORT DATE August 1996	3. REPORT TYPE AND DATES COVERED Technical Memorandum

4. TITLE AND SUBTITLE

A Comparison of Tension and Compression Creep in a Polymeric Composite and the Effects of Physical Aging on Creep Behavior

5. FUNDING NUMBERS

WU 505-63-50-18

6. AUTHOR(S)

Thomas S. Gates, David R. Veazie, and L. Catherine Brinson

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)

NASA Langley Research Center
Hampton, VA 23681-0001

8. PERFORMING ORGANIZATION REPORT NUMBER

9. SPONSORING / MONITORING AGENCY NAME(S) AND ADDRESS(ES)

National Aeronautics and Space Administration
Washington, DC 20546-0001

10. SPONSORING / MONITORING AGENCY REPORT NUMBER

NASA TM-110273

11. SUPPLEMENTARY NOTES

Gates: Langley Research Center, Hampton, VA; Veazie: Clark Atlanta University, Atlanta, GA; Brinson: Northwestern University.

12a. DISTRIBUTION / AVAILABILITY STATEMENT

Unclassified - Unlimited

Subject Category - 24

12b. DISTRIBUTION CODE

13. ABSTRACT (Maximum 200 words)

Experimental and analytical methods were used to investigate the similarities and differences of the effects of physical aging on creep compliance of IM7/K3B composite loaded in tension and compression. Two matrix dominated loading modes, shear and transverse, were investigated for two load cases, tension and compression. The tests, run over a range of sub-glass transition temperatures, provided material constants, material master curves and aging related parameters.

Comparing results from the short-term data indicated that although trends in the data with respect to aging time and aging temperature are similar, differences exist due to load direction and mode. The analytical model used for predicting long-term behavior using short-term data as input worked equally as well for the tension or compression loaded cases. Comparison of the loading modes indicated that the predictive model provided more accurate long term predictions for the shear mode as compared to the transverse mode. Parametric studies showed the usefulness of the predictive model as a tool for investigating long-term performance and compliance acceleration due to temperature.

14. SUBJECT TERMS

Composites; Viscoelasticity; Physical aging; Elevated temperature; Compression; Creep testing

15. NUMBER OF PAGES

34

16. PRICE CODE

A03

17. SECURITY CLASSIFICATION OF REPORT Unclassified	18. SECURITY CLASSIFICATION OF THIS PAGE Unclassified	19. SECURITY CLASSIFICATION OF ABSTRACT	20. LIMITATION OF ABSTRACT