

The Influence of Fiber-Matrix Adhesion on the Linear Viscoelastic Creep Behavior of CF/PPS Composites

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Abstract: The influence of fiber-matrix adhesion on the linear viscoelastic creep behavior of as received and surface modified carbon fiber (AR-CF and SM-CF, respectively) reinforced polyphenylene sulfide (PPS) composite materials was investigated. Short-term tensile creep tests were performed on $\pm 45^\circ$ specimens under four different isothermal condition; 70, 80, 90 and 100°C. As a preliminary step to obtain the time-temperature master curve, as well as to characterize the changes in matrix dominated properties over storage time, physical aging effects were evaluated on both systems using the short-term test method established by Struik. The results showed that the surface treatment carried out in the SM-CF improved fiber-matrix adhesion, enhancing the mechanical performance of CF/PPS composites but with minor effects on the creep response. Increasing retardation times with physical aging was observed in all test conditions. Compared to temperature effects, physical aging showed to have a small contribution on the creep behavior of CF/PPS composites.

Keywords: Viscoelastic Creep Behavior; Physical Aging; CF/PPS Composites, Time Aging-Time Superposition; Time Temperature Superposition Principle (TTSP).

1. Introduction

Polymer matrix composites (PMCs) have long found utility in many engineering applications, where their specific characteristics such as, low cost, easy processing, corrosion resistance as well as enhanced strength-to-weight ratios, have enabled these materials to be used in place of traditional metals. In most cases, epoxy thermoset resins are the polymeric matrix constituent in use. Nevertheless, a growing trend has been the replacement of thermoset matrices with thermoplastics.

One thermoplastic polymer that is a potential substitute for epoxy thermoset resins is polyphenylene sulfide (PPS). However, the use of this polymeric material for such purposes still requires a greater understanding about its behavior under long-term stress/strain. PMCs should hold their mechanical performance throughout their designed life time.

Due to its polymeric matrix constituent, PMCs are viscoelastic in nature and, therefore, their time-dependent creep behavior can be significantly influenced by the exposure to extreme-use environmental conditions (i.e., pressure, temperature, moisture and chemicals), mechanical loads, or a combination of both [1]. Thus, the study and understanding of the long-term exposure on the time-dependent viscoelastic behavior of PMCs is extremely important to their proper design and safe operation.

As most PMCs are designed for long-term service lives, typically up to 20 years, measuring their viscoelastic responses over their entire lifetimes is fairly impractical. As a consequence, many studies have been conducted and published on accelerated test methods to characterize the viscoelastic behavior in PMCs [2-4]. Accelerated test methods use short-term creep data and corresponding models to predict the long-term creep behavior of the material. A successful example of these models is the Time Temperature Superposition Principle (TTSP).

Improvement on the fiber-matrix adhesion of high performance thermoplastic composite has been proven to be the key to increase the performance to cost ratio [5-7]. Therefore, the development of optimized pretreatments and sizings has become one of the main focus of several fibers manufactures around the world. Not much attention, however, has been received on the influence of the fiber-matrix adhesion on the viscoelastic creep behavior of composites.

The goal of this research is to investigate the linear viscoelastic creep behavior of as received and surface modified carbon fiber (AR-CF and SM-CF, respectively) reinforced polyphenylene sulfide (PPS) using elevated temperature as the accelerator of the viscoelastic response. Increased understanding about the relation between fiber-matrix adhesion and the creep response is gained by comparing creep compliance for AR-CF/PPS and SM-CF/PPS. Since creep is a phenomenon mainly dominated by the polymeric matrix (creep in fibers is considered to be negligible) [2, 8], tensile tests of $\pm 45^\circ$ specimens (in-plane shear response) were chosen for this studies. As a preliminary step to obtain the time-temperature master curve, as well as to characterize the changes in matrix dominated properties over storage time, physical aging studies were also carried out using the short-term test method established by Struik (discussed in the next section) [9].

1.1. Creep

Creep is the slow and progressive time-dependent increase in deformation of a material subjected to a constant load. Although most structures may not fail when subjected to constant load for a relatively short time, they may be susceptible to failure or high level of deformations if the constant load is kept for a longer time. In some cases, even if failure does not happen, at a certain level of creep deformation, the structure is considered to be doomed [2].

Both polymers and metals structures can creep, however, polymers typically undergo significant creep even at room temperatures [10]. For linear viscoelastic materials, the creep strain is normalized with respect to the applied stress, allowing creep data from tests at differing stress levels to be compared. This procedure results in a material property called creep compliance, $S(t)$, given by:

$$S(t) = \frac{\varepsilon(t)}{\sigma_0} \quad (1)$$

where ε is the measured strain signal and σ_0 is the applied stress.

Creep curves depicting compliance as a function of time are the most common way of presenting creep results.

1.2. Time Temperature Superposition Principle (TTSP)

The background theory for TTSP was originally developed for using with pure polymers [8, 9], however several researchers have successfully expanded its use to PMCs [2-4]. According to the TTSP, the processes involved in molecular relaxation or rearrangements in viscoelastic materials occur at accelerated rates at higher temperatures. In addition, it is assumed that there is a direct equivalency between time and temperature. Thus, the time over which these processes occur can be reduced by conducting the measurement at elevated temperatures and shifting the resultant data to lower temperatures by a certain shift factor value, a_T , in order to extend the prediction. The thus shifted time scale is often called reduced time, t_{red} , and can therefore be written as:

$$t_{red} = a_T t \quad (2)$$

where t is the elapsed time of a test and a_T is the shift factor specific to a test.

The result of this shifting is a ‘master curve’ depicting creep compliance versus reduced time (t_{red}). A material for which this shifting along the time axis results in a satisfactory master curve is called a Thermorheological Simple Material (TSM); if the curves do not overlap well by only shifting along time axis, the material is called a Thermorheological Complex Material (TCM) [11].

1.3. Physical Aging

Although the three basic constituents of advanced PMCs are fiber, matrix, and interphase, in general, it is mainly the polymer matrix that causes degradation or changes in durability of PMCs. That can be explained by the simple fact that all polymers undergo physical aging at temperatures below their glass transition temperature, T_g [9]. As a result, all the matrix-dominated properties of PMCs are subject to physical aging for in-service temperature conditions.

Physical aging is the slow evolution of a glassy polymer towards thermodynamic equilibrium by time-dependent changes in volume, enthalpy, and entropy, as well as mechanical properties. Unless the temperature is very close to the T_g , this changing will typically take years to complete. Physical aging distinguishes from others aging phenomenon (chemical aging, damage evolution, etc.) due to thermoreversibility. Heating a polymer to a temperature above T_g ('rejuvenation') erases the effects of physical aging [9-12].

Struik [9] showed that for TSM undergoing isothermal physical aging, the short-term compliance response (S) is related to a momentary compliance reference curve (S_{ref}) by the equation

$$S(t; t_e, T) = S_{ref}(a_e t; t_{e,ref}, T) \quad (3)$$

where $t_{e,ref}$ is the isothermal aging time at which the reference curve was defined, t_e is the aging time at which the short-term test is taking place, T is the common isothermal temperature at which both the short-term test and the reference curve were obtained, and a_e is the shift factor due to physical aging. In isothermal aging tests, the short-term compliance response curves (S) are obtained. The duration of the tests are generally chosen to be small enough so that physical aging is assumed to be negligible during tests. Typically, this is accomplished by limiting the test duration, t , to a time corresponding to $0.1 t_e$ [9] or $0.3 t_e$ [13]. This is called the snapshot condition. Creep testing that respect the snapshot condition are called short-term tests, and the individual curves thus obtained by following through these tests are called momentary curves. If the material is loaded for a time that exceeds the snapshot condition, then physical aging occurs during testing and therefore, tests are called long-term tests.

The collection of momentary curves can be individually shifted by a certain aging shift factors, a_e , to a suitable reference (master) curve (S_{ref}). From these physical aging shift factors we can define the aging shift rate, μ_e , as;

$$\mu_e(T) = -\frac{d \log a_e}{d \log t_e} \quad (4)$$

where μ_e is the aging shift rate at temperature T . The aging shift rate, μ_e , can be considered to be a material constant [9].

Once the μ_e has been determined, the aging master curve constructed at a given reference age can be shifted to any other age (i.e., for which no experimental data is available) according to the equation:

$$a_e = \left(\frac{t_{e,ref}}{t_e} \right)^{\mu_e(T)} \quad (5)$$

The approach above is referred to as time–aging time superposition [4, 8-9] and it can be combined with TTSP to predict the momentary response at one temperature (T) using a reference curve defined at a reference temperature (T_{ref}) as;

$$S(t; t_e, T) = S_{ref}(a_e a_T t; t_{e,ref}, T_{ref}) \quad (6)$$

2. Experimental Procedure

2.1. Test Materials and Specimen Configuration

The two materials system investigated in this study were AR-CF/PPS and SM-CF/PPS composites. The PPS thermoplastic resin was fabricated by Ticona and provided by TenCate Advanced Composites in the form of 80 μm thick film. The nominal PPS melt temperature, T_m , is 280°C. As for the CFs, it was used T300J 40B 3K standard modulus PAN CFs fabricated by Toray and provided by TenCate Advanced Composites in the form of 5-harness satin fabric with two different surface treatments:

- (1) CD0286/0000, as provided by Toray to TenCate Advanced Composites - referred to as 'as received' (AR-CF) in this work;
- (2) CD0286/8212, as resulting from industrial surface treatment carried out by TenCate Advanced Composites - referred to as 'surface modified' (SM-CF) in this work.

Composite panels were produced by hand stacking of the CF and PPS resin film and subsequent thermopressing. The stacking sequences used are $[0,90]_{4s}$, where $[0,90]$ represents one layer of fabric: the layers are stacked so that they are symmetric with respect to the middle plane and that the warp side faces the outside. The T_g of the resultant material was found to be 102°C and is defined as the temperature associated to the maximum of the tan delta peak, measured by dynamic mechanical analyzer (DMA) at 1Hz and 2°C/min.

Test specimens measuring approximately 250 mm long (in the load direction) by 25 mm wide were cut from the 2,5 mm thick laminated panels in an angle of $\pm 45^\circ$ to the fibers using a water cooled diamond saw. This specimen size is in accordance to the ASTM Specification D3518/D3518M. Prior to testing, specimens were dried for at least 24 hours at 60°C in vacuum.

Paper tabs were glued to the specimen surface longitudinal ends in order to avoid slipping. Strain gages type KFG-5-120-C1-23 (by Kyowa) bonded with CC-33A Cyano-Acrylate Base adhesive (by Kyowa) were used for strain measurements. In total, 3 strain gages were used for each tested specimen; 2 strains gages were mounted vertically aligned back-to-back in the center of the specimen, so that any possible buckling of the specimen would be detected; 1 strain gage was mounted longitudinally aligned in the center of the specimen.

In order to separate load induced strain from thermal strain, temperature compensation gauges were mounted on another specimen placed close to the test specimen. This specimen remained unloaded throughout the test. The thermal apparent strain measured in this specimen was then used to correct the measured strain in the loaded specimen.

2.2. Test Equipment

Testing in this study was performed using a 10kN Zwick 1445 tensile tester machine equipped with a convection oven. To provide a better accuracy between the temperature displayed at the oven and the 'real' specimen temperature, a thermocouple was attached to the center region of the tested specimen surface. The temperature of the specimen during each set of aging and creep testing was maintained constant ($\pm 0.5^\circ\text{C}$) throughout the test. After each creep test the specimen were stored for 2 days in a desiccator to prevent moisture absorption, rejuvenated and then subjected to a new set of isothermal physical aging and creep testing at a different temperature.

2.3. Creep Testing

Specimens were loaded at $\pm 45^\circ$ to the fiber orientation (in-plane shear response). All tests were carried out at sub- T_g temperatures. The test temperatures selected for this study were 70, 80, 90 and 100°C . These temperatures were selected to ensure that measurable changes in physical aging can be measured within a lab time frame.

To ensure that all tests were performed within the linear viscoelastic range, a preliminary study was carried out to check that proportionality conditions and Boltzmann's superposition would be satisfied [10]. Specimens of AR-CF/PPS and SM-CF/PPS loaded at $\pm 45^\circ$ to the fiber orientation were repeatedly rejuvenated, quenched and subjected to sets of creep and recovery at tensile stresses ranging from 5 to 25MPa with increments of 2.5MPa at both the lowest and highest test temperatures. If the applied load places the material outside of the linear viscoelastic range, the momentary compliance response will vary depending on stress level, indicating that the material is in the nonlinear range. Based on this methodology, a tensile stress level of 10MPa was chosen for this study.

To provide comparability between AR-CF/PPS and SM-CF/PPS, all subsequent tests were performed at the same tensile stress level. The in-plane shear creep compliance response of the $\pm 45^\circ$ specimens is given by:

$$S = \frac{2A(e_x - e_y)}{P} \quad (7)$$

where S is the in-plane shear creep compliance, P is the axial load applied on the specimen, A is the cross-sectional area of the specimen, e_x is strain along the loading direction and e_y is the strain along the transverse direction.

The load schedule for the creep test is illustrated in Figures 1 and 2. Prior to test, the tested specimen was heated to 120°C (T_o in Figure 1) and kept at this temperature for 30 minutes. At the end of this 30 minutes, high-pressured air was used to quench the specimen from 120°C to the test temperature. The physical aging was defined to start immediately after the specimen reached the desirable test temperature.

The physical aging times selected for starting each creep segment were 4, 8, 24, 48 and 96 hours and the duration of each creep tests, t , was chosen to be $0.125 t_e$, thus respecting the snapshot condition previously mentioned. These selections were based on the lab opening hours, since the test machine is not fully automated.

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 the lack of full 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 2.

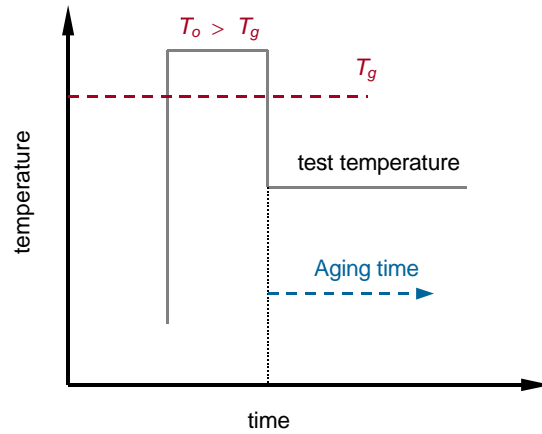


Figure 1. Outline of the procedure of measuring aging effects.

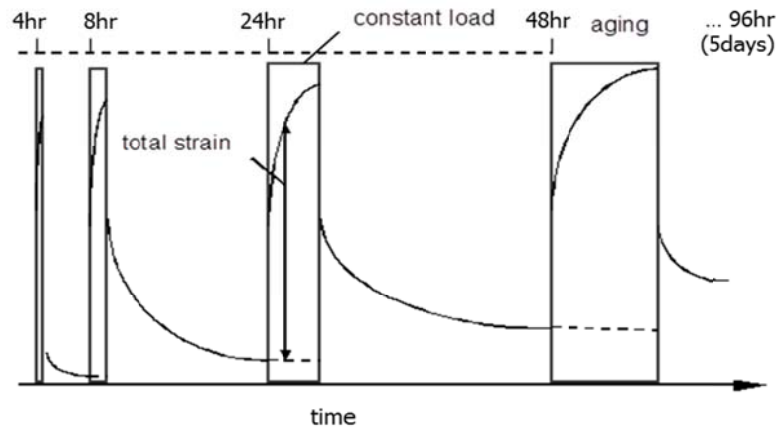


Figure 2. Sequence of creep test for determining the effect of physical aging.

The procedure illustrated in Figures 1 and 2 were repeated for all tested temperatures at both AR-CF/PPS and SM-CF/PPS.

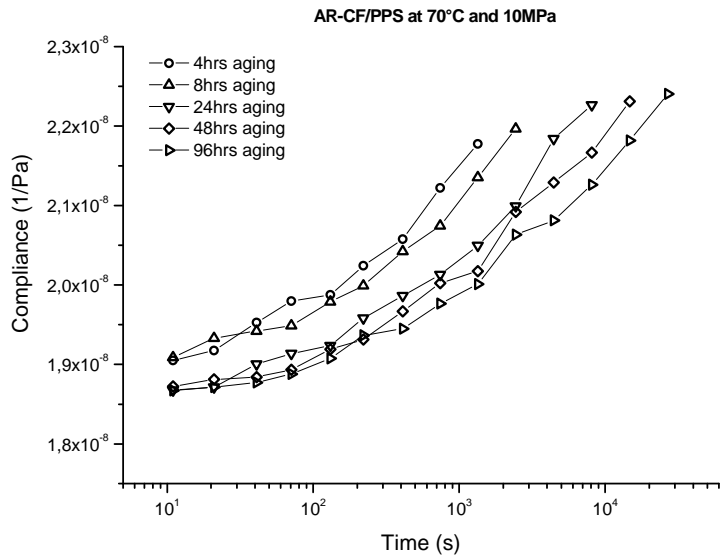
3. Results and Discussion

3.1. Data Reduction

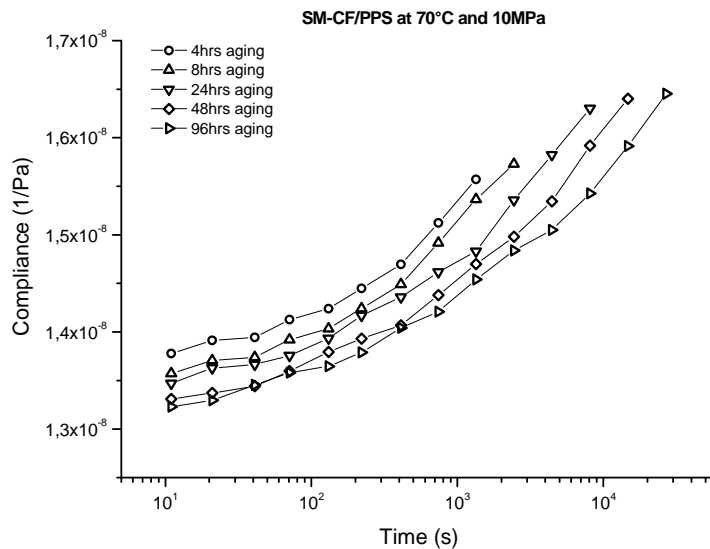
Since all the obtained data in this work were manipulated in log time scale, before starting any data interpretation an algorithm was used to pick data uniformly spaced in log time (reduced data) from a set of data uniformly spaced in time (raw data).

3.2. Physical Aging Effects on the Creep Behavior

Momentary creep compliance versus time curves for AR-CF/PPS and SM-CF/PPS specimens at 70°C are shown in Figure 3. The lines connecting the measured compliance are just guides lines. Similar curves were generate at 80, 90 and 100°C.



(a)



(b)

Figure 3. Momentary creep compliance as a function of time (in semi-log scale) at 70°C, 10MPa and different aging times for (a) AR-CF/PPS and (b) SM-CF/PPS.

As previously mentioned, physical aging leads to a decrease in free volume which, consequently, causes a reduction in segmental mobility (slowing down in creep response) and an increase in the degree of packing (stiffening). The curves shown in Figure 3 confirm these expectations. The creep curves shift towards longer times and the compliance levels decrease with increasing aging times. Similar trends were obtained for all other tested temperatures.

Time-aging time superposition was used to generate a single master curve for all of the data at a reference aging time of 96hours for both AR-CF/PPS and SM-CF/PPS. An algorithm was used to generate the aging shift factors, a_e . These shift factors were chosen by minimizing the mismatching in compliance by a least square method. The thus constructed curve is presented in Figure 4. Similar curves were generate at temperatures of 80, 90 and 100°C.

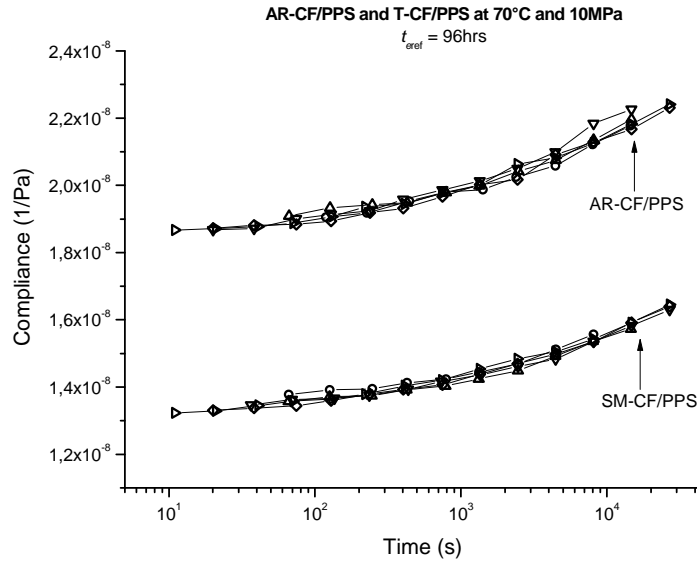


Figure 4. Master curves obtained by time-aging time superposition using momentary compliance curves from Figure 3 for AR-CF/PPS and SM-CF/PPS. The reference physical aging time is 96hours.

Figure 4 clearly indicates that the fiber surface treatment carried out in SM-CF improves the adhesion between the CF and the PPS matrix, resulting in a significant diminish of the measured compliance compared with AR-CF/PPS. Nevertheless, the fact that both curves appear to have an identical shape seems to imply that this improvement in the fiber-matrix adhesion has a minor effect on the creep response of these CF/PPS systems at 70°C. Similar trends were also observed for the curves obtained at 80, 90 and 100°C (see Figure 8(a)).

Figure 5 shows the obtained a_e versus the logarithmic t_e at 70°C. A small difference in a_e for AR-CF/PPS and SM-CF/PPS can be observed at 4 and 24hours of physical aging. Similar difference were also observed at 80°C. These difference can be explained by the uncertainty in the obtained a_e due to data reduction algorithm. Any data point with a certain inaccuracy has a bigger effect on the obtained a_e using the reduction data than using the raw data.

If the curves in Figure 5 are fit using a linear regression, the slope of this straight line is the aging shift rate, μ_e .

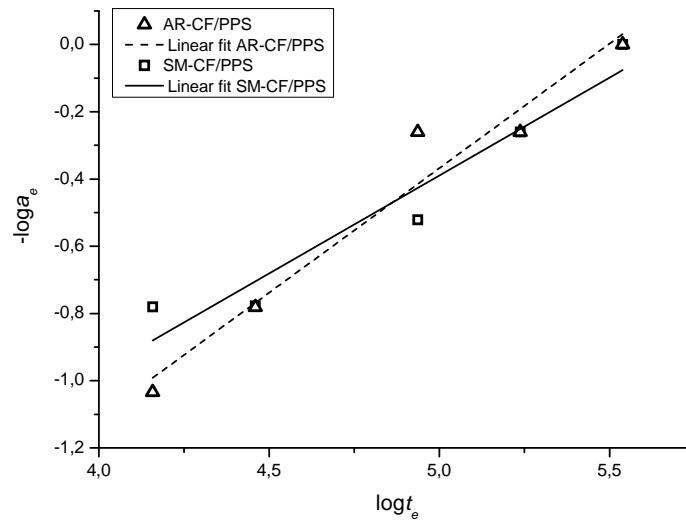


Figure 5. Aging shift factor for time-aging time superposition at 70°C and 96 hours reference curve for AR-CF/PPS and SM-CF/PPS.

Figure 6 shows the shift rate values obtained by repeating all above mentioned procedures at all tested temperatures. It can be seen that the aging shift rate decreases with increasing temperature. Note that above the glass transition, the shift rate should vanish since the material is in thermodynamic equilibrium. Therefore, a sharp decrease of the aging rate towards zero is to be expected above 102°C.

The fact that the obtained shift rates for AR-CF/PPS and SM-CF/PPS at 70 and 80°C are not identical comes from the previously mentioned uncertainty in the obtained a_e due to data reduction algorithm.

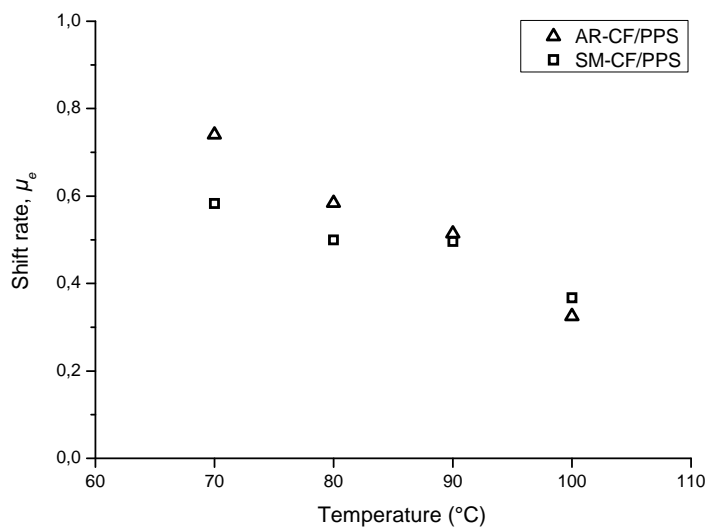
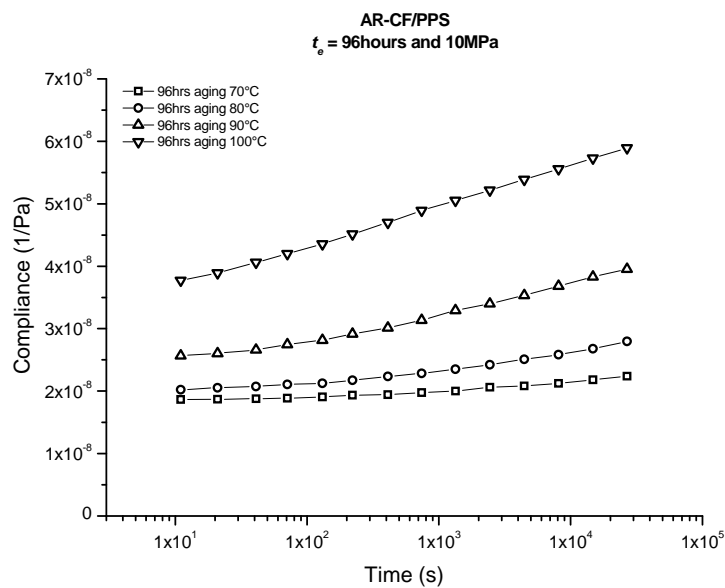


Figure 6. Aging shift rate as a function of temperature for AR-CF/PPS and SM-CF/PPS.

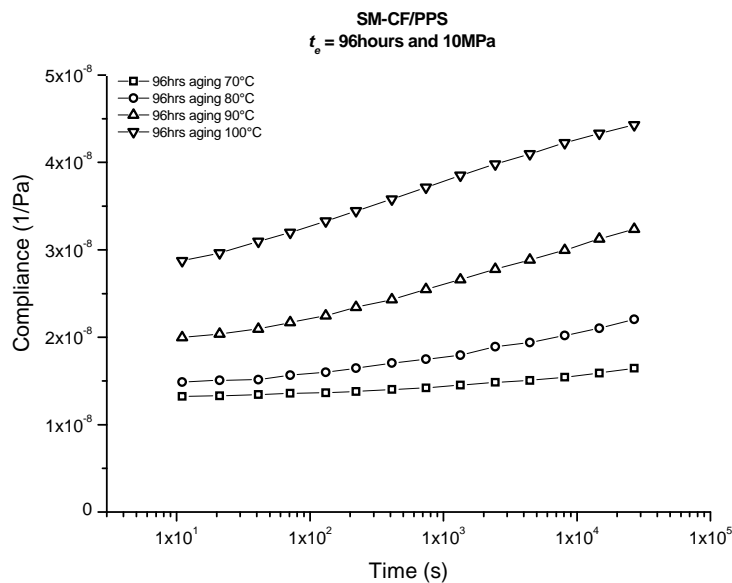
3.3. Temperature Effects on the Creep Behavior

Figure 7 shows time-aging time master curves versus time for AR-CF/PPS and SM-CF/PPS specimens at all tested temperatures. In this work, the 96hours reference curves were chosen to represent the time-aging time master curve. This choice was based on the fact that the 96hours curves are the ones with more data and perhaps the best in terms of mechanical conditioning of the specimen.

As previous mentioned in section 1.2, higher temperatures should accelerate molecular rearrangements in viscoelastic materials while lower temperatures slow it down. The curves shown in Figure 7 confirm this expectations. Furthermore, these curves shows that creep compliance was a function of test temperature, with an increase in temperature resulting in an increase in both compliance and related creep rate.



(a)

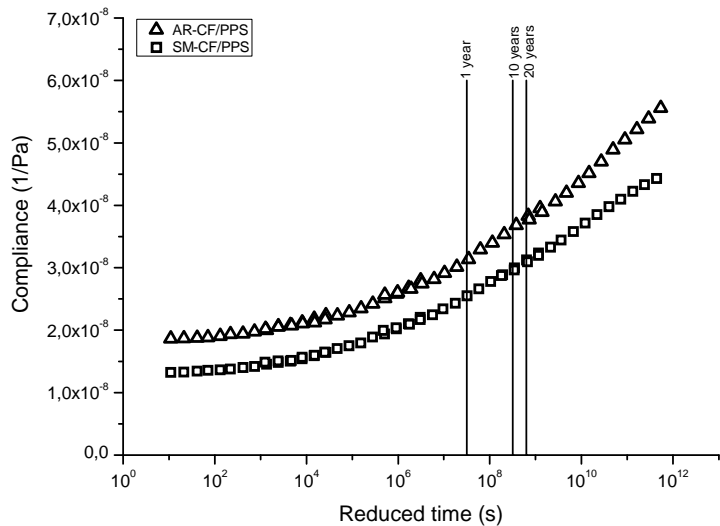


(b)

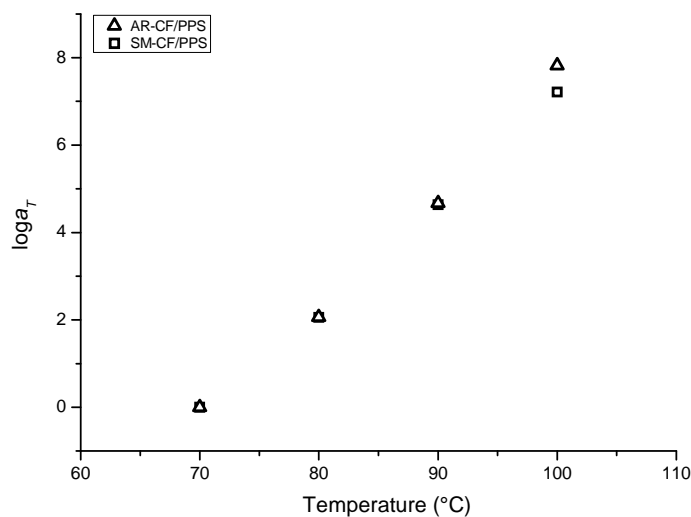
Figure 7. Time-aging time master curves (96 hours curves) at different temperatures for (a) AR-CF/PPS and (b) SM-CF/PPS.

The collection of time-aging time master curve for AR-CF/PPS and SM-CF/PPS at all tested temperatures was then collapsed into single material master curves using TTSP.

The collapse was successfully done using the 70°C time-aging time master curve as the reference and horizontally shifting all other curves. The TTSP allows the obtained master curves, along with the temperature shift factor versus temperature curves, to be used to predict momentary response at any temperature. The master curves and the temperature shift factors used to construct them are presented in Figure 8.



(a)



(b)

Figure 8. (a) Creep master curves of 96hrs aged specimens for AR-CF/PPS and SM-CF/PPS; (b) Temperature shift factors for AR-CF/PPS and SM-CF/PPS.

Comparison between the two obtained TTSP master curves at the reference temperature of 70°C shows that AR-CF/PPS exhibited a higher compliance throughout the extrapolated time. In fact, the observed nearly constant difference in compliance between these two TTSP master curves clearly indicates that the fiber surface treatment (SM-CF) performed by the supplier (TenCate) improves the mechanical performance of CF/PPS composites while having a minor effect on the time dependent

increase in deformation, i.e., creep response. A series of tests on the improvement of short and long-term mechanical performance by fiber surface treatment in CF/PPS composites have been carried out at Delft University of Technology [14, 15]. By analyzing SEM images of fracture areas of AR-CF/PPS and SM-CF/PPS, it was observed that while the surface of the SM-CFs seems to be still covered by the resin, the one of AR-CFs stands almost bare, showing therefore the improved mechanical and chemical adhesion between the fibers and the matrix of the SM-CF/PPS specimens. The results presented in this work seem to be in accordance with the findings obtained in these works.

3.4. Physical Aging Effects vs. Temperature Effects on the Creep Behavior

The previous section showed that both physical aging and temperature influence the linear viscoelastic creep behavior of CF/PPS composites. These influence, however, are characterized by changes in the viscoelastic creep response which seems to be different in time by several orders of magnitude. Furthermore, increasing both temperature and physical aging showed to have opposite effects on the creep response. While higher temperatures accelerate viscoelastic responses, longer physical aging times slow it down.

Figure 9 shows a comparison between the a_e used to shift the 4hours compliance curve at 80°C to collapse to the 96hours reference compliance curve at the same temperature, 80°C, and the a_T used to shift the 96hours at 80°C compliance curve to the 96hours at 70°C reference compliance curve.

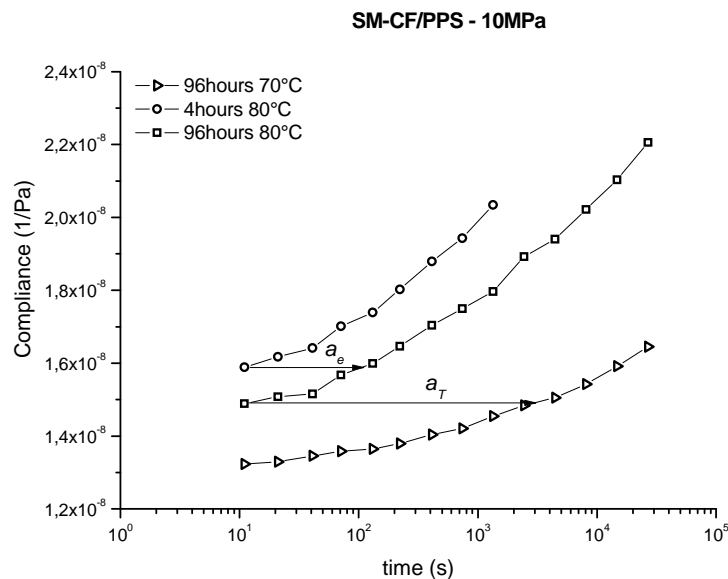


Figure 9. Creep compliance curves for SM-CF/PPS at 80°C, 4 and 96hours of physical aging and; at 70°C, 96hours of physical aging.

Thus, the analyses of Figure 9 indicates that, compared to temperature effects, physical aging showed to have a small contribution on the creep behavior of CF/PPS composites. In fact, a temperature change of 10°C, from 70 to 80°C (or the other way around), generated a temperature shift factor, a_T , that is approximately 20 times greater than the aging shift factor, a_e .

4. Conclusions

In this experimental study, the linear viscoelastic creep behavior of AR-CF/PPS and SM-CF/PPS using elevated temperature as the accelerator of the viscoelastic response were investigated. As a preliminary step to obtain the time-temperature master curve, as well as to characterize the changes in matrix dominated properties over the storage time, physical aging studies were also carried out using the short-term test method established by Struik. Short-term shear compliance curves were obtained for different physical aging times and at different temperatures. All tests were carried out at sub- T_g temperatures.

As expected, the obtained curves showed an increase in retardation times (or stiffening) as the physical aging times were becoming longer. Time-aging time superposition, was successfully applied to form aging master curves using the aging shift factors, a_e , generated by an algorithm. Differences in aging shift rates, μ_e , for AR-CF/PPS and SM-CF/PPS at 70 and 80°C were probably due to uncertainty on the obtained a_e .

Using the 96hours in-plane shear compliance curves as the representative of the time-aging time master curves, TTSP master curve were obtained by shifting these curves by a temperature shift factor, a_T , using the 98hours at 70°C curve as the reference curve.

The results indicated that surface treatment carried out in the SM-CF improved fiber-matrix adhesion, enhancing the mechanical performance of CF/PPS composites decreasing creep compliance of CF/PPS composites throughout the extrapolated time range. This fiber-matrix adhesion improvement, however, showed to have a minor effect on the linear viscoelastic creep response.

To further improve the analyses of the result, an attempt for finding suitable prediction models has been started. Furthermore, additional tests to characterize the effects of the fiber-matrix adhesion on the creep behavior of other PMCs will be carried out in the near future. These experiments will surely provide a useful foundation for new applying area of PMCs.

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6. References and Notes

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