Determination of Aging Shift Factor Rates for Field-Processed Polymers

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Abstract

The aim of this study is to develop a low-cost, short-term testing procedure to predict the time dependent properties of polymer liners over the lifetime of the installation (50 years). These materials are reformed or cured in the field during trenchless relining of sewer pipes. This paper focuses on the determination of the aging shift rate and shows that the time-dependent creep properties of these materials can be modeled within experimental error with slight changes to the effective-time and time-temperature superposition methods. To predict the creep behavior of full-scale specimens, the creep compliance is shifted from the testing age and temperature to the testing age and temperature of the full-scale specimens. It is shown that a rotation, along with a horizontal shift of the aging data, is needed to obtain the aging shift factor rate of the materials.

Introduction

The Urban Wet-Weather Federal Advisory Committee (FAC) and the Environmental Protection Agency (EPA) are enforcing a new set of standards and regulations that the current sewer systems in the United States must comply with. These new standards and regulations will require municipalities to invest heavily on the rehabilitation of the sewer collection systems. Due to high costs and traffic disruptions, trenchless lining of existing pipes is being used. In trenchless lining, a polymer or reinforced-polymer is applied to the inside of the existing host-pipe, without disturbing the soil or any aboveground facilities. The main purpose of the liner is to prevent water leakage into the sewer pipe. Therefore, a liner encased by a host sewer pipe is subject to hydrostatic external pressure, but it may also carry some soil pressure if the host pipe is deteriorated severely. Regardless of the source of external pressure, the encased liner fails by creep buckling under external pressure. If the time-dependent properties of the liner are known, realistic predictions of the time to failure can be made by rigorous numerical modeling of the buckling of a confined pipe.

The purpose of this study is to develop a low-cost, short-term testing procedure to predict the time dependent properties of polymer liners over the lifetime of the installation (50 years). A bending apparatus similar to ASTM D6272-98 is developed in this study to produce momentary curves of creep compliance at various ageing times, t_a, and temperatures, T_i. Age is the time elapsed since quenching after a period of annealing at a temperature above its glass-transition temperature, T_g. Aging times are chosen following Struik's snapshot assumption[1]. Time temperature superposition (TTSP) and the effective-time method are used to predict the time-dependent properties of the liner.

The relationship between stress and strain depends on time due to creep[2] and physical aging[1]. In a related study[3], creep and ageing were studied in the frequency domain using Dynamic Mechanical Analysis (DMA). However, two drawbacks of Dynamic Mechanical Analysis (DMA) testing motivate creep-bending tests (CBT): specimen size and test duration. DMA specimens are small (10 mm x 1.5 mm x 15 mm) and they must be cut from the pipe wall. Instead, CBT specimens use the full thickness of the pipe wall with a maximum testing size of 50 mm x material thickness x 231 mm. The larger size of the CBT specimens allows for a better representation of the material properties to be tested, particularly for field-processed polymers that tend to be more inhomogeneous than materials processed under industrial conditions. Running long-term tests on a DMA ties up the machine and is costly. Therefore, another, less expensive method to test specimens is desirable. The CBT fixture is designed to perform inexpensive, long-term creep testing in a configuration similar to ASTM D6272-98 four-point bending conditions.

Creep of thermoplastic polymer blends and felt-reinforced thermoset polymers involve three types of deformation: elastic, viscous, and viscoelastic. Upon sudden loading, the material experiences sudden elastic deformation and increasing time-dependent viscous and viscoelastic deformations. Upon unloading, the elastic deformation is recovered instantaneously, the viscoelastic deformation is delayed but eventually recovered entirely for very long times ($t \to \infty$), but the viscous deformation is never recovered. Such behavior has serious implications for this study. After each creep test, one would have to wait for a long

time ($t \to \infty$) to recover the viscoelastic deformation. In other words, the specimen never recovers its initial flat shape. The zero deflection of subsequent creep tests must be measured relative to the residual deformation from the preceding test. A novel fixture and associated instrumentation is proposed in this study to address this problem.

This paper focuses on the determination of the aging shift rate, μ. Physical aging starts when a polymer is quenched to a temperature below its T_g, regardless of whether or not the material is under load. Physical aging is accompanied by increases in stiffness, yield stress, density, and viscosity, and decreases in creep rate and stress relaxation rate. The effective-time method of Struik[1] assumes that ageing can be explained by a single shift, a_e, of the entire relaxation spectrum. In a typical Struik-type experiment, the sample is quenched from above the T_g to the testing temperature, T_1, and maintained at that temperature for the ageing time, t_e. Tests are then preformed for a short time interval, one-tenth the age, t_e, of the specimen. This method of testing takes a *snapshot* of the viscoelastic state, which prevents the data from being tainted by ageing.

Five different materials are tested in this study, three of which are polyvinyl chloride (PVC), one is High Density Polyethylene (HDPE), and the other is polyester reinforced with polyester fibers (PRP). The glass transition temperature of the materials is obtained from testing by Dynamic Mechanical Analysis in accordance to ASTM E 1640-99.

Experimental

Tests are conducted using a creep bending testing fixture (CBT) based on ASTM D6272-98 (Figure 1). This fixture uses two loading points with spacing of 64 mm or one third of the support span spacing of 192 mm. Four fixtures were built to support a specimen with a maximum width of 25 mm while four other fixtures were built to support a specimen with a maximum width of 50 mm. All four-load points have a radius of 7.94 mm, which allows for a minimum specimen depth of 4.96 mm according to ASTM D6272-98 Section 6.2. Load-point displacement is measured with a linear optical encoder with an accuracy of 0.005 mm and a maximum travel of 50 mm. Optical encoder, model LDK-4-4-B and spar and scale assembly, model B36679-4-0-50.0 from Dynamics Research Corporation are used. The majority of the testing apparatus is constructed out of aluminum, except for the load supports and the vertical rods where stainless steel is used. Linear bearings are used to allow the carriage to descend freely and apply the load to the specimen. Each specimen is fixed to the load points with rubber bands, which allows the top of the specimen to remain in contact with the bottom of the loading arms at all times during the test (points C and D in Figure 1). The spar-and-scale assembly is wired to the data acquisition system. The scale is mounted on a knife that slides inside the carriage and rests on the top of the specimen (Figure 1). The readout is reset to zero at this stage. This allows an accurate measuring of the deflection of the specimen. The specimen may not be perfectly flat due to imperfections or to incomplete relaxation after a creep test. However, resetting the readout with the knife resting on the specimen assures a true zero reading of deflection when no load is applied. The load is applied when the carriage is lowered, by removing the block underneath it, and only when the specimen touches the end supports A and B (Figure 1).

The following steps describe the process used to perform an ageing study:

1. Set environmental chamber to desired temperature and allow it to reach desired temperature.
2. Anneal the specimen for at least 30 min.
3. Quench the specimen for 5 min, $t_e = 0$ when the specimen is removed from the oven.
4. Place specimen in the carriage by securing the top of the specimen to the bottom of the load points with rubber bands.
5. Place carriage on fixture inside the environmental chamber and use block to keep the specimen from coming in contact with the loading arms (no load is applied). Place knife-edge into carriage making sure it can move freely and is in contact with the specimen.
6. Zero the readout and begin recording deflection.
7. Let specimen age to desired testing age.
8. Lower the carriage onto the loading arms by removing the block underneath it. As the specimen bends the knife-edge remains in contact with the center of the specimen and the optical encoder measures deflection.

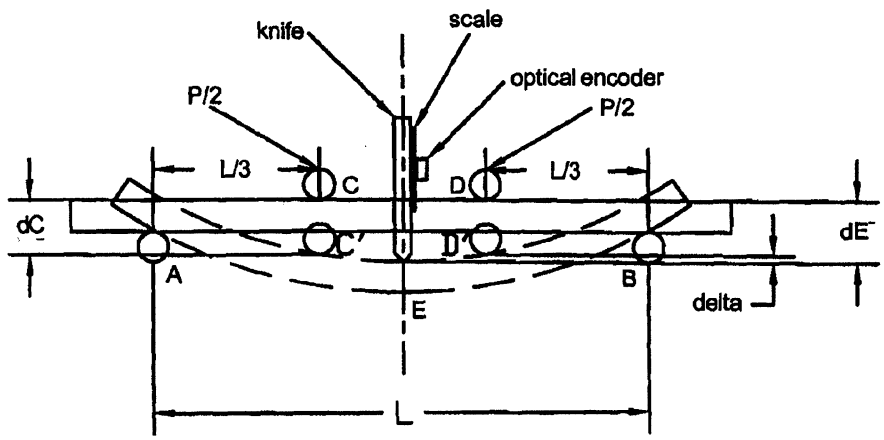

Figure 1. Illustration of specimen under four-point bending, the dashed line represents the deformed specimen due to loading.

Table 1. Typical testing schedule for an aging study.

	day and time test began	t_e *(min)*	*test duration (min)*
test 1	01/23/02 10:11:25 AM	11.42	1.14
test 2	01/23/02 10:16:39 AM	16.65	1.67
test 3	01/23/02 10:30:13 AM	30.22	3.02
test 4	01/23/02 11:33:23 AM	93.38	9.34
test 5	01/23/02 02:57:41 PM	297.68	29.77
test 6	01/24/02 09:54:18 AM	1434.30	143.43
test 7	01/25/02 09:53:56 AM	2873.93	287.39
test 8	01/26/02 01:12:58 PM	4512.97	451.30

9. Once one-tenth the ageing time has passed remove the load by raising the carriage and placing the block underneath it.
10. As the specimen relaxes, the knife is forced upward and the relaxation is recorded.

Data is acquired using an ACS-Tech80 5312B ISA Encoder Interfacing card with four axes. An Intel-based computer running Windows 98 is used to read the card and store the data. Software drivers provided by the DAQ manufacturer were modified to write data to files in specific time increments using Visual Basic 6.0. All tests are preformed in a Cincinnati Sub-Zero model Z-32 environmental chamber that uses a Chromalox 2030 microprocessor controller.

New liner samples are produced at the vendor facility or field installation of each material. In either case, the samples are taken from rounded pipe and processed into a cylindrical configuration. All liner samples are cut perpendicular to the direction of extrusion and have a minimum length of 90 cm. If the installation prohibits the removal of samples at this length, three samples not less than 30 cm in length each are provided. The 90 cm samples collected from the field are cut into three pieces, each with a length of 30 cm.

Next, the samples, except for the thermoset material, are flattened. First, the pipe section is cut longitudinally into three equal sections. These sections are then placed, concave side down, on a piece of flat aluminum with a thickness of 12.7 cm and overall dimensions slightly greater than the sample and placed in an oven. The orientation of the sample indicates the hoop-wise direction of the sample. A second identical aluminum plate is placed on top of the sample and additional weights are added. The samples, aluminum plates and additional weights are then heated to T_H and maintained at that temperature for not less than 48 hours. The specimen annealing temperature, T_H, is selected to be 15 °C above the glass transition temperature (T_g) of the material, but below it's melting point (T_m).

The specimens have a minimum aspect ratio of 16 to 1 and had a maximum width of 50 mm and a minimum depth of 4.96 mm. All specimens are cut to width using a band saw. The thickness and width of each specimen are measured at both ends and at the center of the span using a micrometer as per Section 6.3 of ASTM D790 and then averaged. The specimens, except HDPE, are annealed in an oven at temperature T_H for at least 30 min. The HDPE specimens are annealed at the temperature used to expand the liner during installation. They are then quenched between steel plates for approximately 5 min. After the specimens are quenched, they are placed in the fixture and held in place with rubber bands wrapped around the inner loading nodes (Figure 1). Next, the spar and scale assembly is placed in the fixture. Using the data acquisition software, deflections are zeroed, the test filename and path are entered and recording begins. Finally, the load is applied by lowering the carriage.

Testing Procedure

Tests are preformed at a temperature, T_I, of 40°C. Due to room conditions and the control of the environmental chamber, the actual value of T_I ranges from 39.5°C to 40°C. The width of the specimens is in the range of 7 mm to 13 mm. The testing temperature and specimen width are determined by doing several practice tests to achieve a deflection large enough so that the precision of the optical encoder (0.005 mm) does not interfere with the results. The specimens are tested according to Struik's snapshot assumption[1]. This assumes that if the test time is less than one-tenth the age of the specimen, ageing is not significant during the test time. The testing schedule varies with each study to accommodate the operator's schedule, but the snapshot assumption is always followed.

Tests are conducted at eight intervals during the aging experiment. A typical testing schedule is shown in Table 1. Conducting an ageing study and calculating the shift factors, a_e, determines the required number of tests. Using

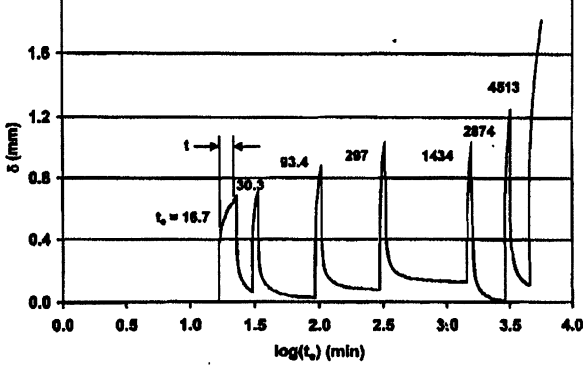

Figure 2. Aging creep testing.

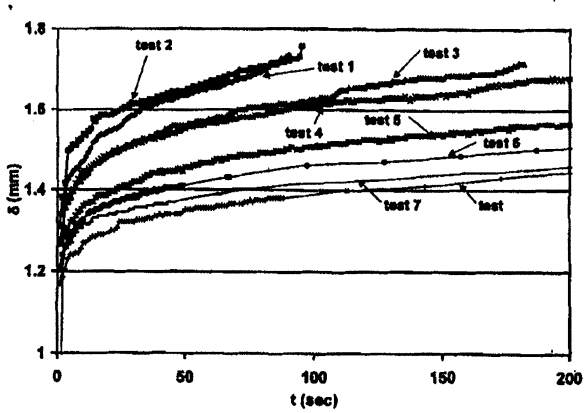

Figure 3. Deflection vs. time illustrating mechanical conditioning.

these shift factors, m is calculated and tests are performed until the value of m remains constant; that is, the slope of the $\log(a_e)$ vs. $\log(t_e)$ does not with the addition of another data point. For all materials tested, this is accomplished with eight intervals of testing over approximately four days.

The deflection and relaxation are written to a data file using the software described previously. The software continuously records the d(t) while the user loads and unloads the specimen. The deflection vs. $\log(t_e)$ is illustrated in Figure 2. One can observe that the specimen never reaches equilibrium during the relaxation period.

Data Analysis

Data reduction was performed using Microsoft Excel 2000 and Microcal Origin 6.0. The files from the CBT software are opened in Microsoft Excel. The load is removed after each snapshot test, allowing the sample to relax, but the initial flat shape is not recovered. This occurs because the relaxation time is finite and because a portion of the viscoelastic behavior is that of a fluid[4]. Each snapshot test starts from a deflected initial position, which is easily found since the optical encoder reads deflection during the relaxation as well as during the creep test. Once the initial deflection is established for each snapshot test, the relative deflection data vs. time represents a snapshot creep curve.

As the material ages, it becomes stiffer, reducing the amount of deflection observed during the test. Therefore, with each successive test, the deflection vs. time curves should lie under one another. This was not observed with the first two tests (loading and unloading of the specimen as shown in Figure 3). This occurrence is because an imperfect specimen must adjust and settle onto the load supports. Therefore, the first two tests for each ageing study are not used in the calculation of μ. The first two tests are subsequently referred to as mechanical conditioning.

Next, the compliance D(t) was calculated. The equation for the compliance under four-point bending was derived using the method outlined next. Referring to Figure 3, the maximum deflection of a beam under four-point bending with 1/3 load span is[5]

$$dE = \frac{-P/2\, b\left(L^2 - b^2\right)^{3/2}}{9\sqrt{3}EIL} + \frac{-P/2\, a\left(L^2 - a^2\right)^{3/2}}{9\sqrt{3}EIL} \qquad [1]$$

where P is the load, L is the support span, E is the relaxation modulus, $I = wh^3/12$ is the moment of inertia of the rectangular specimen of width w and thickness h, $a = L/3$ and $b = L/3$. The deflection at the load supports is[5]

$$dC = \frac{P/2\, a^2 b^2}{3EIL} + \frac{P/2\, b}{6EIL}\left[x^3 - \left(L^2 - b^2\right)x\right] \qquad [2]$$

where $a = L/3$, $b = 2L/3$, and $x = L/3$. The deflection of point E with respect to points C and D is

$$\delta = dE - dC \qquad [3]$$

Thus the compliance is

$$D(t) = \frac{243\left(64\sqrt{6} + 135\right)\delta(t)wh^3}{2117PL^3} \qquad [4]$$

The compliance D(t) is separated into an elastic component D_o and a creep component $D_\alpha(t)$ and the later is modeled with a power law

$$D = D_o + D_\alpha(t) = D_o + D_1 t^m \qquad [5]$$

where D_o is the initial compliance, D_1 is the creep coefficient, t is the time, and m is the power-law exponent. Equation 5 contains three-parameters and it is a straight line in log-log scale

$$\log D_\alpha = \log D_1 + m \log (t) \qquad [6]$$

All data fitting parameters were obtained using Microcal Origin 6.0. The power-law term in Equation 5 is a first-order approximation of the four-parameter model used by Dean et al.[6]

$$D_\alpha(t) = (\Delta D_\alpha)\left[1 - \exp - \left(t/\tau_\alpha\right)^m\right] \qquad [7]$$

Equation 7 contains four parameters and provides a better model for relatively long-term data. However, it is impossible to adjust the four-parameters with short-term data such as that generated for short ageing times because the data does not have enough information to elucidate four parameters. As the age t_e increases, so does the snapshot testing time $t = t_e/10$, and the data can be used to elucidate the four parameters in Equation 7. Expanding the exponential term as a power series yields

$$D_\alpha(t) = \Delta D_\alpha \left[\left(t/\tau_\alpha \right)^m \left(1 - \frac{1}{2}\left(t/\tau_\alpha \right)^m + \ldots \right) \right] \qquad [8]$$

and retaining to only one term, Equation 8 reduces to Equation 5 with $D_1 = \Delta D_\alpha / \tau_m$. When Equation 5 is used to fit data from the longer aging times, the fit is not perfect and the power-law exponent differs form that of the shorter aging data. Nevertheless, all data is modeled with [Equation 5] because (a) it yields a straight line in log-log plot and (b) the same model Equation 5 can be used for all data whether it has a short age or long age. It is worth noting that the reason why the power law exponent m changes with age t_e is due to the inability of Equation 5 to fit perfectly long periods of creep data and not because the material changes. Aging is reflected in changes of parameters D_0 and D_1. As a result, all the creep curves should be parallel lines in log-log scale.

Once all of the curves are fit using Equation 5, the next step is to shift the curves to obtain the shift factors and shift factor rate, μ, for each material. Bradshaw and Brinson[7] selected the curve with the longest aging time as the reference curve, since it spans the longest test time range, and superimposed each momentary curve on it creating a master curve according to[7]. Dividing each time by a shift factor, a_e, the curves are to be shifted on top of the reference curve. However each of the curves could only be shifted so that a few points would match, creating the problem of which points should be used to align the curves onto the reference curve. In other words, the shifting procedure becomes operator dependent. To solve this problem, it is observed that the creep component D_α is a straight line in log-log plot, or

$$\log(D_\alpha) = \log(D_1) + m \log(t) \qquad [9]$$

Once a straight line represents the data, it is noticed that the slopes of each curve did not match. The problem again is which point should be used for the curve shifting. The difference between m_i and the average m represents a rotation of the curve. Chai and McCrum[8] and Guerdoux et al.[9] also discussed the need for rotating the data in addition to a horizontal shift to calculate a_e but did not indicate a systematic procedure for performing the rotation and horizontal shift. To solve this problem, the values of m_i (the slope of the curves) for each test during the aging study are averaged

$$m = \frac{\sum_{i=1}^{N} m_i}{N} = ave(m_i) \qquad [10]$$

and the data for each snapshot test is refit keeping the value of m constant and equal to the average. A comparison of the data fit letting m_i vary and using the average m for the entire ageing study is shown in Figure 4. As it can be seen, there is minimal difference. The need for a rotation of the data occurs because the modified power law does not fit data for large times as well as it does for shorter times. The unrotated data is shown in Figure 5 where it can be seen that the lines are not parallel due to variations in m_i. The rotated data is shown in Figure 6. Each curve is now a straight line with the same slope and can be shifted easily on top of one another.

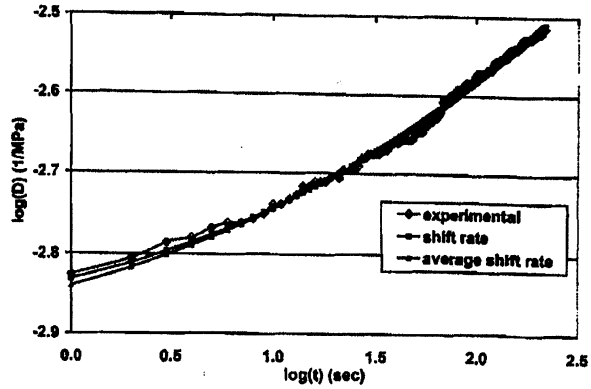

Figure 4. Comparison of experimental data, date fit and rotated data fit.

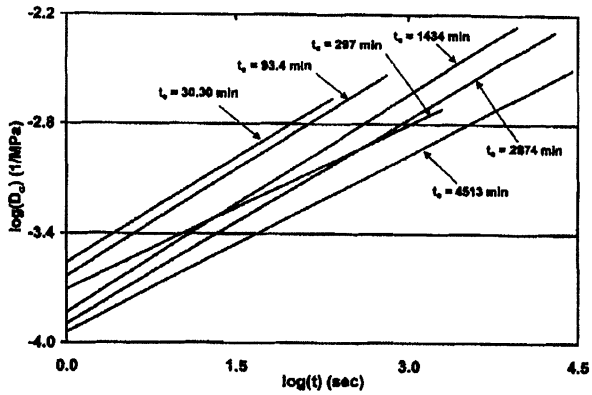

Figure 5. Unrotated aging data in modified power-law plot.

Each shift factor is analytically calculated using the following method. First, an arbitrary value of $\log(D_e)$ is selected, say ~3.2(1/MPa) in Figure 6. The corresponding time at which each test of age t_e reaches the same compliance $\log(D_e)$ is read on the abscissa of Figure 6, or calculated from Equation 9 as

$$\log(t) = \frac{\log(D_e) - \log(D)}{m} \qquad [11]$$

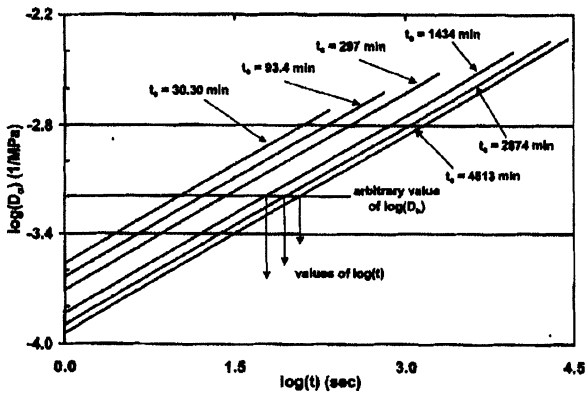

Figure 6. Rotated aging data in modified power-law plot.

Then, using the longest ageing test as a reference (a_e=1), the aging shift factor a_e of each test is calculated as

$$a_e = \frac{\log(t)_e}{\log(t)_{te\,max}} \qquad [12]$$

Since the power-law exponent m is the same for all snapshot tests, the arbitrary value chosen for $\log(D_\alpha)$ does not affect the results calculated with Equations 11-12. For example, this procedure shifts all curves in Figure 6 to the right, on top of the data for t_e=4513 min. The process of calculating the shift factors is illustrated in Figure 7.

For each material, an aging study is done on three specimens separately. For each specimen, all of the snapshot curves are shifted to the longest age t_{emax} and the corresponding shift factors a_e are found with a_e=1 for the data at the reference age $t_{eR}=t_{emax}$. The three specimens have a slightly different longest age t_{emax} due to differences in the testing schedule. In order to facilitate further data reduction, it is desirable to have all data for all specimens to use the same reference age t_{eR}. Therefore, all age factors for all snapshot tests in a given specimen are shifted to t_{eR}=15min with a_e=1 at this common reference age. This was done for each specimen independently using its own shift factor curve. Then, the three specimens have the same reference age, t_e=15 min with a_e=1. By using a common reference age, a_e, values of every snapshot for all specimens can be plotted together, as in Figure 8. Linear regression of $\log(a_e)$ vs. $\log(t_e)$ yields a linear equation that allows us to predict the shift factor $a_e(t_e)$ for any age t_e, including the age of the full-size encased liner specimens tested by Barbero and Rangarajan[10, 11]. The slope of the line is the aging shift factor rate μ.

Prediction of Creep Data

TTSP is a method used to predict the behavior of a material for times longer than the test time. This method accelerates the creep of the material by an increase in the temperature. It assumes that this increase in temperate can accelerate but not change the material property being investigated, in our case compliance.

For TTSP testing, materials are prepared as described in Section 2. They are then placed in the testing fixture at the desired testing temperature to let age. Each specimen is aged to t_e=60 min and then tested for 6 min, following the snapshot assumption to avoid the effects of ageing.

Creep compliance is measured at each temperature over a span of time shorter than t_e/10, where t_e is the age of the material. This is done so that the creep compliance curve is free of ageing effects. Test data at each temperature are plotted on a base-10 log-log scale versus time. One temperature is chosen as a reference temperature, T_R. The remaining curves are superimposed onto the reference curve by a shifting with a horizontal shift factor a_T. Once all of the curves are shifted, a master curve that spans longer time than the longest testing time is created. This master curve can then be shifted to any temperature, and used to predict the value of compliance over time. Since every individual test is free of aging, the master curve is free of aging even if it spans a time range much larger than t_e/10.

Struik[1] proposed that the equivalent time could be determined using the following equation

$$t = t_e\left[\left[\left[\left(\frac{\lambda\alpha}{t_e}\right)+1\right]^{\frac{1}{\alpha}}\right]-1\right] \qquad [13]$$

where t is the equivalent time to compensate for ageing, I is the unaged time on the TTSP curve, a=1-m, and t_e is the age (time at which the CBT test is started). The compliance is then read on the TTSP curve and plotted

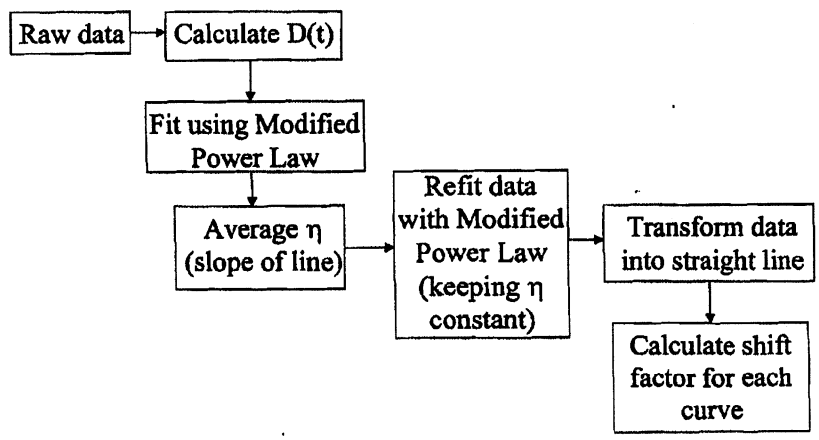

Figure 7. Calculation of aging shift factors.

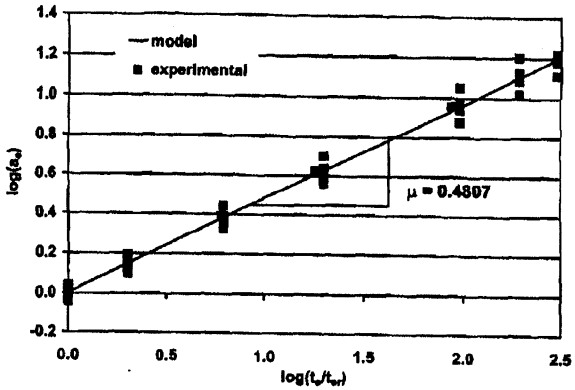

Figure 8. Theoretical and experimental values of the aging shift factor.

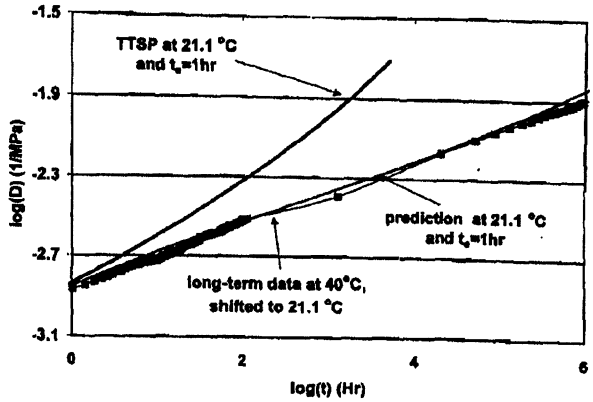

Figure 9. TTSP, prediction and shifted data comparison at t_e=1 hr and 21.1°C.

against the times calculated using Equation 13. A comparison of the TTSP (unaged), predicted (aging), and experimental data shifted for temperature is shown in Figure 9.

Conclusions

It is shown that horizontal shifting alone is not enough to superimpose the creep curves obtained from an ageing study. By using a power law and plotting the creep compliance in log-log scale, the resulting straight lines are clearly not parallel. This demonstrates the need for a rotation of the data before horizontal shifting. A novel two-step data reduction algorithm is proposed, using the average power-law exponent in the second step to fit all data available with parallel lines. This results in a convenient, deterministic procedure to obtain the shift factors accurately and repeatably. Next, by recasting all the data for a common reference age, data from several replicates of the same sample can be used to obtain an accurate measure of the ageing shift rate through linear regression. Such accuracy is necessary to accurately predict creep of polymers at times much longer than the available experimental data. A novel fixture is proposed to measure creep and relaxation continuously over time.

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