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Time-Temperature-Age Viscoelastic Behavior of Commercial Polymer Blends and Felt Filled Polymers

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ABSTRACT

This work presents an experimental investigation of creep behavior of polymer blends and felt-filled plastics used to rehabilitate deteriorated sewer pipelines, with emphasis on characterizing the effects of physical aging and temperature. The procedure for finding the aging shift rate μ is based on Struik's protocol and includes a novel method for rotating the data in addition to shifting. The master curve, obtained by Time Temperature Superposition (TTSP), of short-term data is shifted to the desired test temperature and initial age. A novel method is proposed to incorporate data from several specimens into a single averaged master curve and shift factor plot. Long-term behavior of the samples is predicted using the master curve and Effective Time Theory (ETT). The predicted long-term creep behavior is found to be close to the experimentally measured long-term creep behavior.

§1. INTRODUCTION

Industrial, storm, and sanitary sewers are an important part of the nation's aging infrastructure. These underground facilities are often underneath of other vital constructed facilities, such as buildings, roads, and other pipelines. Conventional dig and replace methods are often costly and inconvenient. Trenchless lining is being used to re-line existing pipelines with felt-filled thermoset plastics or thermoplastic polymer blends, without disturbing aboveground and other facilities. A liner is inserted into the host pipe between existing manholes and expanded against the host pipe using internal water or steam pressure. Thermoplastic polymer blends and felt-filled thermoset polymers are then cured and reformed, respectively, against the interior of the host pipe. The system restores the structural integrity and fluid tightness of the sewer. The mode of failure controlling the design of the trenchless liner is creep buckling under external water pressure. Therefore, long-term

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Table 1
Aging protocol

Step #	Action	Temp. (°C)	Time (min)	Furnace	Load
1	Annealing	$T_g + 15$	10	Closed	Off
2	Quench	Room	5	Open	Off
3	Age	40	5	Closed	Off
4	Mechanical conditioning	40	1	Closed	On
5	Relaxation	40	1	Closed	Off
6	Mechanical conditioning	40	1	Closed	On
7	Age	40	t_e-30 (*)	Closed	Off
8	Creep	40	t_e/10 (*)	Closed	On
9	Repeat			Closed	Off

(*) t_e = aging time in increments: $t_{i+1} = 3^* t_i$ (equal increments in log scale).

These then were cut into several pieces of length 18 mm. The materials referred to in this study are three commercial Poly Vinyl Chloride (PVC) types, a commercial high-density polyethylene (HDPE), and a polyester resin filled with polyester felt. Since the later is a thermoset, the samples were fabricated flat.

Aging experiments were carried out by first annealing the specimens at a temperature 15°C above its T_g. The sample then is quenched from above the glass-transition temperature, T_g, to the testing temperature and maintained at that temperature for the aging time, t_e. Tests then are performed for a short time interval (one-tenth the age, t_e, of the specimen (see Table 1)). The creep curves are shifted to a reference age t_{eR} using a shift factor a_e. In this study, t_{eR} was chosen as the age of the longest aged test. The slope of the double logarithmic plot of log (a_e) vs. log (t_e) is the aging shift rate, μ.

After recording the sample dimensions, the sample is placed in the three-point bending fixture of a Perkin Elmer DMA 7e with the furnace enclosing the specimen. The specimen is kept at the annealing temperature for 10 min, after which the furnace is opened in order to air-quench the specimen at room temperature. The time at which the furnace is opened is considered to be age zero ($t_e = 0$). In the meantime, the furnace is set to the test temperature. After 5 min the furnace, having reached the test temperature, is closed. The sample is allowed to stabilize in the test temperature for another 5 min. Creep tests are performed at the ages of 10, 12, 30, 90, 270, 810, and 2430 min. The duration of each test complies with the snapshot assumption [6], or $\lambda/t_e \leq 10$, where λ is the test time. Since the test duration is short, aging does not affect the creep results. The test time over which aging effects are negligible is called unaged time and denoted with λ to differentiate it from real time t, over which aging effects are significant.

The load and corresponding deflection data for the specimen are automatically recorded. The compliance D (λ) for every point in the test is calculated by dividing the strain by the constant stress. The double logarithmic plot of compliance vs. time at every age of the sample is plotted. The curves are fitted with a power law [5],

$$D(\lambda) = D_o + D_1\lambda^n, \tag{1}$$

where D_o is the initial compliance, D_1 is the linear coefficient, λ is the unaged time, and n is the power exponential.

viscoelastic behavior of liners is the most important aspect for durability and the dimensional stability of trenchless rehabilitation of underground sewers. An extensive understanding of the viscoelastic properties of polymers is available [1–4]. However, accurate experimental methods and models for the prediction of physical aging and its effects on long-term creep of commercial products need further development. The objective of this study was to develop an accelerated testing procedure to predict the long-term creep of liners processed (cured or reformed) in standard field conditions.

When a polymeric material is annealed above its glass transition temperature (T_g) and quenched below T_g, it does not immediately achieve thermodynamic equilibrium. Instead, the material evolves towards thermodynamic equilibrium. This phenomenon is known as physical aging [6], and it is accompanied by increase in stiffness, yield stress, density, and viscosity. Also, there is a decrease in creep and stress relaxation rates and flexural strength.

Beckmann et al. [7] performed creep experiments on semi-crystalline syndiotatic polystyrene (sPS) having different processing histories. They concluded that physical aging behavior depends on the processing history. Therefore, in anticipating the development of standard test methods for the viscoelastic behavior of trenchless liners, the annealing and quenching protocols used in this study were defined to resemble actual installation conditions in the field.

Brinson and Gates [12] described the effects of aging on the effective equilibrium and their relationship to the time-temperature shift factor. In their study, it was shown that when a material was loaded at an initial aging time less than the equilibrium time, the response initially followed the momentary curve, then deviated as the test approached equilibrium time. Then as the combined test time and the initial aging time exceeded the equilibrium time, the response followed parallel to the momentary curve once again. Their results are in good agreement with short-term responses at increasing aging time, but no experimental data supports long-term predictions.

Wang et al. [13] investigated the effect of physical aging on the viscoelastic creep properties of a thermoplastic–toughened cynate ester resin (Fiberite 954-2) and its IM8/954-2 composite. On testing the cynate ester samples, it was found that they failed to rejuvenate. Hence, ETT could not be used. In order to predict long term data, an empirical equation was fit to data up to 26,000 s, assuming that the β region of the curve would remain linear. It was assumed that long-term behavior could be predicted from the empirical equation, provided the assumption of linearity remained correct.

Between 1962 and 1972, Struik [6] did a systematic study of the aging phenomenon and its effects on a variety of mechanical properties of more than 40 materials, most of which were synthetic polymers. He concluded that the temperature range in which the aging occurs generally is not restricted to a narrow band below T_g, but it usually falls between the primary transition (T_g) and the secondary transition (T_β). In this work, it is shown that several modifications to the aging methodology of Struik [6] are desirable in order to successfully predict long-term data (up to 35,000 h shown here) for a variety of commercial products, including PVC, HDPE, and polyester filled with polyester felt.

§2. AGING STUDY

Round pipe liner samples of each material were produced at the vendor facility by lining a 305 mm ID steel pipe. All liner samples were cut perpendicularly to the direction of extrusion and had a minimum length of 305 mm. The samples then were cleaned using a mild soap and water solution. These samples were further cut into four pieces (with longitudinal cuts) into four equal quadrants. A strip of thickness 1.5–2 mm was cut along the thickness.

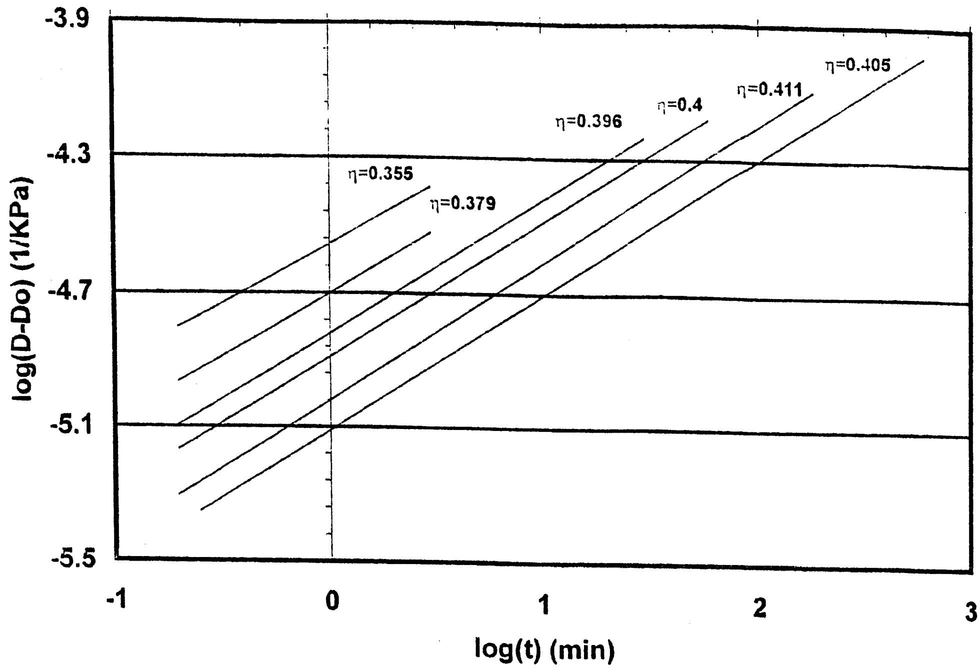

Figure 1. *Momentary creep curves fit with power law having different slopes* (T = 40°C).

The obtained curves have different slopes and thus cannot be superposed (Figure 1). This is because the power law model is not sufficiently accurate to represent the data when the testing time is relatively long. A model with four parameters can represent the data for long test times, but the four parameters cannot be univocally found when the test time is short. In other words, data over short periods of time does not have enough information to allow for a univocal fit of an equation with four parameters. Since the test time is constrained by the snapshot assumption to be one tenth of the aging time, every aging study has both short and long-time data. For the curves to superpose each other, they must have the same shape. Thus, they must be represented by the same equation. This forced us to choose the power law to represent all the curves. As a result, the slope of the curves in log-log scale are not exactly identical, rather they are similar. In fact, a comparison between the actual data and the fit, with and without rotation, shows negligible difference. Hence, these curves are rotated in order to make all curves parallel straight lines. The rotation is accomplished by averaging the slopes of all the curves and refitting every curve with Eq. (1), yet keeping the slope η fixed at the average value [10]. The need for rotating the data was also mentioned in the findings of McCrum [9]. Note that the averaging procedure is systematic and, thus, not prone to interpretation or error of the operator. Also, such a procedure does not involve non-linear minimization or other algorithms that may yield multiple solutions [12].

Next, a reference curve is chosen at age t_{eR}, and other curves at different ages are shifted horizontally by using shift factors, a_e. Note that since the curves are parallel lines in a log-log scale, the shifting is accomplished simply as the distance between two lines. Such a procedure is systematic and not prone to operator interpretation or error. The shift factor moves the curve in the horizontal direction in the time scale, and thus, a master curve is obtained (Figure 2). The aging shift rate, μ, for all materials (Table 2) is found by making a double logarithmic plot of $\log(a_e)$ vs. $\log(t_e)$ (Figure 3) and finding its slope, given as

$$\mu = \frac{\log(a_e)}{\log(t_e/t_{eR})}. \tag{2}$$

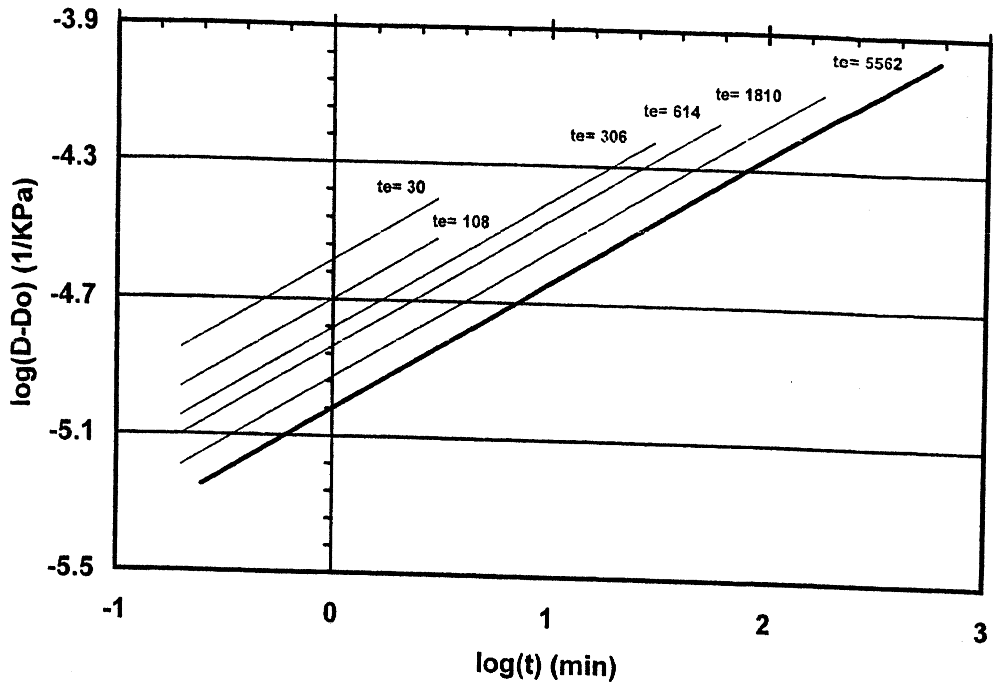

Figure 2. *Momentary creep curves at different aging times, shifted to form a master curve (t_{eR} = 5562 min).*

Using this slope, the shift factor a_e at any given aging time t_e can be found by:

$$a_e = 10^{(\log(t_e/t_{eR})*\mu)}. \tag{3}$$

Multiple specimens yield multiple values of aging shift factor μ, which were averaged. The average and standard deviation are shown in Table 2. As shown in Figure 3, the first two tests (t_e = 10, 12 min) should be discarded because they do not line up with the rest of the data. It was corroborated that this phenomenon occurs regardless of the age of the first two tests (t_{e1}, t_{e2}). It is postulated that each specimen needs to be loaded and unloaded a couple of times to obtain a perfect seat on the supports and loading point. Basically, two tests are performed at ages much shorter than the shortest age of interest t_{e1}, and their associated data are discarded. Such a process is called mechanical conditioning. In this study, the shortest age of interest is $t_{e1} = 30$ min. Consequently, two creep tests were performed at age $t_e = 25$ and $t_e = 29$ min of duration $t = 1$ min each, and the associated data are discarded. Then, an

Table 2
Aging shift rate, μ, for all materials at 40°C

Material	μ (All specimens averaged)	Standard deviation	Number of specimens
F	0.526	.079	3
G	0.877	.095	3
H	0.865	.023	3
I	0.179	.025	3
J	0.787	.036	3

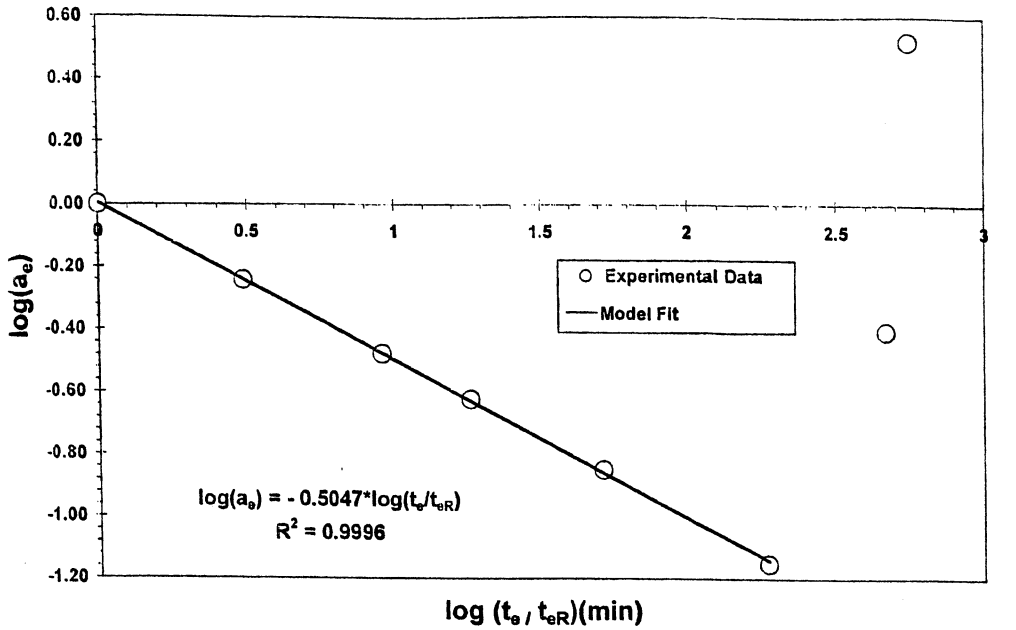

Figure 3. *$Log(a_e)$ vs. $Log(t_e/t_{eR})$ plot to find the aging shift rate ($t_e = 60$ min).*

additional 30 min lapse between the end of the mechanical conditioning tests and the time a which the first useful creep test is performed at $t_e = 60$ min, which allows for sufficient tim for the sample to relax. All subsequent tests in this study were mechanically conditioned.

§3. TIME-TEMPERATURE SUPERPOSITION OF MOMENTARY DATA

In Time Temperature Superposition (TTSP), tests are conducted at different tempera tures and the creep curves thus obtained are superimposed on a reference curve to form master curve (Figure 4). In this study all creep tests are momentary curves obtained by snap shot testing [6]. In a snapshot test, $\lambda/t_e < 1/10$. That is, the testing time λ remains smal when compared to the physical aging time t_e. In our case, the physical aging time was t = 60 min, and the testing time was kept at $\lambda_{max} = 6$ min. If each of the individual curve used to construct the TTSP master curve are not "momentary," unquantified aging effect contaminate the data. The resulting master curve will not, in most cases, be useful to predic long-term data when used in combination with ETT. Furthermore, each of the individua curves must have the same age t_e, so that the TTSP master curve represents the behavior o the material at that age. Because of shifting, the TTSP master curve spans a broader tim span than allowed by the snapshot assumption. Since all the curves used in constructin the TTSP master curve comply with the snapshot assumption individually, the master curv represents the fictitious behavior of a material that does not age. This restricted and specifi interpretation of TTSP allows us to uncouple the influence of temperature and age on th creep behavior. It is only through the use of ETT (Eq. (7)) that the effect of aging is recovere

Tests are performed at temperatures ranging from 7°C to 70°C at chosen intervals s that the curves superpose. The testing method remains the same as described in Section until the sample is air quenched. After quenching for 10 min, the furnace is closed an the temperature is set to 40°C. The sample is allowed to stabilize at this temperature fo 10 min. Two creep tests, each a 1 min duration are performed. These tests are done in orde to mechanically condition the sample. After 45 min of aging time, the temperature of th furnace is set to the test temperature. When the physical age of the specimen reaches 60 mi

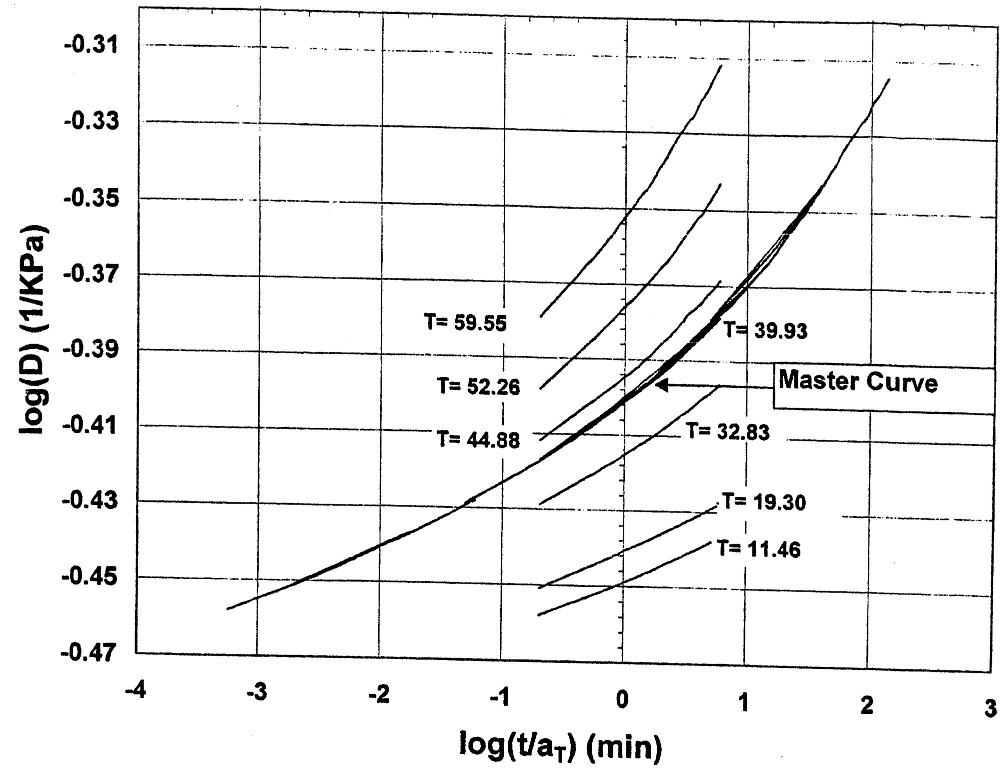

Figure 4. *Momentary creep curves at different temperatures, shifted to form TTSP master curve* ($T_R = 40°C$).

a creep test of 6 min duration is performed. The test is done so that it is a snapshot, in order to avoid aging effects during this creep test of duration λ (see Table 3).

A reference temperature is chosen and all the other momentary creep curves at different temperatures are horizontally shifted along the time axis using a shift factor, a_T, to form a master curve, as shown in Figure 4. A logarithmic plot of temperature T vs. shift factor a_T

Table 3
TTSP protocol

Step #	Action	Temp. (°C)	Time (min)	Furnace	Load
1	Annealing	$T_g + 15$	10	Closed	Off
2	Quench	Room	5	Open	Off
3	Age	40	5	Closed	Off
4	Mechanical Conditioning	40	1	Closed	On
5	Relaxation	40	3	Closed	Off
6	Mechanical Conditioning	40	1	Closed	On
7	Age	40	30	Closed	Off
8	Age	Test (*)	15	Closed	Off
9	Creep	Test (*)	$t_e/10 = 6$	Closed	On
10	Repeat			Closed	Off

(*) test temperature for TTSP between 7–70°C in 5–10°C increments.

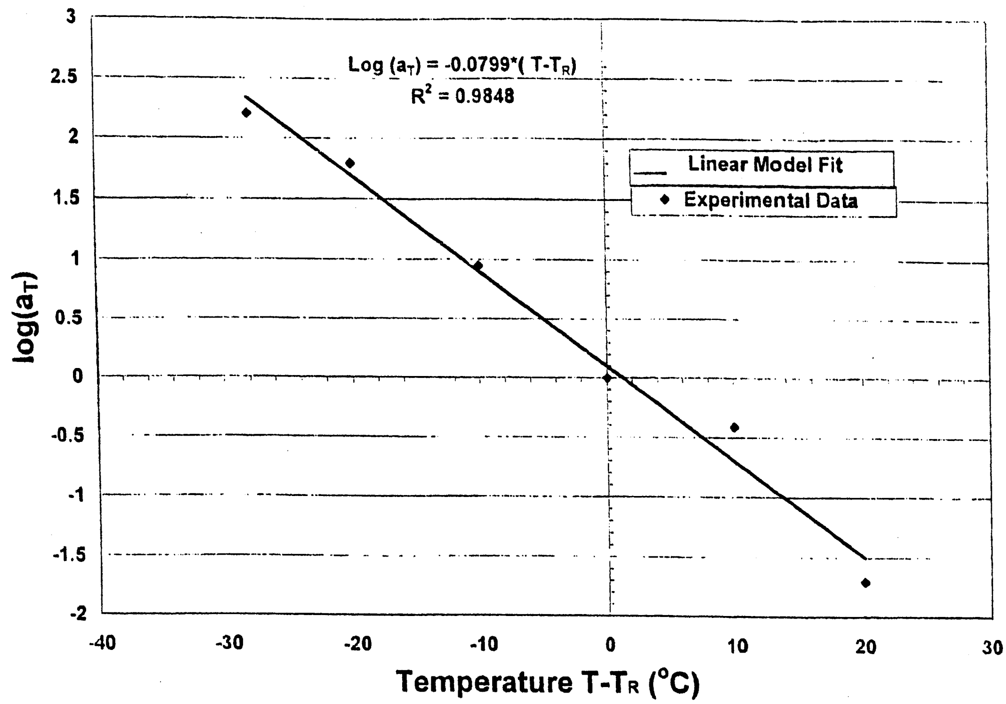

Figure 5. *Log*(a_T) *vs.* ($T - T_R$) ($t_e = 60$ *min*).

is plotted (Figure 5). This plot helps to determine the shift factor for any given temperature (Table 4). A linear model is fitted to the experimental data as

$$\mathrm{Log}(a_t) = \mu_T(T - T_R), \tag{4}$$

where μ_T is the shape. Using this model, the shift factor for any given temperature can be found out by using

$$a_T = 10^{\mu_T(T-T_R)}. \tag{5}$$

Since there are several specimens for the same material, each specimen has its own master curve. Also, the particular set of temperatures that make up a master curve vary from specimen to specimen. Thus, there is a scatter of master curves. Although the set of nominal test temperatures is the same for every specimen (say, 13, 20, 30, 40, 50, 60°C), the actual testing

Table 4
Temperature shift rate, μ_T, *for all materials at* $T_R = 40°C$, $t_e = 60$ *min*

Material	All specimens averaged	Individual specimen Min	Individual specimen Max	Number of specimens
F	−0.193	−0.199	−0.184	5
G	−0.140	−0.181	−0.128	5
H	−0.149	−0.167	−0.127	5
I	−0.088	−0.103	−0.093	5
J	−0.080	−0.087	−0.077	5

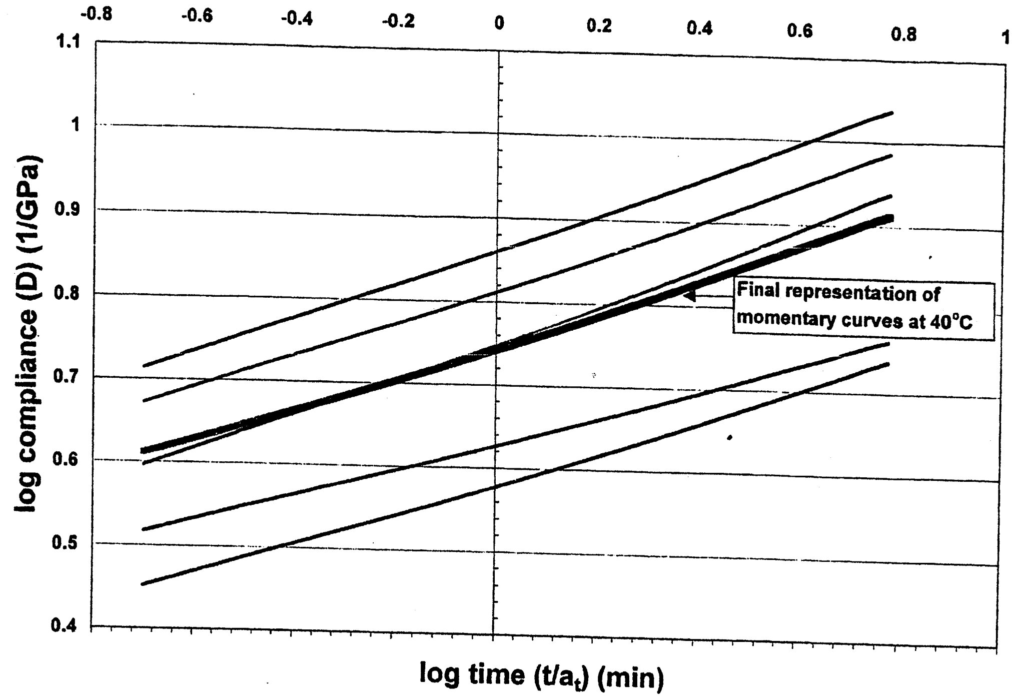

Figure 6. *Typical variability of creep compliance.*

temperature varies from specimen to specimen. Therefore, in trying to average the data, the first step is to shift individual tests to nominal temperatures using the shift factor plot of each specimen. Each shifted curve is fitted with a power law (Eq. (1)). Next, take the average of each D_o, D_1, and n as representation of the material behavior at each nominal temperature (Figure 6). Repeat this procedure for every nominal temperature. Then shift the curves to the reference temperature T_R with shift factor a_T. Thus, creating an averaged TTSP master curve that represents all the data from multiple specimens (Figure 7). Another advantage of the proposed procedure is that the scatter of the data can be conveniently displayed, as in Figure 7.

An alternative procedure to incorporate data from several specimens could be to fit an equation to the individual specimen master curve, then average them. But, such a procedure would require assuming a model equation for the master curve. In addition, there would be no rational way to average the shift factor plots of several specimens, because the testing temperatures and therefore the individual shift factors a_T are not consistent from specimen to specimen.

§4. EQUIVALENT TIME THEORY

According to Equivalent Time Theory (ETT) [6], the unaged time λ in the master curve is related to real time as

$$\lambda = \frac{t_e}{\alpha}[(1 + t/t_e)^{\alpha} - 1] \quad \text{for } \mu \leq 1, \tag{6}$$

where t_e is the age of the sample when the test started, μ is the shift rate, $\alpha = 1 - \mu$, and t is the time. Inverting Eq. (6), we can stretch the unaged time λ of the master curve into real time as

$$t = \left[\frac{\lambda\alpha}{t_e} + 1\right]^{\frac{1}{\alpha}} - 1. \tag{7}$$

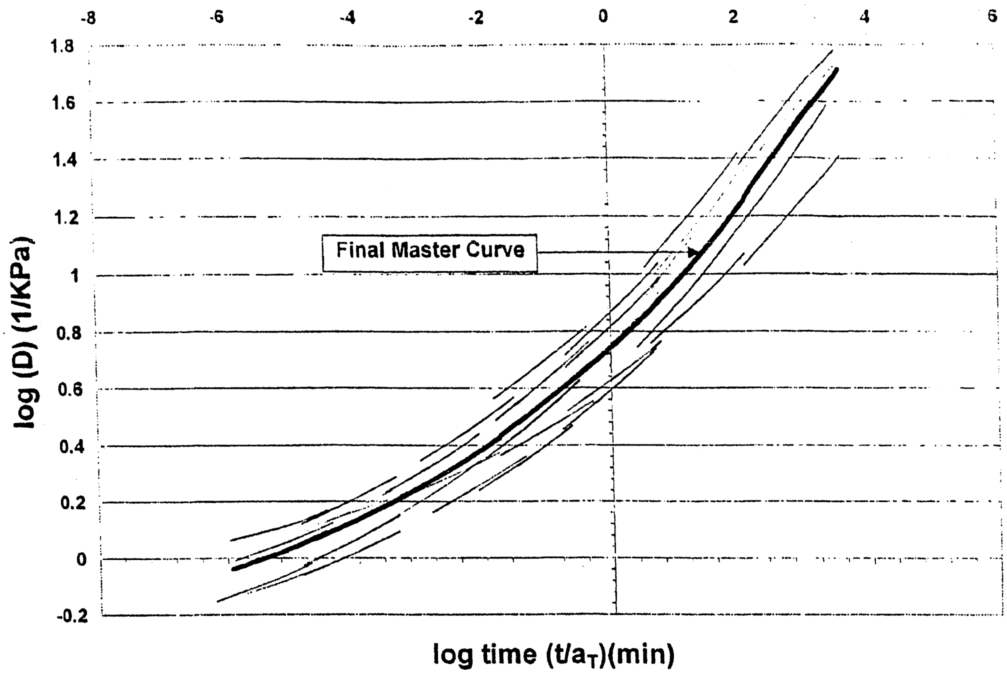

Figure 7. *Final representation of momentary creep curves of 5 specimens at 40°C.*

Using the aging shift factor rate (Table 2), the TTSP master curve (Figure 7), the temperature shift factor rate (Table 4), and Eq. (7) the long-term compliance for every material is predicted at $T_R = 40°C$ and compared with 32,000 minute data from creep bending tests (CBT) [10] and 10,000 minute bending tests performed in the DMA (Figure 8). Due to the uncertainty of the initial position of the displacement transducer of the DMA 7e when operated in creep mode, the 10,000 minute curves were vertically shifted to coincide

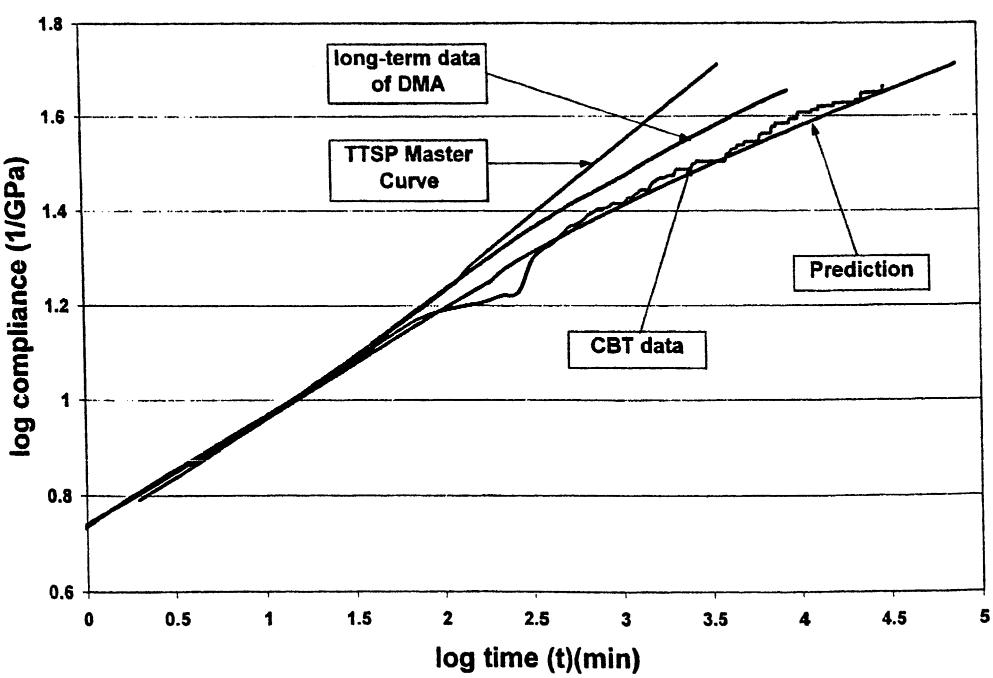

Figure 8. *Comparison of prediction with CBT data and DMA long-term data.*

at a time of 1 min with the average TTSP. It can be seen that the combined TTSP and aging data can be used to accurately predict CBT data obtained in a different fixture using much larger samples (ASTM D790) over 32,000 minutes [10].

Each specimen is repeatedly annealed, quenched, and aged, so that all the creep curves at various temperatures have the same age t_e. Such a procedure assumes that the same specimen can be rejuvenated (by annealing) multiple times without permanent chemical changes. In order to prove that the specimens do not undergo any change in properties due to chemical aging, the specimens are tested for repeatability. A number of tests are done at 40°C on the same specimen. The specimen is taken through the full testing process again and again. A total of 15 tests are done on the specimens and the results show that the data have very slight variation [8, 14]. It is concluded that annealing can rejuvenate the specimen, and there is no chemical aging.

Rejuvenating relies on annealing to erase all previous physical aging. Some uncertainty exists as to how much time is necessary to anneal a specimen. Annealing time is the length of time that the specimen remains at the annealing temperature, which in this work, is about 15°C above the glass transition temperature. Creep tests are done on a specimen annealed for different lengths of time (10, 30, 60, and 120 min) and aged for the same period of time, i.e., 60 min. There is no significant change in compliance due to annealing time [8], implying that equilibrium is attained within 10 min at the annealing temperature $T_g + 15$°C.

The transition towards thermodynamic equilibrium takes place after quenching at a temperature below the glass transition temperature. For the aging study, specimens are aged at the same temperature, which coincides with the testing temperature (40°C in this study). For TTSP, testing temperature changes with every test. There was a question of whether the specimen should be aged at a single aging temperature or at the various testing temperatures. Master curves of specimens that are conditioned at various test temperatures, dictated by the TTSP method, are compared to master curves of specimens that are conditioned at 40°C and then taken to the test temperatures 10 min before testing. Since the two master curves are virtually identical [8], it is concluded within the parameters of this study, that compliance is affected by the temperature at which the specimen is tested and not by the conditioning temperature. For convenience, all specimens are aged at 40°C, then taken to testing temperature 10 min before the test.

Since the polymers for trenchless rehabilitation operate in a wet environment, it was decided to investigate if water had any effect on the material behavior. Three specimens of each sample are immersed in water and continuously monitored for any weight change. The percentage of weight change recorded is insignificant. Hence, it is concluded that moisture does not play a significant role in the long-term creep behavior of the polymers used in this study [8].

§5. Prediction of long-term encased-liner data

In order to predict 35,000 h of encased liner data [11], the master curve must be shifted to the age and temperature of the encased liners. The average TTSP master curve (Figure 7) was obtained at $T_R = 40$°C and age $t_e = 60$ min. The age of the encased liners at the onset of full-size testing is known [11]. For an encased liner, age zero is the time of installation in the pipeline. Then ETT was applied on the TTSP master curve to predict the long-term compliance of the encased liners.

The master curve is composed of a number of curves, which are initially shifted to a reference temperature $T_R = 40$°C to form the TTSP master curve at 40°C. Shifting to the encased-liner test temperature is done by shifting the whole master curve from 40°C to 21°C, using the shift factor, a_T (T), discussed earlier. Also, the aging master curve was of the samples (1.8 m long, 0.3 m diameter pipe), field fabrication encased in imperfect, welded steel pipe, etc. [11]. Despite the significant differences between these studies, the present study, using very small samples, tested only a few minutes, while the other full-size encased liner tested over 35,000 h [11]. Qualitative agreement is favorable, especially taking into account how inaccurate the master curve is in Figure 9, when aging effects are not considered. Note that the master curve and the prediction are very close in Figure 10, because material I experiences little physical aging, as indicated by a small value of μ in Table 2.

§6. Conclusions

Aging momentary curves must be rotated prior to horizontal shifting. Multiple specimens should be used and averaged as per procedure, as described herein, to determine the aging shift factor μ. Individual TTSP curves must be momentary to avoid aging effects. Multiple specimens should be used and averaged as per procedure, as described herein, to obtain the averaged TTSP master curve. Specimens were mechanically conditioned in order to obtain reliable results. Under these conditions, ETT predictions compare well with long-term bending data and long-term encased liner data.

Acknowledgements

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