

# A LABORATORY INVESTIGATION OF THERMO-MECHANICAL PROPERTIES OF POLYMER-MODIFIED ROOFING BITUMENS

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**P**olymer-modified bitumens for low-slope roofing were introduced commercially in Europe, where earliest installations date from approximately 1961-65.<sup>1</sup> Use of polymer-modified bitumen roofing membranes in the United States is more recent, 1976-78<sup>1</sup> and market demand for these materials is strong.

This report describes test development and research sponsored by the National Roofing Foundation to characterize the low temperature thermo-mechanical behavior of selected polymer-modified bitumens and a conventional Type III asphalt. The research was conducted at Clemson University in 1984.

## Nomenclature and Standards for Polymer-Modified Bitumens

Two polymers now widely used to modify roofing bitumens are atactic polypropylene (APP), widely used in Italy, and styrene-butadiene-styrene copolymer (SBS), widely used in France.<sup>2, 3, 4</sup> Synthetic and natural rubbers, uncured or vulcanized, also are used in modified bituminous membranes<sup>5</sup>. One of these, styrene-butadiene rubber (SBR), is a random copolymer compounded as a latex and then added to bitumen. SBR is not yet very widely used in polymer-modified bitumens in the United States.

APP, an amorphous homopolymer, was probably the first polymer used commercially in roofing membranes. It is an inexpensive by-product of isotactic polypropylene synthesis<sup>2, 6</sup> and is reported to have a good service history in Italy<sup>2</sup>. Satisfactory properties are generally achieved with about 25 to 35 percent, by weight, APP in the blend.<sup>6,7</sup> APP-modified bitumen exhibits excellent resistance to creep at elevated service temperatures. However, the product is reported to be somewhat brittle at low temperatures, -17C (0F), for example.<sup>7</sup> APP-modified bitumens have been reported to offer greater resistance to weathering from ultraviolet radiation than blends modified by SBS.<sup>6, 8</sup>

SBS is a block copolymer of two polymers with significantly different glass transition temperatures.<sup>9</sup> A unique network is formed at service temperatures, in which glassy polystyrene blocks are connected by flexible chains of polybutadiene. At service temperatures, SBS behaves like a crosslinked polymer, yet it dissociates as a thermoplastic polymer upon heating. Piazza, et al.,<sup>7</sup> reported that nominal quantities of about 7 to 10 percent SBS give the best properties, although Kraus<sup>9</sup> recommended 12 to 14 percent to ensure continuity of the rubber-rich phase. SBS-modified bitumens exhibit excellent flexibility at low temperatures, yet are more susceptible than APP blends to creep at higher temperatures.<sup>7</sup> SBS-modified bitumens must be protected from ultraviolet radiation by a reflective surface, such as mineral aggregate. Unprotected SBS bitumens are more

resistant to weathering than conventional surfaced roofing bitumen.<sup>8</sup>

Performance standards for modified bituminous roofing membranes have been developed by the Canadian General Standards Board (CGSB),<sup>10</sup> the Midwest Roofing Contractors Association (MRCA),<sup>11</sup> and producers of modified bitumens.<sup>12</sup> The MRCA recommendations rely heavily on the testing methods defined in the CGSB Standard and also incorporate several original features, including a test procedure for measuring thermally-induced loads in the range of 70 to -30F. Producers<sup>12</sup> have defined minimum requirements on the basis of anticipated service conditions, especially with regard to temperature and loading time.

The NRF-sponsored research project conducted at Clemson addressed fundamental characterization of mechanical behavior of modified bitumen specimens. Principal emphasis was placed on thermal loads and elongation at low temperatures.

## Mechanics of Thermally-Induced Stresses

Mechanical behavior of elastic materials is commonly described by Hooke's law, in which stress response is assumed to be proportional to strain. Isotropic materials with a constant coefficient of thermal expansion,  $\alpha_0$ , have the following form for strain,  $\epsilon_x$ <sup>13</sup>:

$$\epsilon_x = \frac{1}{E}(\sigma_x - \nu(\sigma_y + \sigma_z)) + \alpha_0 T \quad (1)$$

where:  $\sigma_x, \sigma_y, \sigma_z$  = stress components along the x, y, and z-axes respectively,

E,  $\nu$  = elastic modulus and Poisson's ratio, respectively,

T = temperature change, considered positive for a temperature increase.

For a uniaxial tension member subjected to a temperature decrease and restrained from contraction, thermally-induced stress is reduced to:

$$\sigma_x = E\alpha_0 T \quad (2)$$

The thermal shock factor defined by Mathey and Cullen<sup>14</sup> was based on an earlier analysis by Cullen and Boone<sup>15</sup> that indicated membrane thermal stresses could be estimated by equation 2. Researchers have shown, however, that bitumen behavior depends on duration and rate of loading and on temperature.<sup>16</sup>

Behavior of viscoelastic materials, including some types of bitumens, is sometimes described by the Boltzmann superposition principle, which states that the resultant creep strain,  $\epsilon(t)$ , caused by a series of stress increments equals the sum of the products of each stress increment, and the creep

compliance evaluated for the interval of each stress increment:<sup>17</sup>

$$\epsilon(t) = \sigma_0 J(t) + \Delta\sigma_1 J(t - \tau_1) + \dots + \Delta\sigma_n J(t - \tau_n)$$

where:  $\sigma_0, \Delta\sigma_1, \Delta\sigma_n$  = initial stress, first, and  $n$ th stress increments, respectively, applied at times, 0,  $\tau_1$  and  $\tau_n$ , respectively,

$J(t), J(t - \tau_1), J(t - \tau_n)$  = creep compliance, evaluated in the time frames of each stress.

For a continuous loading process, the Boltzmann superposition principle becomes a convolution integral.

For some polymers, creep data measured at various isotherms yield a family of parallel curves when the creep compliance is plotted against time on logarithmic scales. Thus, the time,  $t_r$ , required to attain a certain value of creep compliance at one temperature may be computed from the product of a shift factor,  $a_T[T]$ , and the time  $t_R$ , required to reach that same compliance at some arbitrary reference temperature:

$$J(t_r) = J(a_T[T]t_R).$$

A reduced time scale may therefore be defined as equivalent to the time scale for the reference temperature:

$$t_R \equiv \xi = t_r / a_T[T] \quad (3)$$

The shift factor  $a_T[T]$  is assumed to vary only with temperature. Materials which obey this reduced time relationship are thermorheologically-simple materials. Some bitumens obey this principle of time-temperature superposition.<sup>16</sup>

The extension of time-temperature superposition to transient temperature conditions has been discussed in detail by Christensen.<sup>18</sup> Thermally-induced stress,  $\sigma(t)$ , in a prismatic, linearly viscoelastic material restrained longitudinally during a temperature decrease is given by:

$$\sigma(t) = \int_0^t E[\xi(t) - \xi(\tau)] \frac{\partial}{\partial \tau} (\alpha_0 T[\tau]) d\tau \quad (4)$$

where:

$\xi(t) - \xi(\tau) = \int_{a_T[T(t')] }^1 dt' =$  reduced time evaluated for a prescribed temperature history,  $T(t')$ ,

and:  $E[\xi(t) - \xi(\tau)]$  = stress relaxation modulus evaluated for the argument,  $[\xi(t) - \xi(\tau)]$ ,

$\alpha_0$  = coefficient of thermal expansion,

$T[\tau]$  = temperature change as a function of the dummy variable of integration,  $\tau$ .

Bonafont<sup>19</sup> used the viscoelastic formulation to estimate rupture temperatures for bitumen restrained from movement in the plane of the bitumen.

Nonlinear mechanical behavior also may be described by a strain-dependent modulus,  $F(\epsilon)$ , in the form:

$$\sigma = F(\epsilon)\epsilon. \quad (5)$$

Pommersheim and Mathey<sup>20</sup> found that data measured for some conventional membrane components could be described by equation 5 by approximating the strain-dependent modulus with an exponential function. Nonlinear viscoelastic behavior is sometimes described by two separable functions, the linear viscoelastic function and a strain-dependent function.<sup>21</sup> Hence, for the stress relaxation problem:

$$\sigma = G(\epsilon)E(t) \quad (6)$$

where:  $G(\epsilon)$  = function of strain that approaches the strain at small deformations,

$E(t)$  = stress relaxation modulus.

## TESTING PROGRAM

Two modified bitumens, designated MB-I and MB-II, were tested using tension tests conducted on 1/2-inch diameter cylindrical specimens of 2-inch test length. Specimen ends were tapered to reduce stress concentrations from the grips. Grips were designed to prevent specimen slippage.

Modified bitumens can undergo appreciable elongation so the definition of natural, or true, strain was used:<sup>22</sup>

$$\epsilon = \ln(g/g_0)$$

where the argument,  $g/g_0$ , is the ratio of instantaneous to initial gage lengths. A linear correlation was observed between crosshead displacement,  $x$ , and the gage ratio, so that strain was calculated indirectly by the relation

$$\epsilon = \ln((L_e + x)/L_e) \quad (7)$$

where  $L_e$  represents a constant effective gage length.

The stress on the actual cross-section was calculated by:

$$\sigma = (P/A_0)e^{2\nu\epsilon} \quad (8)$$

where  $\sigma, \epsilon$  = stress and strain, respectively,

$P/A_0$  = nominal stress, based on original area of the specimen,

$\nu$  = Poisson's ratio, measured as 0.5 for MB-I and MB-II.

For small strains, on the order of 5 percent or less, equations 7 and 8 reduce to the nominal definitions for strain and stress commonly used to describe elastic behavior.

Stress data for modified bitumen, MB-I, were analyzed to formulate a constitutive equation for this material. For linear viscoelastic behavior, the stress response,  $\sigma(t)$ , to a uniform rate of strain,  $R$ , at isothermal testing conditions should obey the equation:

$$\sigma(t) = \int_0^t E(t - \tau) (R) d\tau \quad (9)$$

By applying properties of Laplace transformation, the stress relaxation modulus,  $E(t)$ , can be shown to be the time derivative of the ratio of stress to uniform strain rate:

$$E(t) = d/dt[\sigma(t)/R]. \quad (10)$$

Accordingly, an increase in strain rate must produce an equivalent increase in stress at common values of time in the range for which linear viscoelasticity is applicable. If the ratio,  $\sigma(t)$  to  $R$ , varies linearly with time, then relaxation does not occur, and the material may be described by Hooke's law.

A testing machine was designed to extend specimens at exponential rates so that rates of natural strain would be constant for each tensile test. An environmental chamber permitted load-deformation characterization at temperatures ranging from 28 to -40C (82 to -40F). Specimens were immersed in an ethylene glycol bath which was cooled by refrigerant circulated through copper coils located at the bottom of the test chamber. Air was bubbled up past the coil system to mix the bath medium thoroughly.

Thermal splitting tests were conducted for specimens of Type III asphalt and modified bitumens, MB-I and MB-II. Specimens were 23 1/2 inches long, with a prismatic test region of 20x1/2x1 1/2 inches. A test chamber was constructed

for restraining specimens at essentially constant length. Specimens were immersed in a bath of methanol, and then subjected to a rapid temperature decrease by bubbling liquid nitrogen from the bottom of the tank.

The coefficient of linear thermal expansion was measured for specimens of polymer-modified bitumen, MB-I, using a quartz-tube dilatometer (ASTM D696). Specimens of 3/16-inch diameter and 2 inches long were tested in the bath chamber constructed for thermal splitting experiments.

## RESULTS AND DISCUSSION

Modified bitumen, MB-I, exhibited plastic behavior, as shown in Figure 1. Rupture stress increased with decreasing temperature and with increasing rates of strain for specimens tested at or above  $-17\text{C}$  ( $+1\text{F}$ ). Specimens tested at  $-30$  and  $-40\text{C}$  ( $-22$  and  $-40\text{F}$ ) ruptured before attaining a plateau of plastic flow.

Plots of the ratio of stress to strain rate against time did not superpose data measured at common temperatures, as shown in Figure 2. Therefore stress relaxation did not obey the theory of linear viscoelasticity for the range of strain rates used to test specimens of MB-I. Time-independent initial moduli were calculated from the slope of the stress-strain curves (Table I). The following equation was used to describe temperature dependence of the data:

$$E_t = 2220 - 89.1(T) + 1.16(T^2), \quad \text{psi, for } (-31\text{C} \leq T \leq 28\text{C}). \quad (11)$$

This equation then was used to predict thermally-induced stresses developed in specimens of MB-I subjected to the thermal loading test. Additional tests will be needed to measure the strain-rate dependence of these moduli.

The coefficient of thermal expansion was calculated for material MB-I from the slope of the contraction strain-versus-temperature curve (Figure 3). Because a change in slope occurs at the material's glass transition temperature,<sup>17</sup> the coefficient of thermal expansion was estimated to be 0.00018 inches per inch per degree C and 0.00011 inches per inch per degree C above and below the glass transition temperature, approximately  $-17\text{C}$  ( $+1\text{F}$ ), respectively.

Specimens of MB-II bitumen sustained large extensions, even at cold temperatures. Data in Figure 4 were truncated at a strain of 1.50 because the linear relationship between crosshead displacement and gage ratio (equation 7) did not apply at higher extensions. Specimens ruptured only at test temperatures of  $-31$  and  $-40\text{C}$  ( $-24$  and  $-40\text{F}$ ). The upward concavity of the stress-strain curves at high extensions indicates that the material may have a nonlinear constitutive law of the form given by equation 6. Rupture data for materials MB-I and MB-II are compared in Table II.

A sample of Type III asphalt having a 96C softening point and 16 pen was obtained to provide a reference for comparison between thermally-induced loads for conventional roofing bitumen and those for polymer-modified bitumens, MB-I and MB-II. The creep compliance of Type III asphalt also was estimated from Van der Poel's nomograph,<sup>16</sup> using a value of 3.7 for the penetration index. Values of the stress relaxation modulus were calculated from the creep compliance by an approximate method used by Aklonis and Tobolsky.<sup>21</sup> These values are shown in Figure 5 for various temperatures, and as a master curve at  $-4\text{C}$  ( $25\text{F}$ ) in Figure 6. The shift function has been plotted against temperature in Figure 7. The following functions thus were derived from

the stiffness nomograph for the stress relaxation modulus,  $E_{rr}(\xi)$ , and shift factor,  $a_r[T]$ , respectively:

$$E_{rr}(\xi) = 362,500, \text{ psi, for } (0 \leq \xi \leq 6.19 \times 10^{-8} \text{ sec})$$

$$E_{rr}(\xi) = 19130(\xi)^{-2.791 - 0.006137 \ln \xi}, \text{ psi} \quad (12)$$

$$\text{for } (6.19 \times 10^{-8} \leq \xi \leq 1.8 \times 10^8 \text{ sec})$$

and  $a_r[T] = 0.3283 e^{-0.301T}$  (13) for reduced time,  $\xi$ , in seconds and temperature,  $T$ , in degrees C.

Results of the thermal splitting experiment are shown in Figures 8 and 9 for the two polymer-modified bitumens and for the Type III bitumen, respectively. Specimens of modified bitumen, MB-II, did not rupture, even when subjected to temperatures as low as  $-81\text{C}$  ( $-114\text{F}$ ). Specimens of modified bitumen, MB-I, ruptured at temperatures in the range of  $-44$  to  $-55\text{C}$  ( $-47$  to  $-67\text{F}$ ). Specimens ruptured near the gripping blocks. Therefore, lower temperatures might have been attained in the absence of such local stress concentrations. The solid line shown with the data for specimens of MB-I corresponds to the stress increase estimated indirectly from the modulus, equation 11, and coefficient of thermal expansion by the equation:

$$\sigma = (2220 - 89.1(T) + 1.16(T^2)) e, \text{ psi, for } (T \geq -31\text{C}) \quad (14)$$

where

$$0.00018(T_0 - T), \text{ for } (-17\text{C} \leq T \leq T_0)$$

$e =$

$$0.00018(T_0 + 17) + 0.00011(-17 - T), \text{ for } (-31\text{C} \leq T < -17\text{C}).$$

Type III asphalt specimens ruptured at temperatures ranging from  $-20$  to  $-30\text{C}$  ( $-4$  to  $-22\text{F}$ ). Untapered specimens ruptured near the face of the gripping blocks, whereas specimens which were tapered to a thickness of  $1/4$ -inch ruptured in the test region. Rupture temperatures measured for all specimens were consistent. The solid line shown in Figure 9 corresponds to the numerical solution of the stress relaxation equation for a linear temperature decrease:

$$\sigma = \int_0^t E[\xi(t) - \xi(\tau)] (\alpha_0 m) d\tau, \text{ psi} \quad (15)$$

where

$E[\xi(t) - \xi(\tau)] =$  relaxation modulus evaluated for the reduced time argument,  $[\xi(t) - \xi(\tau)]$ , equation 12

$$\xi(t) - \xi(\tau) = 10.12 e^{0.301T_0} (e^{-0.301m\tau} - e^{-0.301mt}) / m, \text{ cf. equations 4 and 13}$$

$T_0 =$  initial temperature,  $21\text{C}$

$m =$  rate of temperature decrease,  $0.068\text{C/sec}$ .

$\alpha_0 =$  coefficient of thermal expansion, assumed to be 0.0002 per degree C (19).

The dashed line in Figure 9 corresponds to Bonafont's solution,<sup>19</sup> divided by a factor of two to account for uniaxial restraint:

$$\sigma = 18.75 m^{0.4} e^{-0.104T_0} (e^{0.104(T_0 - T)} - 0.757), \text{ psi} \quad (16)$$

where the rate,  $m$ , and initial temperature,  $T_0$ , were taken as  $0.068\text{C}$  per second and  $21\text{C}$ . Close agreement noted between equations 15 and 16 is expected since both are based on the Van der Poel stiffness nomograph.<sup>16</sup> The amount by which equations 15 and 16 overestimate the thermally-induced stress data is considerable, however, and may be caused by assumption of an inaccurate coefficient of thermal expansion for bitumen. Baastrup-Larsen<sup>24</sup> has cited a value of ap-

proximately 0.00012 per degree C for bitumen below the glass transition temperature.

Rigden and Lee<sup>25</sup> found that bitumens approach a constant breaking strength when tested below the Fraass brittle temperature, rupturing at a hydrostatic tension (mean of principal normal stresses) value of about 6 to 10 Kg/cm<sup>2</sup>. For uniaxial tension, the data from Rigden and Lee correspond to ultimate strengths of about 250 to 430 psi (18 to 30 Kg/cm<sup>2</sup>). However, de Bats, et al.,<sup>12</sup> concluded that the critical thermal stress for roofing bitumens ranges from about 72 to 20 psi (500 to 140 KN/m<sup>2</sup>), depending on the penetration index. Rupture stresses measured for Type III asphalt specimens subjected to the thermal splitting test were approximately 18 to 52 psi.

## SUMMARY AND CONCLUSIONS

Testing procedures and equipment were developed for measuring mechanical properties of polymer-modified roofing bitumens at cold temperatures and large extensions. The two modified bitumens exhibited different types of stress-strain curves, plastic behavior for MB-I and stiffening at large extensions for MB-II. The behavior of these materials, in the range of strain rates tested, could not be modeled using linear theory of viscoelasticity because stress relaxation was not evident in the stress-strain curves. These strain rates, however, were rapid relative to magnitudes of strain rate anticipated for roofs in service, and linear viscoelasticity may be applicable at infinitesimally slow rates of strain.

Heat capacity was measured as a function of temperature by differential scanning calorimetry for the Type III asphalt and the two polymer-modified bitumens. The onset temperature of the glass transition for the Type III asphalt was approximately -10C (14F). However, distinct discontinuities were not evident in the heat capacity curves for the two polymer-modified bitumens, so that differential scanning calorimetry did not detect the glass transition of these materials. Dilatometry measurements and stress-strain data indicated a glass transition for material MB-I at approximately -17C (+1F).

A low-temperature thermal splitting test was developed to compare rupture temperatures of the polymer-modified bitumens with those of a sample of Type III roofing asphalt. The polymer-modified bitumens exhibited much greater resistance to low-temperature cracking than the Type III asphalt. One type of modified bitumen did not break when subjected to temperatures as low as -81C (-114F), whereas specimens of Type III asphalt ruptured in the range of -20 to -30C (-4 to -22F).

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Test Temperature, T, degrees (C)	Test Temperature, T, degrees (F)	Strain Rate, R, Sec <sup>-1</sup>	Modulus, E <sub>r</sub> <sup>1</sup> , psi	Modulus Value from Eq. (11), psi
28	(82)	0.0082	709	
28	(82)	0.0025	613	635
11	(52)	0.0022	1493	1380
11	(52)	0.00043	1110	
0.5	(33)	0.0023	2452	2180
0.5	(33)	0.00092	2049	
-17	(1)	0.0024	4296	4070
-16.5	(2)	0.00043	3780	
-31	(-24)	0.0026	6146	6100
-31.5	(-25)	0.00044	6117	
-40.5	(-41)	0.00043	5648	
-41	(-42)	0.00042	5919	

<sup>1</sup>The modulus was defined as the slope of a least squares regression line through the linear portion of the stress-strain curve.

Table I Initial Modulus for MB-I Specimens

Material	TEST CONDITIONS		PROPERTIES AT BREAK					
	Temperature, T degrees C (F)		Rate, R, Sec <sup>-1</sup>	Load, P, lbs.	Extension, (100x)/L <sub>0</sub> , percent	Strain, $\epsilon$	Stress, $\sigma$ , psi	Strain Energy Density, psi <sup>1</sup>
MB-I	-17	(+1)	0.0024	75	46	0.38	600	190
MB-I	-16.5	(+2)	0.00043	56	46	0.38	450	160
MB-I	-31	(-24)	0.0026	154	20	0.18	1020	100
MB-I	-31.5	(-25)	0.00044	165	22	0.20	1100	110
MB-I	-40.5	(-41)	0.00043	116	16	0.15	730	60
MB-I	-41	(-42)	0.00042	143	19	0.17	920	80
MB-II	-19.5	(-3)	0.0011	> 71	> 330	> 1.46	> 1550	> 790
MB-II	-19.5	(-3)	0.0018	> 70	> 320	> 1.44	> 1520	> 790
MB-II	-31	(-24)	0.0011	150	300	1.38	3030	1500
MB-II	-32	(-26)	0.00092	148	310	1.40	3060	1500
MB-II	-40	(-40)	0.00086	196	190	1.08	2930	1800
MB-II	-40	(-40)	0.00084	177	130	0.82	2060	1200

<sup>1</sup>Strain energies were estimated by measuring the area under each stress-strain curve with a planimeter.

Table II Rupture data of MD-I and MB-II polymer-modified bitumens at cold temperatures

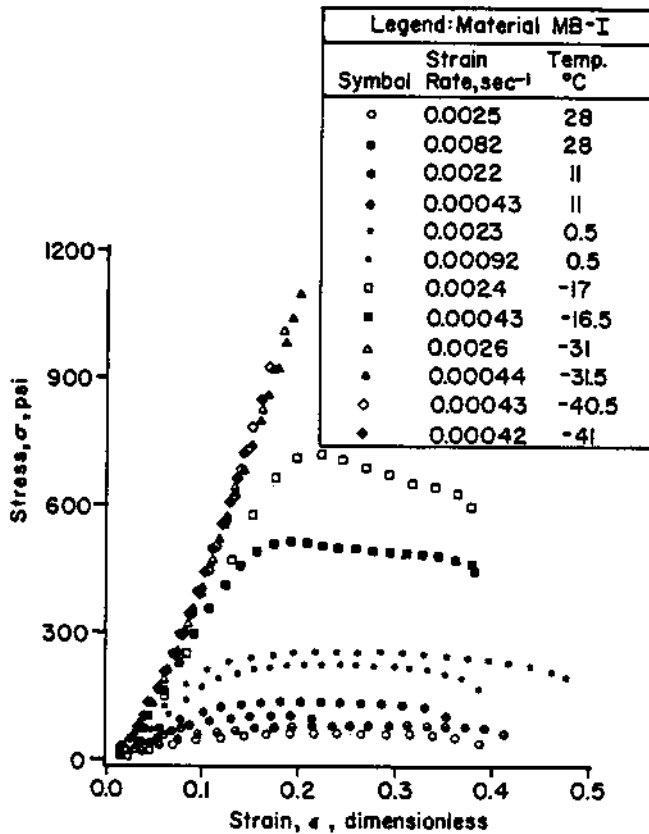


Figure 1 Stress-strain curves for material MB-I

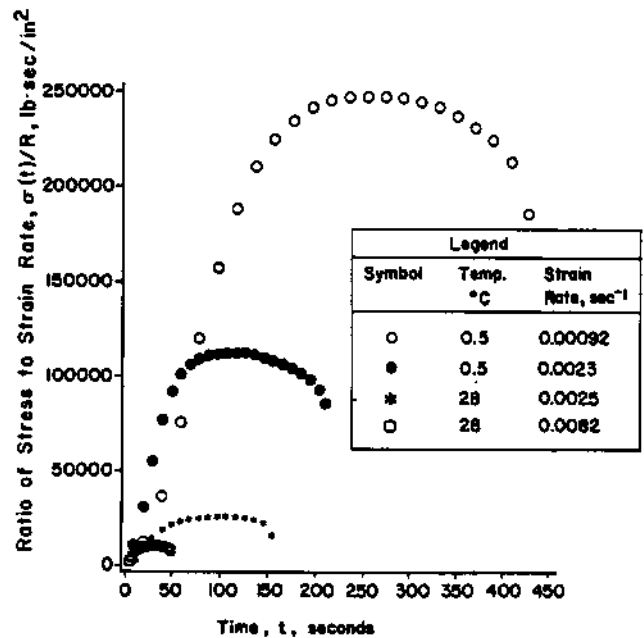


Figure 2 Stress data versus time for material MB-I

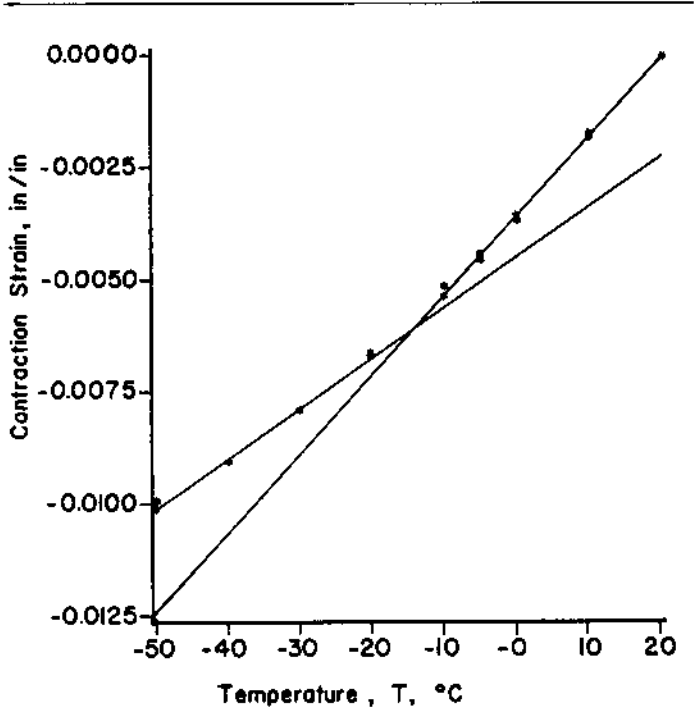


Figure 3 Thermal contraction data for material MB-I

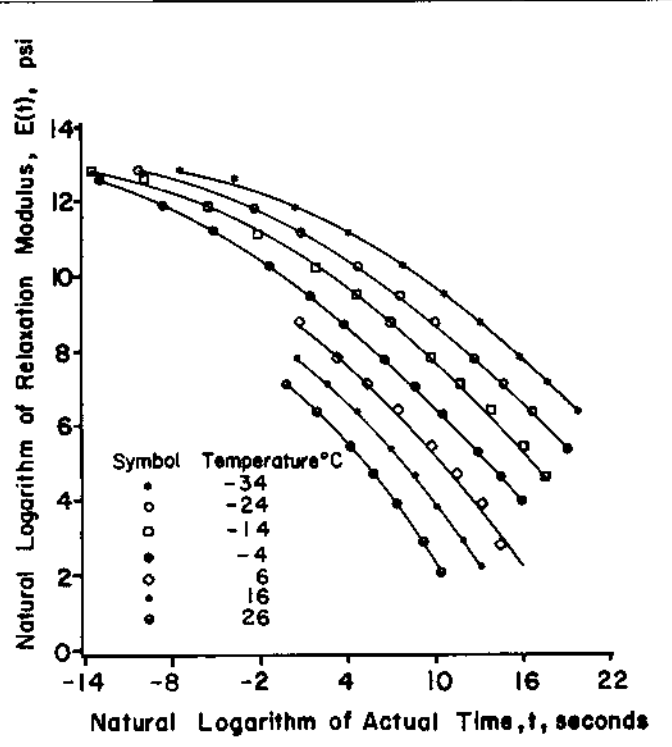


Figure 5 Relaxation modulus of Type III asphalt at various temperatures (from nomograph [16])

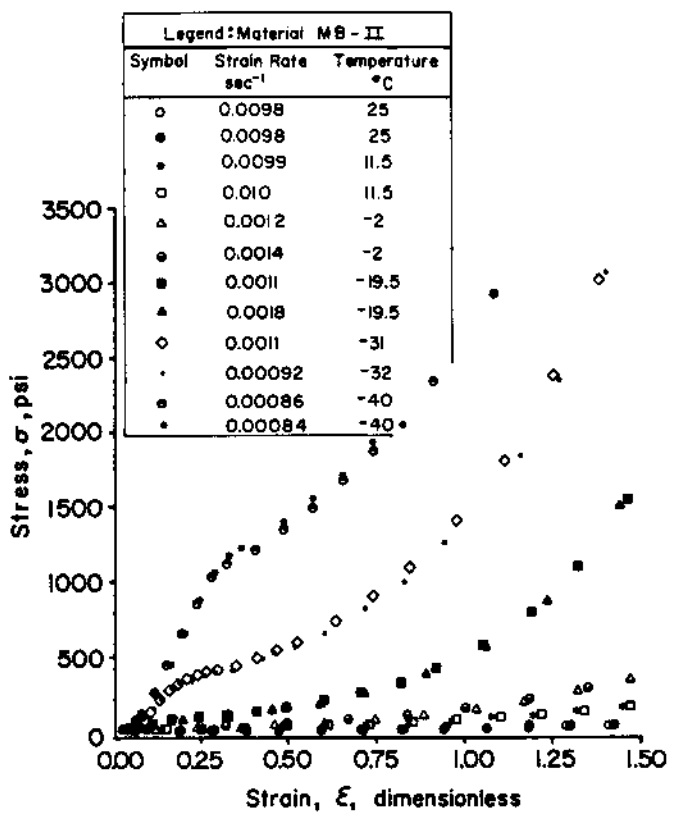


Figure 4 Stress-strain curves for material MB-II

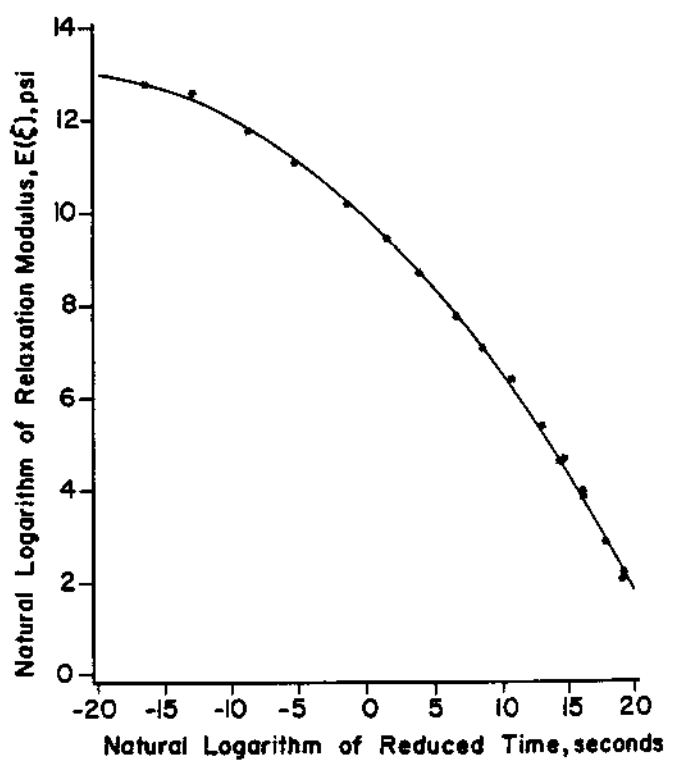


Figure 6 Relaxation modulus of Type III asphalt versus reduced time

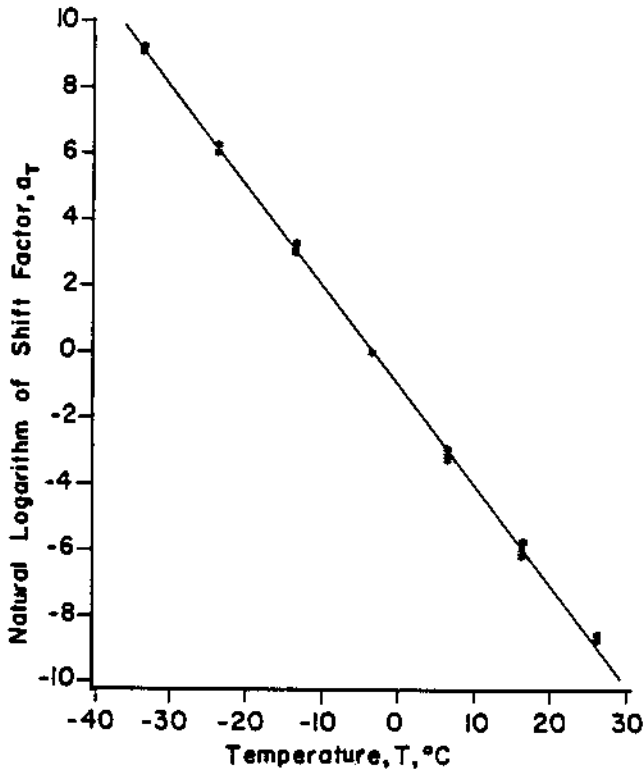


Figure 7 Shift factor for Type III asphalt (from nomograph [16])

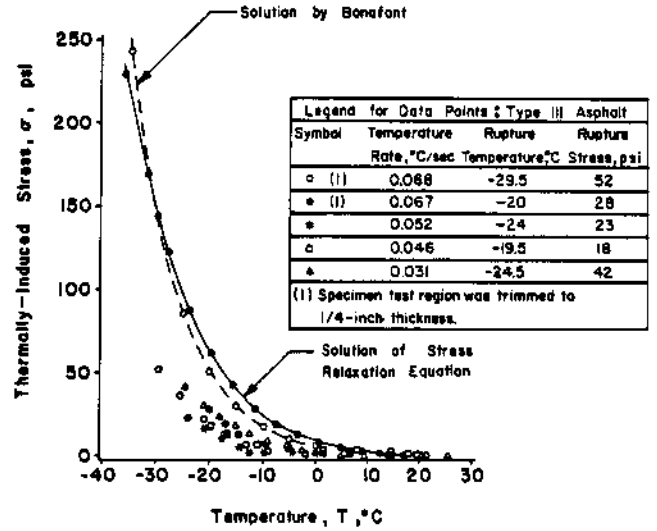


Figure 9 Stress induced by temperature changes for Type III asphalt

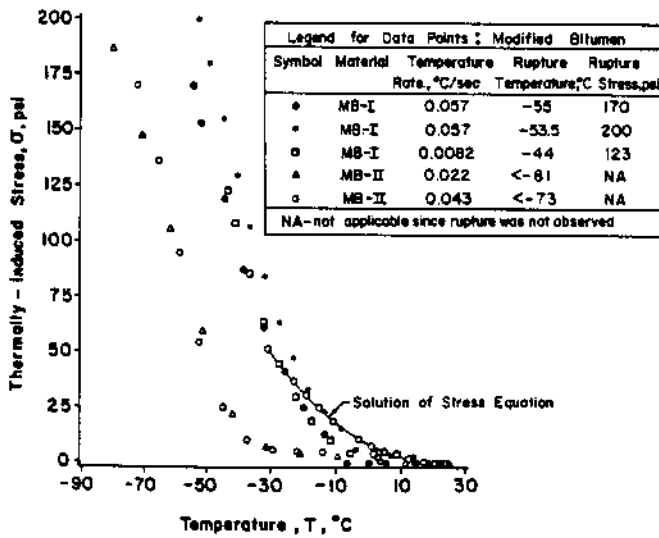


Figure 8 Stress induced by temperature changes for MB-I and MB-II