STATE OF THE ART REPORT 7

Low-Temperature Properties of Paving Asphalt Cements

Transportation Research Board
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Committee on Characteristics of Bituminous Materials

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Introduction and Scope

It is generally known that the behavior of asphalt paving mixtures under conditions of induced stress is affected by the response of the asphalt cement in the mix. During the service life of the pavement, asphalt is exposed to low temperatures, which tend to alter its rheological response. It is important to study the low-temperature rheology of asphalts to gain a better understanding of the complex factors that affect the low-temperature behavior of flexible pavements.

A storehouse of information and research data pertaining to low-temperature properties of paving asphalts exists in the literature. However, much of the data is fragmented and scattered.

This brief state-of-the-art report provides as many references as possible for each test parameter so that the reader can retrieve pertinent information in a specific area of interest.

Although tests on asphalt cements and asphaltic concrete are interrelated, this report concentrates on asphalt cements. The term "low temperature," as used in this report, refers to temperatures below 70°F (21°C).

A section on modified asphalt cements (e.g., high-float asphalt residue, sulfur asphalt, and asphalt rubber), which exhibit improved low-temperature behavior, is also included.

Penetration

The penetration test measures the consistency of paving asphalt, which is expressed as the distance in tenths of a millimeter that a standard needle vertically penetrates a sample of the material under known conditions of loading, time, and temperature. ASTM D5 gives the test procedure for measuring penetration at 77°F (25°C) and lower temperatures.

This test was invented by Bowen in 1889 *(1)*. The historical development and refinement of this method by Dow, New York Testing Laboratory, and the Office of Public Roads and Rural Engineering was described by Welborn and Halstead *(2)*. Some data were also given by other researchers *(3–7)*.

Although the penetration test was developed empirically and questions have been raised about the validity of the relation of penetration to viscosity, some studies have shown that the test does measure viscosity *(8)*. However, attention must be given to stress levels or shear rates, or both, to attain direct comparison of penetration and viscosity results. In general, the penetration test involves higher stresses and shorter loading time than do most viscosity tests. Because paving asphalts are usually non-Newtonian at or below ambient temperatures, the viscosity measured is much lower at the high stress level. The applicability of the penetration test at low temperatures can be achieved by varying loads, times of loading, and cup and needle sizes in place of the standard ASTM conditions.

The relationship between penetration and viscosity was studied by Saal and Labout *(9)*, Traxler and Pittman *(10)*, Rhodes and Volkmann *(11)*, Mack *(12)*, Pendleton *(13)*, Welborn et al. *(14)*, Puzinauskas *(15)*, Hoffman *(16)*, and van der Poel *(17)*.

Penetration measurements at low temperatures were used by Shoor et al. *(18)* to determine the glass transition temperature of paving asphalts. Heukelom *(19)* related asphalt stiffness to its penetration value. He also introduced the modified asphalt penetration index (PI), which is a measurement of temperature susceptibility and is used to determine asphalt stiffness at low temperatures *(20)*. The PI is determined from penetration measurements at two or three temperatures. Paving asphalt specifications that use penetration measurements at temperatures lower than 70°F (21°C) have been recommended to improve low-temperature performance *(21, 22)*.

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Ductility

The ductility of a paving asphalt is measured by the distance to which it will elongate before breaking when two ends of a briquet specimen are pulled apart at a specified speed and temperature. ASTM D113 gives the test procedure to measure ductility at 77°F and lower temperatures.

The significance of the ductility test as a means of asphalt quality control has been debated because of its empirical nature and poor reproducibility. Exactly what property is being measured is difficult to decide. It is possible that the ductility test result obtained at 39.2 or 60°F (4 or 15.6°C) indirectly reflects the relationship between viscosity and shear susceptibility at these service temperatures. According to Traxler (*1*, pp. 62–65), low ductility values are demonstrated by asphalt with a greater degree of complex flow (low values of C). Welborn et al. *(2)* and Kandhal and Wenger *(3)* reported good correlation between ductility and shear susceptibility at 45 and 60°F (7 and 15.6°C) for various asphalts, regardless of source. Barth (*4*, p. 316) stated that ductility is a valuable indicator of the physicochemical state of the asphalt colloid system. It is likely, as pointed out by Halstead *(5)*, that the ability of asphalt to undergo elongation is not the primary characteristic affecting durability, but rather that the ductility test results are an indication of an internal phase relationship of the asphaltic constituents, which in turn have an important bearing on the serviceability factors of asphalt. According to Siegmann (*6*, pp. 155–188), when ductilities of bitumens derived from different crudes are compared at equal penetrations, there are great differences, which can be correlated with the PI.

Surface condition was related to the ductility of the recovered asphalt from 47 pavements in a study undertaken by the Ohio Department of Highways and the then Public Roads Administration *(7)*. Clark *(8)* concluded that the ductility of asphalt, especially after the asphalt has been incorporated into the pavement in thin films and had its initial hardening due to volatility, is of prime importance in determining the quality of a bituminous structure. Doyle *(9)* measured ductility at 55°F (12.8°C) and observed extensive pavement cracking when the ductility dropped below 5 cm. Halstead *(5)* demonstrated that the pavements containing asphalt with penetration in the range normally considered satisfactory (30 to 50) but with low ductilities are likely to show poorer service than pavements containing asphalts of the same penetration but with high ductilities. Reporting on the Zaca-Wigmore Project, Hveem et al. *(10)* found increasing evidence that the ductility test on asphalts recovered from pavement during its service life is an important method for judging pavement performance. The ductility results for unsatisfactory asphalts on the Zaca-Wigmore Project were very low. Serafin *(11)* reviewed data on cores taken after 7 years' service on the Michigan Bituminous Experimental Road and reported that the section containing the lowest ductility had the most pitting and cracking, whereas there did not appear to be any significant differences in penetration. Abson and Burton (*12*, pp. 213–288) found that low ductility and penetration are the direct causes of cracking and raveling or disintegration. Vallerga and Halstead *(13)* studied 53 highway pavements throughout the United States and reported that severe raveling occurred in cold climates when the ductility at 60°F dropped to 3 cm or less.

Kandhal *(14)* studied 10 test pavements in Pennsylvania and concluded that aging of the pavement results in progressively lower penetration and higher viscosity values. However, the accompanying decrease in low-temperature ductility was determined to be an important factor. After the penetration of asphalt drops below 30 because of hardening, the pavements containing asphalt with low ductilities showed poorer service than pavements containing asphalts of equal penetration but with high ductilities.

A subsequent study by Kandhal and Koehler *(15)* of six experimental test sections in Pennsylvania indicated that lower ductility values were associated with higher incidence of load-associated longitudinal cracking.

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Viscosity

Because of the complex rheological behavior of asphalt cement at low temperatures, it is generally acknowledged that there is a need for an accurate and reliable measure of its consistency at temperatures below 77°F. Unlike the empirical tests of penetration and ductility, viscosity is a fundamental consistency measurement in absolute units that is generally not affected by changes in testing conditions, such as the configuration of test instruments or the geometry of the sample *(1)*. Thus, for complete definition of the low-temperature consistency properties of asphalt cements, viscosity tests are sufficiently flexible and universal to evaluate the effects of temperature, heating, and rate of deformation or loading. This is not true for other available tests of consistency *(2)*.

Viscosity is essentially, at any given temperature and shear rate, the ratio of shear stress to shear rate *(3, 4)*. At high temperatures such as 275°F (135°C), asphalt cements behave as simple Newtonian liquids when subjected to ordinary shear rates; that is, the ratio of shear stress to shear rate is constant. However, as the temperature decreases, the flow properties of asphalts become more complex and they tend to behave as viscoelastic semisolid materials *(2)*. At low temperatures, the ratio of shear stress to shear rate is not a constant and this non-Newtonian behavior can be represented by a generalized power law in which viscosity is inversely proportional to the shear rate raised to a power C, which is sometimes called the complex flow index and is a measure of shear susceptibility *(5, 6)*. Thus, over a certain range of shear rates at a given temperature, a plot of log viscosity versus log shear rate will be a straight line and the slope of that line is an indication of the asphalt's shear susceptibility. However, at relatively low shear rates, the viscosity will be essentially constant and independent of shear rate. Such limiting viscosity is often referred to as "initial viscosity." After a certain rate value has been exceeded, the viscosity values (shear thinning or thixotropic breakdown) decrease with increasing shear rate and these shear-dependent viscosities are often called "apparent viscosities" *(2)*. This behavior can be further complicated if the asphalt exhibits shear thickening, or dilatancy, in which viscosity increases with increasing shear rate at low values of shear rate *(3)*. In addition, the slope of the plot of log viscosity versus shear rate can vary so that shear susceptibility is also a function of shear rate.

Although the behavior of asphalts at low temperatures can be quite complex, viscosity measurements do have certain advantages over empirical tests such as penetration, ductility, and softening point, which have been studied from theoretical aspects and found to have serious limitations *(7)*. Because consistency measurements are influenced by factors such as sample preparation, loading mode (e.g., using a sequence of loading that results in high to low shear stress values as opposed to low to high values), and duration of load (i.e., creep behavior), viscosity measurements can be conducted over a wide range of well-defined shear stresses and shear rates in which the effects of these factors can be easily determined. Penetration and ductility tests cannot be used conveniently for such an evaluation because the shearing conditions are variable and difficult to define *(1)*. For example, it is quite difficult to determine the shear stress due to the loading of the penetration needle because the first 5.4 mm of the needle is a truncated cone, which results in a continually decreasing shear stress up to penetration values of 54 *(8)*.

Studies have also shown that there are poor correlations between initial viscosity and penetration or ductility values and that these relationships become poorer with decreasing temperature. Again, this is due to the unknown and variable shear rates associated with these empirical tests at low temperatures *(1, 2)*. The need for and importance of low-temperature viscosity measurements are also reflected in studies of temperature susceptibility and the effects of heating on asphalt-cement properties. Temperature susceptibility indexes [e.g., PI and penetration-viscosity number (PVN)] that are based on penetration measurements or on viscosity values at high temperatures such as 275°F (135°C) or 140°F (60°C) generally do not correlate well with each other and can vary as a function of the temperture range selected for the calculation of such indexes. In general, viscosity at low temperature is affected more by heating than viscosity at high temperatures. The point is that consistency measurements at high or moderate temperatures cannot be used to predict behavior of asphalt at low temperatures and that only viscosity, because of its universality and fundamental nature over a wide range of temperatures, is suitable for this purpose *(1)*. In addition, it has been pointed out that, in most cases, asphalt properties as defined by current specifications do not relate directly to performance at low temperatures. For example, two asphalts of the same grade based on viscosity at 140°F may have significantly different low-temperature consistencies. Thus the selection of an asphalt that will resist thermal cracking in the pavement should depend, if possible, on a direct measurement of low-temperature consistency and not on the use of inputs such as PI, PVN, or softening point to predict asphalt stiffness (e.g., van der Poel's nomograph) and cracking temperatures *(9, 10)*.

Numerous researchers have pointed to the need for the development of a method to measure low-temperature consistency that is complicated enough for accurate definition of the complex rheological behavior of asphalt and yet simple enough for relatively rapid and routine laboratory testing by technicians *(7, 8, 10–12)*. Obviously, no single method or device for measuring viscosity is adequate over the entire range of service temperatures for asphalt. At 140°F and higher, capillary-type viscometers are suitable because of the Newtonian behavior of asphalt in these temperature ranges. However, at low temperatures, a viscometer must be able to quantify viscosity as a function of both temperature and shear stress or rate. This complicating factor has resulted in the development of a large number of devices for measurement of shear-dependent viscosities.

In 1974, Schweyer *(13)* presented a pictorial overview of many of the experimental viscometers that have been used to study the viscoelastic behavior of asphalts. There is quite a large variety, including (a) rotational types, which utilize coaxial cylinders or a cone and plate; (b) rheometers, a specialized capillary type in which a piston is used to drive the asphalt through the capillary tube; and (c) miscellaneous types such as the sliding-plate microviscometer *(3)*. Schweyer and others have done considerable work on the use of the capillary rheometer *(5)* and the development of several generations of the constant-stress rheometer *(4, 6, 7)*, which has been used to examine both asphalt stiffness and viscosity at low temperatures. Other devices that have been investigated include the falling-plunger viscometer *(2, 11)*; the sliding-plate rheometer *(14)*, which measures asphalt stiffness but can be used for determing viscosity at low temperatures; and the forced-sphere and forced-cylinder viscometers *(15)*, which were found to give only an approximation of initial viscosity.

Of the methods that are available for determining low-temperature viscosity, the most widely used are probably the cone-plate viscometer or the sliding-plate viscometer *(16)*. These are the only two currently included as ASTM standard test methods *(17)*, and are capable of examining viscosities over a wide range of shear rates. The cone-plate (or cone-and-plate) viscometer, which was developed by Sisko at the American Oil Company *(12)*, is designed to cover the widest possible shear rate range and involves the suspending of weights from a cord attached to a cone of specified size that causes the asphalt sample to be sheared between the cone and the plate. Angular velocities of the cone, from which the viscosities are determined, can be recorded either manually or by means of a rotary variable differential transformer. The development of the sliding-plate microviscometer is generally credited to Griffin et al. *(8, 18, 19)* of the Shell Oil Company. This method involves the shearing of a thin film of asphalt between two parallel steel, aluminum, or glass plates under controlled loading or controlled rate of deformation.

Research studies have been conducted by the Bureau of Public Roads *(20)* and others *(2, 21)* using the sliding-plate microviscometer and by the Asphalt Institute using the cone-plate viscometer *(1, 2)*. The results of these and other studies have pointed out the advantages and problems associated with the measurement of low-temperature viscosity. For example, it is possible to use the viscosity data to develop family curves of viscosity versus shear rate over a range of temperatures, or to plot log log initial viscosity versus log absolute temperature ("Walther coordinates"), or even to combine the data into a so-called master curve for each asphalt. The data can also be used to analyze temperature susceptibility and age hardening as a function of both temperature and shear rate. However, it is the complexity of these data that makes it difficult to establish a standard low-temperature specification. For instance, studies have found that shear susceptibility correlates well with ductility *(10)* and correlates with tensile strength better than with ductility *(1)*. But these relationships are at specified shear rates, and other properties, such as aging indexes, can also vary with shear rate and can also vary depending on the sequence of loading and time of sample conditioning *(1, 22)*. Although suggestions have been made for specifying viscosity at a certain shear rate (e.g., 0.05 reciprocal sec) or shear stress, this may not be practical for all grades of paving asphalt. A low-temperature consistency specification is desirable, but considerably more work is needed to relate low-temperature viscosity and other properties to actual pavement performance.

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Glass Transition Temperature

Glass transition temperature (*Tg*) is usually determined by one of three methods: penetration versus temperature, volume versus temperature, or differential thermal analysis. In each case, *Tg* represents a sudden change in a physical property as the material passes through a solid state to a fluid state. Frequently the information is used as one of the parameters in the Williams-Landel-Ferry (WLF) *(1)* equation for viscoelastic materials.

Sakanoue *(2)*, Wada and Hirose *(3)*, and Hirose et al. *(4)* demonstrated the use of *Tg* in the WLF equation to calculate the superposition shift of rheological properties. Dilatometric methods were applied to determine *Tg* of asphalts by Schmidt and Barrall *(5)*, Schmidt et al. *(6)*, Schmidt and Santucci *(7)*, and Jongepier and Kuilman *(8)*. Inflection points in plots of volume versus temperature were taken as *Tg*. Schmidt et al. investigated 52 Bureau of Public Roads asphalts and found *Tg*'s in the range from 1 to –27°F (–17.2 to –32.8°C). Information was applied to predict the properties of asphalt at low temperatures by using the WLF equation. Jongepier and Kuilman *(8)*, using dilatometry, concluded that the "universal" constants in the WLF equation were seen to vary with stress frequency. They concluded that *Tg*'s do not seem to be suitable parameters for describing asphalts.

Breen and Stephens *(9)* performed gel-permeation chromatographic separation on asphalts whose *Tg* had been determined by Schmidt et al. *(7)*. In general, the correlation between the glass transition temperature and the molecular size distribution was poor.

Penetration was used to determine *Tg* by Shoor et al. *(10)*. Inflection points on curves produced by plotting penetration versus temperature between –20 and –10°F (–28.9 and –23.3°C) determined the *Tg*.

Connor and Spiro *(11)* applied differential thermal analysis to determine *Tg*. For this approach, *Tg* represents the temperature at which a phase transition occurs, that is, from solid to liquid.

The effect of pressure on *Tg* was examined by Schweyer *(12)*, who concluded that there is a linear relation between *Tg* and pressure.

The viscoelastic response to stress near *Tg* was evaluated by Majidzadeh and Schweyer *(13)*. At 16°F (–8.9°C), the four asphalts examined exhibited an instantaneous elastic deformation but at higher temperatures of 25 to 41°F (–3.9 to 5°C), no such deformation was observed.

Dobson *(14)* argues that the difficulties in applying the WLF equation to asphalt are due to the small values of *Tg*.

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Tensile Strength of Thin Films

Mack *(1)* was one of the early pioneers in the investigation of physical properties of thin films. He observed that in strength, thin films between 0.02 and 0.07 cm behaved like solids. Strengths were measured with a Baldwin testing machine. Photographic plates showed unusual flow patterns of thin films under pressure. Marek and Herrin *(2)* observed that the variabilities in tensile strength include film thickness, rate of deformation, temperature, consistency, and source of bitumen. A linear variable differential transformer was used to measure strengths, which varied between 500 and 600 psi (3447 to 4137 kPa) for film thicknesses between 10 and 100 μ, respectively.

The tensile strength of thin films of 12 asphalts was studied by Sisko *(3)* in three different conditions: unaged, aged in the thin-film oven (TFO) test, and aged in roads up to 11 years. Strength of films increased as much as 140 percent as the temperature decreased from 80 to 0°F (26.7 to –17.8°C). At low temperatures, TFO aging produced no significant differences in the tensile strength of the asphalts, but road aging did. All tests were performed on 0.0005-in. (0.0127-mm) asphalt films in an Instron tester.

Haas and Anderson *(4)* demonstrated that asphalt source or type can have a major influence on low-temperature cracking of a bituminous layer, and they suggested that thin-film tension testing of asphalts shows some promise as a simple analytical tool. Stiffness modulus was measured with Instron stress-strain equipment. Temperatures as low as –60°F (–51°C) were investigated.

On the basis of the data collected, Marek *(5)* developed a technique to enable estimation of the relationship between tensile strength and log film thickness for asphalt cements of the normally used consistency, for normal temperatures above the glass transition temperature, and for rates of deformation ranging from 0.005 to 0.1 in./min. (0.127 to 2.54 mm/min). Average tensile strengths ranged from 1,000 psi (6895 kPa) at 10-μ film thickness to 200 psi (1379 kPa) at 100-μ film thickness.

Using centrifugal force, Averbakh et al. *(6)* measured the force at which a certain fraction of glass marble beads with a diameter of 180 μ separates from a 5- to 10-μ thick bitumen planar layer in a centrifuge at 500 to 7,750 rpm. Separation strength of the beads from the thin bitumen layer was 2.0×10^6 dynes/cm^2 for a typical sample.

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Absolute-Rate Theory and Asphalt Fracture

The applicability of the absolute-rate theory in explaining the flow behavior of paving asphalts was investigated by Herrin and Jones *(1)* in 1963. This theory can be applied to any process in which a movement or flow of particles takes place. It is based on the assumption that the mechanics of flow consists of the movement of atoms, molecules, or groups of molecules (flow units) into vacancies in a material or by the displacement of the vacancies themselves and that for this movement to take place, a strain-energy barrier must be overcome.

Subsequently, Herrin et al. *(2)* enlarged on the scope of this study and tested six paving asphalts for flow behavior over the temperature range of 32 to 122°F (0 to 50°C). They concluded that the absolute-rate theory seemed to be applicable to paving asphalts:

1. As the temperature decreases, the flow unit will be composed of more molecules of asphalt and will be larger in size.
2. The energy needed to produce a movement of the flow units varies directly with the size of the flow unit and thus greater energies must be available to produce movement of the flow units at lower temperatures.

Moavenzadeh *(3)* tested three asphalts down to –7°F (22°C) to evaluate whether the Griffith theory of brittle fracture can be used in determining fracture susceptibility of the asphalts at low temperatures. He concluded that at sufficiently low temperatures, the asphalts behave as brittle, amorphous materials and that this theory can be applied. The value of the critical strain-energy release rate is influenced by the rate of loading, the test temperature, and the type of asphalt.

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Viscoelastic Properties

Viscoelastic properties of paving asphalts and asphalt concrete have been studied by various researchers using different testing equipment and conditions.

Pagen *(1, 2)* conducted dynamic and creep tests over a range of temperatures from 0 to 125°F (–18 to 52°C). His experimental data indicated that both the time-temperature superposition principle and linear viscoelastic theory are applicable to the asphalt concrete tested at a satisfactory level of approximation.

Majidzadeh and Schweyer *(3)* studied the viscoelastic response of four asphalts in the temperature range of 16 to 41°F (–9 to 5°C) using cylindrical specimens. At extremely low temperatures such as 16°F (–9°C), the asphalts exhibited some instantaneous elastic deformation, which is represented by the spring in the Maxwell element. Subsequently, they reported *(4)* on the viscoelastic response of aged asphalt cements.

Sisko and Brunstrum *(5, 6)* used the Weissenberg rheogoniometer to determine the viscoelastic properties of unaged and aged asphalts over a range from 0 to 80°F (18 to 27°C). They reported that at traffic stress frequencies, both unaged and aged asphalts have essentially the same mechanical properties at low temperatures (0 to 20°F).

Schweyer and Busot *(7)* used an Instron rheometer to study asphalt cements at 32°F (0°C) and attempted to separate the elastic from the flow effects.

Jongepier and Kuilman *(8)* determined the linear viscoelastic properties of a series of bitumens (ranging from extreme sol to extreme gel type) at various frequencies and over a temperature range from –4 to 320°F (–20 to 160°C) using a Weissenberg-Sangamo rheogoniometer. The dilatometric glass transition temperatures were also determined.

Dobson *(9)* investigated the viscoelastic behavior of asphalts down to 13°F (11°C) by a dynamic method in which a sinusoidal stress is applied to the asphalt to determine the storage and loss moduli.

Pink et al. *(10)* used a Rheometrics mechanical spectrometer (RMS) to make accurate low-temperature dynamic viscoelastic measurements on asphalts down to –137°F (–94°C). Dynamic master curves were developed to separate the effects of time and temperature. Button et al. *(11)* also used the RMS to measure viscosity of asphalts from 32 to –50°F (0 to –46°C).

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Asphalt Stiffness

In asphalt rheology, the use of stiffness or resistance to stress as a function of time dates from Nijboer and van der Poel's 1953 description *(1)*, although Nutting *(2–4)* proposed a generalized stress-strain-time concept in 1921.

The subject of stiffness was updated in 1958 by Saal and Labout *(5)*, whose rather interesting results gave stiffness values for several types of asphalt bitumens and mixes over a range of temperatures from –50 to 50°C (–58 to 122°F) and at different stress levels.

In 1969, Reiner *(6)* treated rheology theoretically and discussed asphalt deformations. Stiffness was defined as the stress divided by the cumulative strain after a specified time and is therefore a function of temperature, time, and loading procedure.

Schweyer et al. *(7)* proposed a model that accommodates the shear susceptibility by a feedback mechanism. This model is essentially a modified version of the Burgers model for asphalt mentioned previously by Saal and Labout *(5)*.

Later, Schweyer and Burns *(8)* attempted to clarify the meaning and significance of stiffness, to suggest a format for data reporting, to demonstrate by experiment a proposed model and the relations between stiffness and other rheological properties, and to illustrate the variations in stiffness for certain selected asphalt bitumens at temperatures below 25°C (77°F) by using a constant stress shear mode apparatus.

Schweyer and Burns concluded that a stiffness evaluation model at small values of time requires directly observed data; otherwise, it is vitiated by the initial transient viscoelastic effects. However, long-term values of stiffness may be estimated from viscosity measurements, which also can be used with the elastic modulus to estimate short-term stiffness.

In order to accommodate the large amount of data for shear stress and temperature effects, various authors have proposed master curves and temperature-susceptibility correlations. The van der Poel empirical nomograph *(9)* is one approach.

One of the early dynamic studies on asphalt bitumen using a piezoelectric device was reported in 1960 by Wada and Hirose *(10, 11)* for complex compliance.

Jongepier and Kuilman *(12)* proposed a shift factor for the separate curves at different temperatures to produce a master curve with parameters based on the temperature at which different asphalts have the same viscosity (equiviscosity temperatures). Also discussed was the "spectrum" of relaxation times expected for the various components in the materials under study at different test conditions. They suggested a "glass modulus" of 10^8 Pa and also discussed certain free volume and glass transition phenomena related to the relaxation spectrum width. They concluded that the glass transition temperature was not an equiviscosity temperature.

Dobson *(13–15)* in 1969 suggested the use of a reduced-state master curve for data on bitumen obtained in an oscillating-cone device.

Dickinson and Witt *(16)* proposed a hyperbolic equation for the empirical correlations of dynamic data based on a concentric device vibrated in the axial direction at temperatures above 35.6°F (2°C).

Heukelom and Klomp *(17)* and Heukelom *(18)* presented a variety of interesting analyses of data based on the stiffness or the ratio of stress to total strain at any time. Heukelom also combined creep-test and tensile-strength test data for correlations with stiffness and indicated that tensile strength maximizes at about 55 kg/cm^2 at a stiffness range of 4×10^6 to 5×10^6 Pa for a generalized curve. He further commented on the general relationship of stiffness to penetration and suggested that time and temperature effects disappear only at high values of these parameters. Heukelom related stiffness to penetration and reported maximum stiffness values of 2.7×10^9 Pa (about 4×10^5 psi).

Majidzadeh and Schweyer *(19)*, using creep data, evaluated the complex modulus and its components for four different asphalts and studied aging of the asphalt. Similarly, work was reported by Verga et al. *(20)* for asphalt-elastomer mixtures using creep compliance procedures with static creep. Haas and Anderson *(21)*, using a thin-film tensile stress-strain technique over temperature ranges from –60 to 60°F (–50 to 15°C) studied stiffness modulus at 10 to 800 sec and related this to mixes at the same magnitudes.

Asphalt cracking over a range of cooling rates has been related to stiffness and loading time *(22–25)*. Schmidt *(22, 23)* suggested a limiting temperature as a criterion for cracking when the stiffness at 10,000 sec reaches 20,000 psi (1.4×10^8 Pa) at a cooling rate of 5°C/hr.

The Shell sliding-plate rheometer *(26)* conveniently measures low-temperature asphalt stiffness and has been used *(27)* to determine the accuracy of different asphalt stiffness prediction techniques *(28)*. Recently the sliding-plate rheometer was modified to extend its upper stiffness limit from 1×10^8 to 1.5×10^9 N/m^2 *(29)*.

In North America, the most commonly used asphalt stiffness prediction procedures *(29)* are based on van der Poel's nomograph *(9)*, one using PI *(30)* based on Pfeiffer and van Doormaal's original index *(31)* and another a modified procedure introduced by McLeod *(32)* using PVN. Researchers *(28, 33–37)* have shown good agreement between measured bitumen stiffness and observed field cracking when low-temperature penetrations were used with Heukelom's *(30)* improved bitumen test data chart. Although Heukelom showed that his chart could be used with a wide variety of asphalts, its use to predict thermally induced cracking or to estimate the low-temperature stiffness of mixes was shown by others to be limited to a small variety of asphalts *(22, 23)*.

Values referred to as "limiting stiffness" and defined as the asphalt stiffness modulus above which pavement cracking is imminent have been reported by several researchers *(9, 28, 35, 38)*. They range in value from 20,000 to 70,000 psi (1.38×10^8 to 5×10^8 N/m^2) at a loading time of 10,000 sec. Other researchers have established methods to predict pavement cracking based on "fracture temperature" *(25, 33, 36)*, that is, the temperature at which an infinite beam of asphalt cement restrained on each end will crack when cooled at a certain rate.

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Modified Asphalts

A number of additives and extenders for asphalt cement are being used currently. However, not all additives and extenders modify the low-temperature properties of an asphalt cement. In the following discussion, therefore, only those modifiers that have any potential benefit to the low-temperature characteristics of asphalt cements or mixtures, or both, are identified. These include sulfur, fibers, carbon black, high-float emulsions, rubber, and polymers.

Sulfur-asphalt mixtures have received considerable attention during the past 10 years. Two basic approaches have been used to incorporate sulfur into asphalt mixtures: either it can be added directly to the mixture or it can replace a portion of the asphalt [sulfur-extended asphalt (SEA)]. SEA has emerged as a potential binder in which sulfur replaces up to one-half the weight of asphalt normally used. The role of sulfur in SEA mixtures has been described as an asphalt extender, a binder, a structuring material, a filler material, or a combination of all four *(1)*. There is no documentation of any test results at low temperature on sulfur-asphalt binders. However, the results of an extensive amount of testing of mixtures over a range of temperatures indicated that the stiffness modulus of sulfur-asphalt mixtures at low temperatures is primarily affected by the consistency of the asphalt used in the binder and by the temperature *(2, 3)*. The addition of sulfur does not have any significant effect on the low-temperature stiffness of SEA mixtures, which has been verified by field experiments *(4)*. At high temperature, the addition of sulfur to a soft asphalt (e.g., 300 to 400 penetration) can increase its stiffness in terms of resilient modulus to that of the mixture made with 40 to 50 penetration alone *(2)*. However, at low temperature, the addition of sulfur has no effect on stiffness. Thus the addition of sulfur is quite significant at high temperatures and insignificant at low temperatures. This provides increased flexibility because asphalt grade and sulfur-asphalt ratio can be varied to satisfy the requirements of both low and high service temperatures.

The use of special composite materials (fibers) to increase resistance to fatigue and thermal cracking has been of interest to asphalt paving technologists. Processes for producing high-tenacity fibers with small diameters have been developed. Two commonly used fibers in asphalt mixtures are asbestos and polypropylene. Haas *(5)* conducted a study on the modification of temperature susceptibilities of asphalt-asbestos paving mixtures. The experimental results were obtained in terms of stiffness, failure strain, and strength over the service temperature range from –20 to 140°F (–29 to 60°C). A major finding was that at low temperatures, the properties of all types of mixtures were primarily a function of the asphalt type used, but at medium to high service temperatures, asbestos fiber modification could significantly improve mix properties in comparison with standard conditions. A major implication of this finding was that where a softer asphalt was to be used for low-temperature cracking considerations, asbestos fiber modifications may be used. Such modification will not appreciably alter low-temperature stiffness but will increase medium to high service temperature stiffness and thereby possibly avoid violations of fatigue and permanent deformation limits. Polypropylene fibers *(6)* have also been used as an asphalt reinforcement. They disperse readily in either hot- or cold-mix systems and can be processed and applied with conventional equipment. Polypropylene fibers are water insoluble and retain dimensional stability with changes in humidity. They are also highly durable and offer excellent chemical and abrasion resistance. However, temperatures above 290°F (143°C) will damage polypropylene *(7, 8)*. Because both polypropylene fiber and asphalt are derived from a petroleum base, compatibility and bonding of fiber to asphalt are excellent. The reinforcement of asphalt with polypropylene fibers *(9)* results in mixtures with improved elongation capability, better resistance to freezing and thawing, and a more durable and longer-lasting service life, particularly in cold climate regions.

Another commonly used additive is carbon black. The carbon black pellets are specifically designed for incorporation into asphalt as a reinforcing agent consisting of a high-structure type carbon fluxed with a high-durability maltene oil. The carbon black is of submicrometer size and therefore fine enough to become an integral part of asphalt and serve as a reinforcing agent *(10)*. The effects of the carbon black filler on the properties of a given asphalt will vary somewhat depending on the characteristics of the asphalt and the grade and dosage of the filler pellets. In general, on the basis of all laboratory data reported *(11, 12)* on a variety of asphalts, the filler has been found to increase asphalt durability and decrease temperature and viscosity susceptibility. A series of tests have been performed using 21.2 parts per 100 of MICROFIL 8 and MICROFIL 25 and three Canadian asphalts having penetration values of 85 to 100, 150 to 200, and 300 to 400. The viscosities of their MICROFIL dispersions were measured over a range of temperatures from 0 to 140°F. The results indicated *(13)* that additions of either MICROFIL 8 or MICROFIL 25 increased viscosity at 140°F (60°C). MICROFIL 25 addition gave both increased viscosity at 140°F and increased penetration at 39.2°F (4°C), a demonstration of improved temperature susceptibility. All three asphalts with MICROFIL 25 had slightly lower viscosities than the base asphalt at temperatures of 39.2°F or lower, and higher viscosities above 77°F (25°C). It should be pointed out that the carbon black pellets are bound together with a material similar to motor oil *(14)*.

Emulsification of a base asphalt containing some additives derived from tall oil is considered to be a workable method of providing a residue with improved temperature-susceptibility characteristics. In this regard, the use of high-float emulsions has been common. The major emphasis of improved temperature-susceptibility characteristics of the high-float emulsions, however, has been on the dividends associated with stability at high ambient temperatures *(15)*. The recognition of the impact of their improved temperature susceptibility on low-temperature characteristics of bituminous mixtures has remained relatively dormant. The dividends associated with high-float emulsification in terms of improved low-temperature properties were illustrated in some recent studies *(15–17)*. A highly temperature-susceptible asphalt cement with a PVN of –1.7 was used to make a high-float emulsion. The PVN of this emulsion residue was determined to be –0.14, a marked improvement in temperature susceptibility.

Furthermore, absolute viscosity values for the above-mentioned binders were determined at −10, 0, and 39.2°F (−23.3, −17.8, and 4°C) using the ball penetrometer and the standard penetrometer *(18)*; these values were reported to be 8.67×10^{10}, 1.58×10^{10}, and 9.8×10^{7} poises, respectively, while those for the high-float emulsion residue were determined to be 2.69×10^{10}, 8.41×10^{9}, and 4.5×10^{7} poises, respectively. The effect of improved low-temperature viscosities and temperature susceptibility has also been demonstrated on the characteristics of the mixtures. A comparison of limiting stiffness at −10°F of the mixtures made with the above-mentioned asphalt cement and high-float emulsion rendered the values of 1.15×10^{6} and 0.7×10^{6} psi, respectively.

Rubber has also been used with asphaltic materials to improve their properties as cementing materials for aggregate. It has been demonstrated by many studies that addition of these materials in amounts as little as 0.1 percent, but more normally in amounts of the order of 1 to 5 percent, materially affects the properties of asphalt *(19, 20)*. It has been found that the characteristics of rubber-asphalt blends are affected by the type of rubber used (natural, synthetic, or reclaimed), the nature and source of asphalt, temperature and time of heating, and the amount of stirring in the preparation of the blends. In general, addition of rubber increased the viscosity and softening point of asphalts and decreased their penetration, flow, and temperature susceptibility. Rubber greatly increased the toughness and tenacity of asphalt and introduced a high degree of elastic recovery into the binder *(21–23)*. The flexure tests *(24)* showed that the rubber-asphalt concrete is about 30 percent more ductile than normal asphalt concrete. This increase in ductility was most marked at higher temperatures. However, even at −4°F (−20°C) a 10 percent increase in ductility is obtained, suggesting that the material will perform noticeably better at low temperatures than standard asphalt concrete. Kennedy *(25)* conducted a study on low-temperature dynamic properties of asphalt-rubber mixtures using a Rheovibron viscoelastometer over a range of temperatures from −31 to 59°F (−35 to 15°C). The results of this analysis showed that the brittle temperature of reclaimed rubber-asphalt mixtures was reduced by approximately 1°C for each part of added rubber and that the addition of rubber also reduced the temperature susceptibility so that the rubber-asphalt mixture was more viscous at elevated temperatures than the asphalt without rubber.

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