Rheology Based Specifications for Asphalt Binders used in Roofing

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Introduction

Specification requirements for roofing and industrial asphalts serve several purposes. Foremost, they assure uniformity of the asphalt before application or use, thus assuring the purchaser that the asphalt performance will not vary substantially and the asphalt utilized will not require departure from standard application and use practices. Specification requirements also assure the user that, with proper application or use, the asphalt will perform its design function throughout anticipated service life.

Standard specifications for roofing and industrial asphalt in the US currently include ASTM D 312 for asphalt used in roofing as inter-ply adhesives or flood coats for built-up roof (BUR) membranes; ASTM D 449 "Standard Specification for Asphalt Used in Damp-proofing and Waterproofing," for asphalt used as mopping coats for damp-proofing and plying or mopping cement in construction of waterproofing membranes; and ASTM D 2521 "Standard Specification for Asphalt Used in Canal, Ditch, and Pond Lining," for oxidized petroleum asphalt used as a canal, ditch, and pond lining.

Common empirical properties used in standardization and specification of these asphalts are softening point and penetration, with softening point being the primary empirical test used as a grading criterion. Ranges of softening point temperature are used to differentiate between different types and grades of asphalt generally focusing on the upper temperature limit of asphalt performance. Softening point temperature is generally measured in accordance with either ASTM D 36 "Standard Test Method for Softening Point of Bitumen (Ring-and-Ball Apparatus)" or ASTM D 3461 "Standard Test Method for Softening Point of Asphalt and Pitch (Mettler Cup-and-Ball Method)," penetration measurements are made in accordance with ASTM D 5 "Test Method for Penetration of Bituminous Materials."

Paving asphalt specifications in the United States, Europe, and elsewhere in the world are increasingly dependent upon basic principles of rheology, in particular dynamic techniques such as dynamic shear rheology.

Rheological performance is the critical determination of satisfactory asphalt quality in that asphalt must possess sufficient stiffness at higher temperatures to resist flow and mechanical damage, and also be flexible enough at lower temperatures to resist cracking and de-bonding. Fundamental rheological testing can also be used to define accurately the rheological response of roofing asphalts over a wide range of temperatures and loading times. Baumgardner and Rowe [1] have demonstrated the usefulness of rheological parameters to replace the current empirical parameters, as well as introduce high and low temperature rheological parameters for climatic consideration.

Dynamic Shear Rheology

Asphalt binder testing using a dynamic shear rheometer (DSR) is becoming the primary mode to evaluate asphalt binders worldwide; an understanding of the rheological performance of asphalt binders has become increasingly important. Such understanding of the basic principles of rheology is essential for those developing and applying specifications, as well as developers and producers of new and enhanced roofing binders. Fundamental rheometry testing can be used to accurately define the rheological response of asphalt over a wide range of temperatures and loading times. The focus of this white paper is on dynamic shear rheology, in particular parallel plate measurements.

In parallel plate dynamic shear rheology the asphalt specimen is sheared between two parallel plates. The measurement principles of parallel plate dynamic shear rheology consists of applying an oscillatory load to a thin "hockey puck" shaped asphalt specimen squeezed between the two parallel metal plates. One plate is fixed while the other is caused to oscillate sinusoidally.

Dynamic shear rheology equipment used to test asphalt can typically be divided into two categories, controlled strain, where the rheometer applies a strain to the specimen and measures the resulting stress, and controlled stress, where the rheometer applies a stress to the specimen and measures the resulting strain. For the purposes of this paper, these differences are not considered to be important to the user.

The DSR is used to measure the modulus and phase angle of asphalt binders at varying temperatures. The complex modulus (G*) is a measure of the overall stiffness of the binder under dynamic shear loading while the phase angle (δ) is a measure of the relative proportion of viscous and elastic behavior of the asphalt specimen. These measurements are considered to be dynamic since the stresses or strains applied to the asphalt sample vary with time in a sinusoidal fashion, in other words they are not static, thus “dynamic.”

Rowe and Baumgardner, [2] describe a test method for the determination of the complex shear (stiffness) modulus (G*) and phase angle (δ) of bituminous binders over a range of temperatures and frequencies when tested in harmonic sinusoidal oscillatory shear mode using a DSR with parallel plate test geometry where both plates are controlled at the same temperature. In addition, the data are extended by testing with the bending beam rheometer (BBR), torsion bar (TB) specimens made of only binder, and the direct tension test (DTT) to provide further information on the cold temperature behavior. Data collected were employed in construction of master curves defining overall performance of asphalt roofing binders.

Material Analysis and Master Curve Development

The primary purpose for the construction of a master curve is to enable the rheological changes in the binder to be observed over time. In addition, the master curve allows the combination of datasets from additional testing methods such as BBR, TB and DTT with DSR data in a manner enabling material properties to be estimated at conditions beyond the measuring capabilities of any single piece of equipment.

In the work of [2] rheological properties of six roofing asphalt binders were obtained from the four different tests. Frequency sweep tests, ranging from 0.1 to 100 Hz (0.6284 to 62.84 rad/s), were conducted on the DSR at temperatures ranging from 0 to 105°C in 15° steps. Figure 1 displays storage (G′) and loss (G″) moduli data from five frequency sweep isotherms of one of the six roofing asphalt binders. G* was determined directly from frequency sweep moduli data from each isotherm.

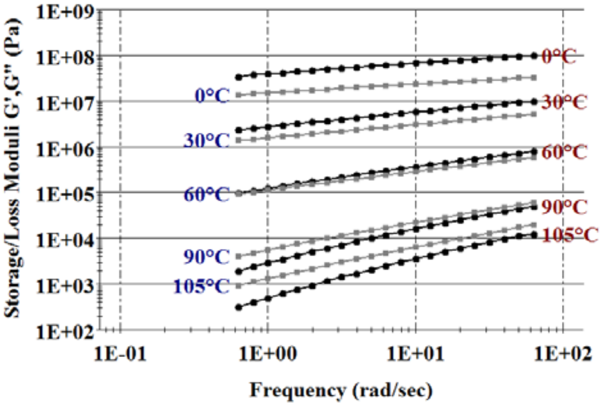


FIG. 1, Frequency Sweep Isotherms from 0°C to 105°C.

Additional testing was performed using the BBR, and the DTT. The data from the BBR and DTT measure values of S(t) or E(t). These data were converted to G* and δ and then combined with the data from the DSR to produce master curves of rheological properties.

Rheological data analysis was performed using RHEA™ software [3]. The method adopted followed the traditional approach of shifting the modulus values (either G(t) or G′ and G″) along the horizontal axis to form smooth modulus curves. The shift procedures for producing a master curve have been developed by various researchers as identified by [2]. A typical resulting master curve (of G′ and G″), from [2], showing data from all four test types is illustrated in Fig. 2, whereas the same data in the form of G* and δ are presented in Fig. 3. The line fits on both these figures are those produced from a discrete spectra analysis assuming linear viscoelastic behavior.

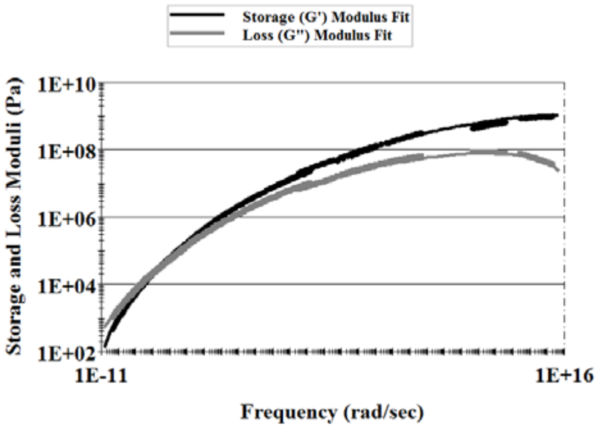


FIG. 2, Master Curve, G′ and G″, Resulting from Combination of DSR, BBR, TB, and DTT Data, T_{ref} =25°C.

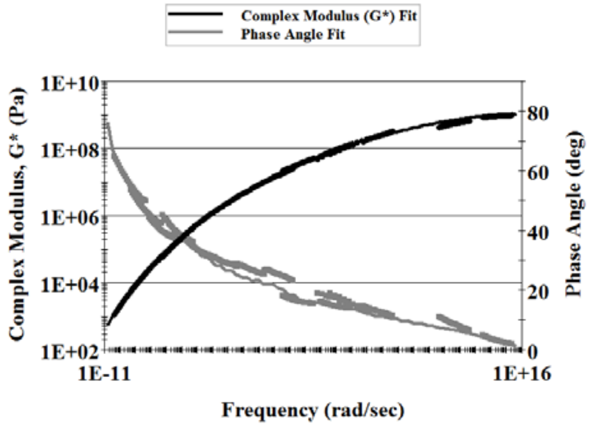


FIG. 3, Master Curve, G* and δ, Resulting from Combination of DSR, TB, and DTT Data, T_{ref}=25°C.

While producing valuable material information, development of master curves as described can be very time consuming and may not be practical for specification purposes. Most often for specification purposes the process is abbreviated by performing tests at specific frequencies and temperatures or by testing at fixed temperatures over a range of frequencies (frequency sweep) or at a fixed frequency over a range of temperatures (temperature sweep). Data obtained from the abbreviated method can then be compared to historical data or data from full material master curves. One such method as used by the British Institute of Petroleum (IP) is described in Method IP PM CM/02, “Determination of the Complex Shear Modulus and Phase Angle of Bituminous Binders—Dynamic Shear Rheometer (DSR) Method.” It describes a method for full master curve development similar to the full frequency/temperature sweep method described above as well as a more rapid “abbreviated” method which consists of testing at 0.4 Hz (2.5 rad/s) over a range of temperatures. Figures 4 and 5, from [1], are examples of data from the abbreviated IP method with the exception that temperature was allowed to equilibrate at each test temperature.

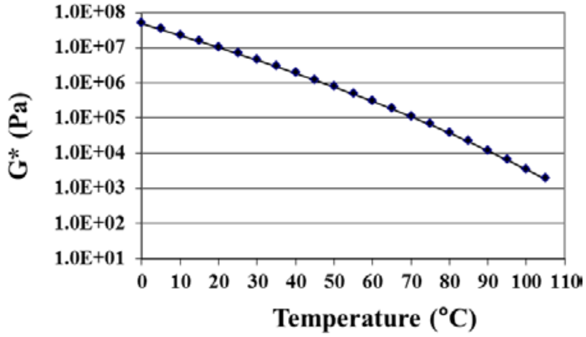


FIG. 4, Complex Modulus (G*) Master Curve from Temperature Sweep at 0.4 Hz (2.5 rad/ s).

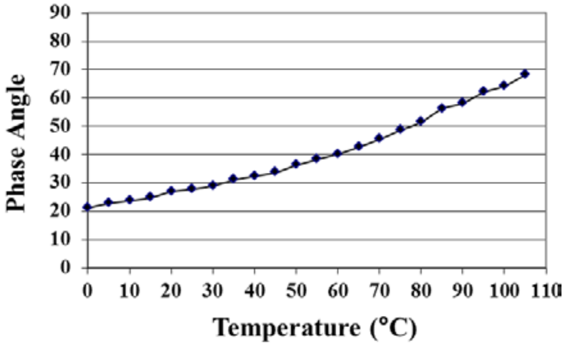


FIG. 5, Phase Angle (δ) Master Curve from Temperature Sweep at 0.4 Hz (2.5 rad/s).

Empirical Property Comparison

Performance of roofing materials has traditionally been assessed using empirical properties, discussed previously, penetration and softening point in particular. These empirical properties have been related to performance via long term historical observations. While these tests have been useful in development of specifications and standards for materials, they lack scientific rigor in their development.

Attempts have been made in recent years to relate the measurements from rheological tests to empirical values. Penetration can be related to rheology with reasonable accuracy; however, similar correlations, while not impossible, are more difficult to establish with softening point. This was noted by Gershkoff [4] and is consistent with the data analyzed in the work of [1]. Earlier research of Van der Poel [5] has shown that the penetration test relates well to the stiffness or modulus of asphalt at a loading time of 0.4 s. Through linear regression analysis, Gershkoff [4] proposed the following relationship between complex modulus (G*) at 0.4 Hz and penetration:

$$\mathrm{Log}(G^*_{(0.4\ \mathrm{Hz})}) = 8.80 - 1.95(\log(\mathrm{Pen})) \qquad (1)$$

where:

$G^*_{(0.4\ \mathrm{Hz})}$ = G*(Pa) at a loading time of 0.4 Hz, and
Pen = Penetration (mm/10 or dmm), 100 g, 5 s.

Figure 6 shows the relationship of $G^*_{(0.4\mathrm{Hz})}$ versus log (penetration) as presented by Gershkoff [4] for a selection of asphalt binders. Molenaar et al., [6], as well as Rowe and Baumgardner [2], while supporting the findings of Gershkoff, also propose alternative relationships.

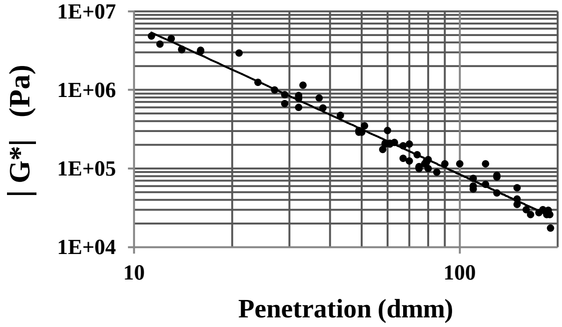


FIG. 6, Complex Modulus (G*) at 0.4 s versus measured penetration values (R^2 = 0.98).

Gershkoff also cites work of Pfieffer and Van Doormaal [7] which indicates that penetration at the softening point was approximately 800 dmm for many but not all asphalts. The exact values vary with penetration index (PI) and wax content. Solving Eq. 1 for a penetration value of 800 dmm yields a complex modulus (G*) value 1.3804×10^3 Pa or 1.38 kPa for most asphalts. This value is considered to be a reasonable estimate of softening point or "high equi-stiffness temperature." This is also in general agreement with the widely held consensus that the softening point represents, to a first approximation, an equiviscous temperature with a viscosity of approximately 1200 Pa·s.

Rheological Specification Elements

ASTM D 312 specifies four types of asphalt designated as Type I, Type II, Type III, and Type IV (D 312 Table 1). These asphalt grades are also commonly known as "dead level," "level," "steep" and "extra-steep" roofing asphalt, respectively. Testing required by this specification, which is considerably time consuming, could take as long as 3 to 5 hours to complete.

Baumgardner and Rowe [1] proposed a performance grade specification for roofing asphalts comprised of four similar types or performance grades developed by using a temperature sweep test in which the temperature is continuously increased at a rate of 2.5°C while measuring the complex modulus at a frequency of 0.4 Hz. This frequency was suggested as it is consistent with that of the penetration test discussed earlier. Testing time required to complete grading in accordance with this proposed specification could be as little as 1 to 2 hours.

The following elements were suggested for development of a DSR performance-based specification for roofing and industrial asphalts to replace current specifications of ASTM D 312:

Flash Point — Minimum, required to maintain safety during application of the material.

High Equi-stiffness Temperature ($T_{(2kPa)}$) — The temperature at which G* is equal to 2.0 kPa at 0.4 Hz (2.5 rad/ s) determined from the plot of G* versus temperature. This will be considered the rheological equivalent of ring-and-ball softening point. This temperature may by higher or lower than the actual measured softening point via the ring-and-ball method. High equi-stiffness temperature should be measured before and after heat exposure (roofing kettle simulation) to ensure retention of properties.

Complex Viscosity (η*) at 71.1°C — *S*teady state viscosity represented by G* of the asphalt measured at 71.1°C with a loading rate of 0.159 Hz (1 rad/s). Complex viscosity should be measured before and after heat exposure (roofing kettle simulation) to ensure retention of properties.

$G^*_{(5)}$ — The complex stiffness modulus at 5°C and frequency 0.4 Hz. (2.5 rad/s) considered to be an estimated equivalent to the penetration of a 100 g loading for 5 seconds measured at 5°C.

$\Delta_{(low)}$ — The phase angle from the temperature equilibrated data at 5°C and at a frequency of 0.4 Hz. (2.5 rad/s).

$G^*_{(25)}$ — The complex stiffness modulus at 25°C and frequency 0.4 Hz. (2.5 rad/s) considered to be an estimated equivalent to the penetration of a 100 g loading for 5 seconds measured at 25°C.

$G^*_{(50)}$ — The complex stiffness modulus at 50°C and frequency 0.4 Hz. (2.5 rad/s) considered to be an estimated equivalent to the penetration of a 100 g loading for 5 seconds measured at 50°C.

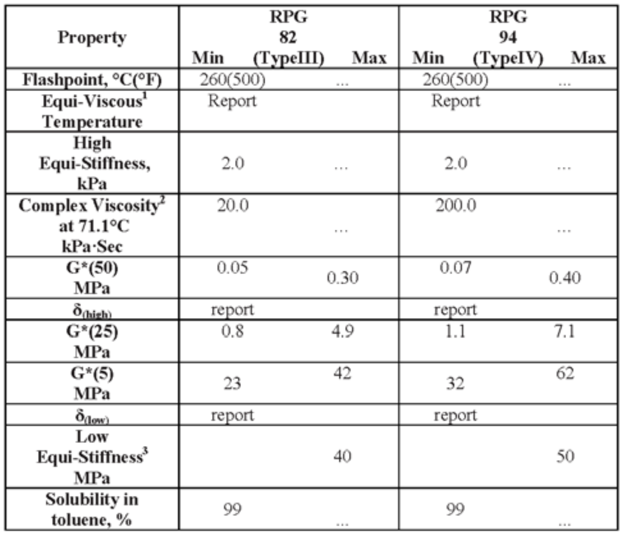
$\delta_{(high)}$ — The phase angle from the temperature equilibrated data at 50°C and at a frequency of 0.4 Hz. (2.5 rad/s).

Low Equi-stiffness Temperature ($T_{(2MPa)}$) — *R*epresented by G* of the asphalt measured at 25°C and a loading time of 15.9 Hz (100 rad/s).

Solubility in Toluene — Minimum, required to ensure purity of the material. Toluene is selected in lieu of the former trichloroethylene (TCE) due to environmental concerns.

A proposed performance grade specification incorporating these suggested specification elements, from [1], is presented in Table 1 for the two most common ASTM D312 grades, Type III and Type IV. This proposed specification and its elements are intended for discussion purposes only. Considerable testing would be required to establish final specification requirements.

Table 1, Proposed Performance Grade Specification for Roofing Asphalts (For Discussion Purposes Only)



Property	RPG 82 Min	(TypeIII)	Max	RPG 94 Min	(TypeIV)	Max
Flashpoint, °C(°F)	260(500)		...	260(500)		...
Equi-Viscous[1] Temperature	Report			Report		
High Equi-Stiffness, kPa	2.0		...	2.0		...
Complex Viscosity[2] at 71.1°C kPa·Sec	20.0		...	200.0		...
G*(50) MPa	0.05		0.30	0.07		0.40
$\delta_{(high)}$	report			report		
G*(25) MPa	0.8		4.9	1.1		7.1
G*(5) MPa	23		42	32		62
$\delta_{(low)}$	report			report		
Low Equi-Stiffness[3] MPa			40			50
Solubility in toluene, %	99		...	99		...

[1] Equi-viscous Temperature (EVT) = temperature (°C) where viscosity, measured by rotational viscometer, equals 125 X 10^{-3} Pa·Sec

[2] Complex Viscosity at 71.1°C = G* at 71.1°C with a loading time of 6.25 Hz (1 radian/second)

[3] Low Equi-stiffness = G* (MPa) measured at 25°C with a loading time of 625 Hz (100 radians/second)

The roofing industry would be substantially benefited by revised ASTM specifications featuring DSR measurements. The focus here has been primarily on rheological parameters to facilitate a replacement specification for ASTM D 312, however, the concept can be extended to include other specifications as well. As an example, additional specifications can be developed for asphalt materials falling under the category of roofing and industrial asphalts such as saturants and coatings. Saturants are used to impregnate organic or fiberglass felts used in BUR roofing felts, roofing shingles, and rolled roofing products, while coating grade asphalt is used to coat impregnated organic felts of glass-fiber felts. Comparable ASTM standards specifications do not exist for saturants and coating asphalt. These asphalts are generally produced to parameters desirable to provide the necessary impregnating and coating characteristics for specific finished products. Industry practice is to use empirical properties similar to those in ASTM D 312, in particular softening point to specify and evaluate asphalt used as saturants and coating grade asphalt.

Rowe and Baumgardner [8] have recently presented results of work employing rheological models in evaluation of modified roofing asphalt systems used in production of modified bitumen roofing membrane systems. This work exhibits the utility of rheology in evaluation of roofing binder systems used in membrane manufacture and construction. Similar work has been reported by Brown and Sparks [9] on unmodified roofing binders studying the effects of asphalt chemistry on rheological performance.

Conclusion

Current specifications are generally purely empirical in nature, relying on properties such as softening point expressed in units of temperature (°C or °F), penetration expressed in tenths of millimeters (dmm), and ductility expressed in centimeters (cm) to indicate consistency. Unfortunately, empirical properties, expressed in units of temperature and length, do not access the fundamental engineering properties of roofing and industrial asphalts. Granted, efforts have been made to adopt viscosity-based specifications, [10], [11], [12], [13]; however, viscosity-based specifications are also empirical in nature. Empirical tests used in ASTM specifications for roofing, though useful for purchasing and application, do not provide the fundamental engineering properties necessary to accurately predict performance of roofing and industrial asphalts. While these tests have been useful in the development of specifications and standards for materials, they lack scientific rigor in their development.

Fundamental rheometry testing can be used to accurately define the rheological response of asphalt over a wide range of temperatures and loading times. In particular, dynamic testing techniques such as DSR, which have become increasingly popular in asphalt specifications, can provide rapid reproducible information based on fundamental engineering properties. Establishing relationships between asphalt rheological properties to asphalt empirical properties can provide a smooth transition from purely empirical specifications to specifications based on fundamental engineering properties in which users can have a reliable comparison to historical performance. Such relationships have been established relating penetration to rheology with reasonable accuracy; similar correlations, while not impossible, are more difficult to establish with the softening point.

Proposed DSR-based specifications, unlike the current empirical property-based specifications, address asphalt performance over a comprehensive range of service temperatures. DSR testing provides for evaluation of asphalt fundamental engineering properties at application temperature, as well as high, intermediate, and low service temperatures. Additional provisions are made to include viscosity measurements at rooftop service temperatures including the effect of heating of asphalt prior to application.

References

1. Baumgardner, G. L. Rowe, G. M. “Specifications for Roofing and Industrial Asphalts Using Dynamic Shear Rheometry (DSR),” *Journal of ASTM International* Vol. 4, No. 8 Paper ID JAI101044, 2007.
2. Rowe, G. M., Baumgardner, G.L., “Evaluation of the Rheological Properties and Master Curve Development for Bituminous Binders Used in Roofing,” *Journal of ASTM International* Vol. 4, No. 9 Paper ID JAI101016, 2007.
3. Rowe, G. M. and Sharrock, M. J., “Development of Standard Techniques for the Calculation of Master Curves for Linear-Visco Elastic Materials,” Technical Note, *Journal of Applied Asphalt Binder Technology*, Vol. 1, Issue 1, pp. 72–81, April 2001.
4. Gershkoff, D., “Polymer Modified Bitumens—Performance in Empirical and Rheological Tests,” *Proceedings of the 1st European Workshop on the Rheology of Bitumen Binders*, Brussels, Paper #34, April 1995.
5. Van der Poel, C. J., “A General System Describing the Visco-Elastic Properties of Bitumens and Its Relation to Routine Test Data,” *Journal Applied Chemistry*, Vol. 4, pp. 221–236, 1954.
6. Molenaar, J. M. M., Hagos, E. T., and Van Der Ven, M. F. C., “An Investigation into the Specification of Rheological Properties of Polymer Modified Bitumen,” *3rd Eurasphalt & Eurobitume Congress*, Vienna, Paper 203, Book II, pp. 2080–2091, 2004.
7. Pfeiffer, J. P., and Van Doormaal, P. M., “The Rheological Properties of Asphaltic Bitumens,” *Journal of the Institute of Petroleum Technologists*, Vol. 55, 1936.

8. Rowe, G. M., and Baumgardner, G. L., “Application of Rheological Models to Modified Roofing Asphalts,” *Journal of ASTM International* Vol. 8, No. 10 Paper ID JAI103825, 2011.
9. Brown, A. B., and Sparks, J. W., “Composition and Rheology of Roofing Asphalt,” Special Technical Publication (STP) ASTM International, STP280-EB 1960.
10. Puzinauskas, V. P., “Properties of Roofing 1982.
11. Cullen, W. C. and Johnson, C., “Characterization of Roofing Grade Asphalts,” *Proceedings, 3rd International Symposium on Roofing Technology*, NRCA/NBS 1991.
12. Cramp, A. P., Cullen, W. C., and Tyron, M., “Slippage of Built-Up Roof Membranes, Cause and Prevention,” NBS Report #10950, September 1972.
13. Puzinauskas, V. P., “Specifications for Roofing and Industrial Asphalt,” *Proceedings, 7th Conference on Roofing Technology*, NRCA/NBS Conference Series 1981.

Symbols

δ – Phase Angle
G′ – Storage Shear Modulus
G″ – Loss Shear Modulus
G* - Complex Shear Modulus
G(t) – Shear Relaxation Modulus
E(t) – Extensional Relaxation Modulus
S(t) – Flexural Bending Stiffness at time (t)
Hz – Hertz
Pa – Pascal
kPa – Kilopascal
MPa - Mega Pascal
Pa·s – Pascal Second
Rad/s – Radians per Second

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