DEVELOPMENT OF LABORATORY-BASED EVALUATION CRITERIA FOR HOT-POUR BITUMINOUS SEALANTS

by

Haithem Soliman.

A Thesis

Submitted to the Faculty of Graduate Studies in Partial Fulfillment of the Requirements for the Degree of

Master of Science

Department of Civil Engineering
University of Manitoba
Winnipeg, Manitoba

THE UNIVERSITY OF MANITOBA

FACULTY OF GRADUATE STUDIES

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Abstract

Sealing pavement joints and cracks is one of the essential pavement maintenance practices to protect subsurface layers from the ingress of moisture and debris. Hot-pour sealants are the most prevalent type of sealants and they consist of bitumen modified with polymers. Existing ASTM standards for hot-pour sealants are empirically based standards and may not correlate well with the field performance of sealants. Currently, field studies are the most reliable method to evaluate the performance of sealants in cold climates but such studies are slow and not cost-effective.

This research proposes a test protocol for full characterization of hot-pour bituminous sealants. Four test methods are adopted in this test protocol: Rotational Viscometer (RV), Dynamic Shear Rheometer (DSR), Bending Beam Rheometer (BBR), and Dynamic Mechanical Analyzer (DMA). These four tests characterize the rheological properties of sealants over the wide range of temperatures that they experience during installation and in-service. The major advantage of using these methods is that the equipment is readily available in the asphalt binder and polymer characterization laboratories; and the procedures are familiar to laboratory technicians.

Eight hot-pour sealants were evaluated using the proposed test protocol. Seven parameters were selected as laboratory-based indicators for sealant performance. For each sealant, the values of these performance indicators were evaluated. Sealants were ranked according to their laboratory performance indicators. Results were verified from an ongoing field study that started in 2004. A good correlation was found between the

proposed simplified evaluation method and the field performance. This laboratory evaluation method can replace costly and time-consuming field studies, and provide the ability to test new sealing materials to evaluate their performance as soon as they become available in the market.

Acknowledgements

I would like to thank my academic advisor, Dr. Ahmed Shalaby, for his professional guidance, support and motivation throughout this research. Sincere thanks to the members of my examination committee, Dr. Jeannette Montufar and Dr. Raghavan Jayaraman. In addition, I would also like to thank Mr. Scott Sparrow and Mr. Peter Czehryn for providing the technical support for the laboratory testing for this research.

I would like to acknowledge the support of this research by the Manitoba Graduate Scholarship at the University of Manitoba.

Finally, I would like to extend my sincere gratitude and appreciation to my father, mother, and brothers for their support and continuous encouragement over the course of my study.

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Notation

COV	Coefficient of variation
$D\ (t)$	Creep compliance at time t
D_o	Glassy creep compliance
D_i	Material constant
E^*	Complex modulus
E'	Storage modulus
E''	Loss modulus
G^*	Complex shear modulus
$G\ (t)$	Shear relaxation modulus at time t
G_o	Initial shear modulus
G_∞	Shear relaxation modulus at infinity
G_i	Material constant
g_i	Material constant
I_T	Thermal stress index
m_{60}	*m*-value at 60 seconds
m_{240}	*m*-value at 60 seconds
S	Creep stiffness
S_{60}	Creep stiffness at 60 seconds
TS	Temperature susceptibility

T_g	Glass Transition Temperature
τ_i	Relaxation time
σ_F	The failure stress at -29° C
σ_T	The thermal stress at -29° C
δ	Phase angle

1 Introduction

1.1 General Overview

Joint sealants are widely used in Canada to protect pavements from the infiltration of water and incompressible debris. ASTM standard D6690 (ASTM 2005) is the most commonly used standard for hot-pour sealants in North America. Existing ASTM standards are empirically based standards and may not correlate well with the field performance of sealants. In cold climates, sealants are subjected to a large annual temperature differential that may exceed 80° C. For this reason, field studies are conducted to evaluate the field performance of sealants. Field performance is typically assessed every 10 years, which does not provide a timely response to progressive market changes and the availability of new products.

Joint sealant provides two main functions: protect pavement structure from moisture and prevent the retention of incompressible materials in joints (Lynch 1996). To achieve these functions successfully, the candidate sealant should have: adequate adhesion strength with the pavement, the ability to dissipate tensile stresses in sealant, and adequate stiffness to resist the penetration of incompressible materials. Factors influencing adhesion strength were extensively addressed in the literature such as: sealant viscosity during installation (Masson and Lacasse 2000), the type of aggregate used in pavement mix (Fini et al. 2006), crack preparation (Evans et al. 1995), and installation procedures (Evans et al. 1999).

Hot-pour sealants are the most prevalent type of sealants and they consist of bitumen modified with polymers. The type of polymer can affect the performance of sealants. Hot-pour bituminous sealants can be considered elastomeric polymers with viscoelastic behaviour. Stiffness modulus of a viscoelastic material changes with the change of temperature and time of loading. Therefore, several tests are required to provide a full characterization of sealants over the temperature range that they would experience during installation and in-service.

Field evaluation studies in Manitoba showed that most sealant failures were adhesion failures (Worms 2005). Adhesion between sealants and pavements is affected by sealants viscosity during installation, the aggregate used in pavements mix, crack preparation, and installation procedures. Adhesion failure of a sealant can also be due to the high stiffness of the sealant and the sealant's inability to dissipate the tensile stresses generated at low temperature which lead to a build-up of tensile stresses at the sealant-pavement interface. Sealant's stiffness and ability to dissipate stresses are related to the fundamental rheological properties of sealants (Masson 2000).

Several laboratory evaluation methods were proposed for evaluating the parameters that influence the field performance of sealants at different temperatures. The selected parameters could be the mechanical properties of sealants, their adhesion to different pavement materials, or the chemical composition of sealants. Recently, the utilization of the widely-available asphalt binder performance grading equipment to characterize the rheological properties of crack sealants has been examined.

1.2 Research Objectives

The purpose of this research is to provide a test protocol for laboratory characterization of hot-pour bituminous sealants. Laboratory characterization of sealants can be considered as a cost-effective and rapid alternative to field performance studies. It will provide the ability to test new sealing materials and evaluate their performance as soon as they become available in the market.

The objectives of this research are to:

- Characterize the properties of sealants over the range of temperature that they would experience during installation and in-service.
- Utilize the existing laboratory tests for characterizing the rheological properties of asphalt binder; and the feasibility of using these tests to characterize sealants.
- Propose procedures for laboratory testing of sealants and the required modifications for asphalt binder tests to be applicable for sealants.
- Propose laboratory-based selection indicators for sealant performance to replace or complement field evaluation studies. These selection indicators provide information about sealant properties at the installation and in-service temperatures.
- Compare the proposed selection indicators and actual field performance of sealants.

1.3 Organization of Thesis

This thesis is organized as follows:

- **Chapter 2:** Literature Review

This chapter outlines the function of sealants, the types of sealants, sealant failure modes, and a summary of the several laboratory evaluation methods that have been developed for evaluation of sealants.

- **Chapter 3:** Research Methodology

This chapter presents the tested materials in this research, and outline the experimental program for laboratory evaluation.

- **Chapter 4:** Complex Shear Modulus (G*) and Viscosity of Sealants

This chapter outlines the preparation of the sealant specimen, the testing procedures, and the results of the Dynamic Shear Rheometer and Rotational Viscometer tests.

- **Chapter 5:** Creep Stiffness and Creep Rate at Low Temperature

This chapter presents the preparation of the sealant specimen, the testing procedures, and the results of the Bending Beam Rheometer test.

- **Chapter 6:** Glass Transition Temperature and Dynamic Modulus

This chapter outlines the preparation of the sealant specimen, the testing procedures, and the results of the Dynamic Mechanical Analyzer test.

- **Chapter 7:** Laboratory-Based Performance Indicators for Sealants

This chapter discusses the laboratory test protocol followed in this research, the proposed laboratory selection indicators for sealant performance, and the correlation between laboratory evaluation and field evaluation of sealants

- **Chapter 8:** Conclusions and Recommendations

This chapter provides a summary of the thesis, the conclusions, and some recommendations for future work to improve the proposed method for laboratory evaluation.

2 Literature Review

2.1 Introduction

Joint and crack sealants protect pavement structures from moisture, and prevent the retention of incompressible materials in joints (Lynch 1996). The properties of sealing materials have been improved in the last two decades due to the availability of new materials that could have better sealing performance. Establishing a selection criteria based on laboratory evaluation can decrease the uncertainty about sealant field performance and the suitability of a sealant to site climate. Current ASTM tests for sealants are empirically-based and do not reflect the field performance of sealants. Field studies are the most reliable alternative to ASTM tests to evaluate sealants performance; however they are not cost effective. Several laboratory evaluation methods proposed for evaluating the parameters that influence the field performance at different temperatures. The selected parameters could be the mechanical properties of sealant, its adhesion to different pavement materials, or the chemical composition of sealant.

2.2 Hot-pour Sealants; Classification and Failure Modes

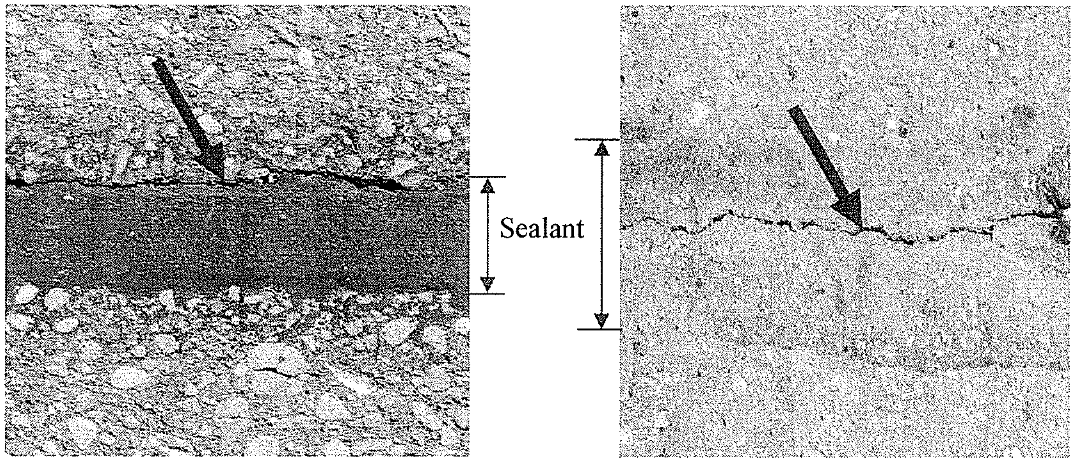
2.2.1 Classification of Hot-pour Sealants

Hot-pour sealants are the most prevalent type of sealants and consist of bitumen modified with polymers. The type of polymer affects the low-temperature performance of sealants. Hot-pour sealants are classified into four groups according to ASTM standards D 6690 (ASTM 2005):

- Type I: A joint and crack sealant capable of maintaining an effective seal in moderate climates. Low temperature performance is tested at -18° C using 50% extension.
- Type II: A joint and crack sealant capable of maintaining an effective seal in most climates. Low temperature performance is tested at -29° C using 50% extension.
- Type III: A joint and crack sealant capable of maintaining an effective seal in most climates. Low temperature performance is tested at -29° C using 50% extension. Special tests are included.
- Type IV: A joint and crack sealant capable of maintaining an effective seal in climates experiencing very cold temperatures. Low temperature performance is tested at -29° C using 200% extension.

2.2.2 Sealant Failure Modes

A sealant is considered to have failed if it can not perform its function properly, which is protecting pavement from the ingress of moisture and debris. Sealant failures are classified into two types: cohesion failure and adhesion failure. Cohesion failure occurs when the cohesion between sealant particles can not withstand the external stresses applied on it and the sealant cracks or ruptures. Adhesion failure occurs when the bond between a sealant and a joint face is not sufficient to resist stresses due to pavement contraction. There are several parameters that may affect the mode of failure, for example: stiffness, ability to dissipate tensile stress with appropriate rate, and climate conditions. Figure 2.1 shows the two modes of sealant failure.



a) Adhesion Failure: separation between sealant and joint face

b) Cohesion Failure: crack in the sealant material

FIGURE 2.1: Modes of Sealant Failure

2.3 Properties of Hot-pour Bituminous Sealants

Hot-pour sealants consist of bitumen modified with polymers. The properties of sealants are affected by the type of polymer. Hot-pour bituminous sealants can be considered elastomeric polymers with viscoelastic behaviour. Stiffness modulus of a viscoelastic material changes with the change of temperature and time of loading. The state of hot-pour sealants changes from a viscous liquid at very high temperatures to an elastic solid at very cold temperatures. Hot-pour sealants are installed at a very high temperature ranging from +180° C to +200° C. At the installation temperature, the state of sealants changes from a rubber to a viscous liquid.

Sealants are subjected to two types of loading after installation: traffic loading and thermal loading. Traffic loading produces repeated shear strains on sealants. Thermal loading is caused by the variation of pavement temperature during the year. Thermal loading produces tensile and compressive strains in sealants. Sealants should operate at the viscoelastic range to be able to dissipate these tensile strains before they induce failure.

2.4 Mechanical Tests Developed for Sealants

Several testing methods are based on evaluating the performance of sealants under monotonic or cyclic stress, or strain. Type of loading and test temperature are selected to simulate the conditions that sealants are subjected to. Some of these testing methods were originally developed for testing asphalt binders or viscoelastic materials and are being adopted for testing sealants.

2.4.1 Tensile Adhesion and Relaxation Tests

Zanzotto (1996) introduced a testing protocol for evaluating sealant performance at low service temperatures based on stress relaxation test and tensile adhesion test. In stress relaxation test, a cylindrical sealant sample of 25.4 mm height and 25.4 mm diameter was subject to 50% extension at a rate of 1 mm/minute and a temperature of -30° C. Each sample was tested for one hour, 12.5 minutes to apply 50% extension and 47.5 minutes to measure stress relaxation. The developed load due to sealant extension was recorded. In tensile adhesion test, a cylindrical sealant sample of 25.4 mm height and 25.4 mm diameter was pulled off a concrete brick at a rate of 10 mm/minute and at a temperature of -30° C. Displacement and corresponding load were recorded until debonding occurred.

Fourteen sealants used in field projects in Alberta, Ontario, and Quebec were tested in the laboratory to correlate stress relaxation and tensile adhesion to field performance. Four parameters, two from stress relaxation test and two from tensile adhesion test, had a good correlation with field performance. These parameters were: maximum peak load during stress relaxation tests, rupture of sealant sample during extension, maximum extension at debonding between the sealant sample and the concrete block in the tensile adhesion test, and the work necessary for debonding. Table 2.1 shows the correlation between these parameters and field performance which can be used as a guideline to predict the field performance of sealants.

TABLE 2.1: Correlation between Field Performance and Parameters from Stress Relaxation and Tensile Adhesion Tests (Zanzotto 1996)

Field Performance	Test Type			
	Stress Relaxation		Tensile Adhesion	
	Peak load (N)	Breakage during extension	Max. extension at debonding (mm)	Work required for debonding (J)
Pass	< 500	Yes	> 5	> 2
Fail	> 500	No	< 1	> 1

2.4.2 Tensile and Compressive stiffness

Abd El Halim et al. (1997) used a displacement controlled test to evaluate the performance of hot and cold-pour sealants in tension and compression at different temperatures. Three types of sealants were evaluated: silicone sealant, cold-pour sealant, and hot-pour sealant. Sealant cubes with a side length of 50 mm were used for compression tests, while dog-bone specimens with a gauge length of 50 mm were used for tensile tests. Four displacement rates were used 2.5, 12.5, 25, and 50 mm/minute.

Test results showed that sealant behaviour in tensile and compressive tests was not affected significantly by changing the rate of displacement.

Tensile and compressive tests were conducted at temperatures of -40°C, -20°C, 0°C, +20°C, and +40°C. In tensile tests, the stiffness of the silicone and the cold-pour sealants were less sensitive to test temperature, while the hot-pour sealants showed higher sensitivity to test temperature. The same behaviour was noticed in compressive tests except that the silicone sealant showed much higher compressive stiffness at temperature -40°C than compressive stiffness at temperatures from -20°C to +40°C.

The calculated tensile modulus at a strain of 10% and a temperature of -40°C for the hot-pour sealant was around 7 times that of the cold-pour sealant and 70 times that of the silicone sealant.The compressive modulus calculated at strain of 10% and a temperature of -40°C for the hot-pour sealant was around 8 times that of the cold-pour sealant and 3 times that of the silicone sealant. The silicone sealant appeared to lose most of its compressive strength by changing temperature from -40°C to -20°C, where the compressive modulus of the silicone sealant at temperature -40°C it about 30 times its value at temperature -20°C.

2.4.3 Cyclic Shear and Horizontal Deflection

Al-Qadi et al. (1999) evaluated the performance of two types of rigid pavement joint sealants under cyclic shear and tensile strain. A fixture designed and fabricated at Virginia Tech was used for this purpose. The tensile strain simulated the effect of concrete slabs contraction at low temperature, while the cyclic shear was used to simulate traffic loading.

The tested specimen consisted of a sealant strip between two 50.80 mm concrete cubes. The width to depth ratio of the sealant strip was chosen to be one. Two types of aggregate were used for preparing the concrete blocks (granite and limestone) to study the effect of aggregate type on sealant performance. Two types of sealants were characterized in this study: a low modulus one-part cold applied silicone sealant, and a one-part cold applied polyurethane sealant. Five values for thickness of sealant strip, which represent joint width, were evaluated. These values are 12.7, 15.9, 19.1, 22.2, and 25.4 mm.

The effect of test temperature was studies in pilot tests, where sealants specimens were tested at temperatures ranging from -36°C to +26°C. Results showed that there is no significant effect of changing test temperature between -36°C and +26°C on sealant response. Therefore, tests were conducted at temperature +23°C (room temperature).

The shear strain amplitude and frequency were chosen to simulate a truck moving at a speed of 88 km/h. The applied shear strain had a pulse waveform (with 0.05 sec loading period, 0.25 sec rest period) and 3.2mm amplitude. The horizontal deflections were varied between 6.2% and 58% of the joint width. A sealant sample was considered to have failed when 20% of the sealant showed adhesion and/or cohesion failure. The number of cycles required for failure was recorded.

Testing results showed that:

- The number of shear cycles required for failure increases with the decrease of joint width for both types of sealants, which means that the resistance to shear deformation increase with the decrease of joint width.
- The number of shear cycles required for failure increases with the decrease of horizontal strain for both sealants, which complies with the previous observation.
- Both types of sealants showed better performance with granite aggregate than their performance with limestone aggregate.

2.4.4 Cyclic Tension and Compression

Worms (2005) evaluated the performance of eight hot-poured sealants, labeled as sealant A to H, using a cyclic tension and compression test at three temperatures: -30°C, 0°C, and +30°C. Two of these sealants were classified as type I according to ASTM standards, while the remaining sealants were classified as type IV. The tested specimen consisted of a sealant strip with 10 mm thickness placed between two concrete blocks with cross-section 50x75mm and height 50mm.

Sealant samples were subjected to a sinusoidal displacement with an amplitude of 2mm. This amplitude was chosen to simulate a temperature variation of 70°C. Pilot tests were conducted to select the suitable frequency and number of cycles that can be applied at each temperature without damaging the specimen. A 0.003 Hz frequency and 25 cycles were used at temperature -30°C; a 1 Hz frequency and 5000 cycles were used at temperature 0°C; and a 1 Hz frequency and 25000 cycles were used at temperature

+30°C. The corresponding load to the applied displacement was recorded. Test was stopped when a cohesion failure noticed or when the measured load dropped by 85% of its initial value.

Based on tests results, three criteria were used to evaluate sealants performance: percent load drop versus temperature, normal stress, and surface stress. The percent load drop, for both tensile and compressive loads, was defined as the percent of the difference between the initial load measured at the first cycle and the final load measured at the end of the test with respect to the initial load. This criteria was used as an indicator for sealant ability to dissipated stresses and sealant initial stiffness. The eight sealants were categorized to three groups according to the percent of load drop: good, satisfactory, and poor performing sealants. Sealants that maintained a consistent percent load drop in the range of 40 to 60 percent, in tension and compression, at all test temperatures were classified as good performing sealants (Sealants D and E).

Three sealants (Sealants F, G, and H) were categorized as satisfactory performing. There were two trend lines for the percent of load drop for this group of sealants. The first tend line was for the percentage of drop in tensile load; a 60 to 80 percent tensile load drop was recorded at -30°C and decreased to 30 to 50 percent at +30°C. The second trend was for the percentage of drop in compressive load; a 40 to 50 percent compressive load drop was recorded at -30°C and increased to 50 to 70 percent at +30°C. These trends showed that this group of sealants had difficulty in resisting thermal loading.

The remaining three sealants (Sealants A, B, and C) were categorized as poor performing sealants in cold climates. These sealants had a higher percent of load drop than the satisfactory performing sealant, where the percentage of drop in tensile load at -30°C ranged from 85 to 90 and the three sealants experienced adhesion failure. This percentage reduced slightly with the increase in test temperature, where the percentage of drop in tensile load at +30°C ranged from 75 to 81. In compression, the percentage of drop in load ranged from 30 to 50 at -30°C, and increased dramatically to 86 percent at +30°C. These results showed that these sealants had a very stiff behaviour at temperature -30°C. Table 2.2 shows the percent of drop in tensile and compressive loads for all sealants.

The second criteria used for sealant evaluation was the developed normal stresses in the sealants due to applied displacement. The normal strains were calculated by dividing the measured displacements (ΔL) for each sealant by the initial thickness of sealant strip (L). The normal stresses were calculated by dividing the recorded load by the nominal cross-sectional area of sealant sample (50 mmx50 mm). The hysteresis loops were developed for each sealant at each test temperature by plotting the strain against the stress. At each test temperature, sealants were classified into three groups according to their performance: good, satisfactory, and poor. This classification was based on the following criteria: initial tensile stresses, the dissipation of stresses with testing cycles, and the shape of hysteresis loops. These three parameters were used to evaluate the sealant stiffness and its performance at each temperature. Table 2.3 shows the classification of sealants at each test temperature according to the normal stress.

TABLE 2.2: Percent Load Drop at the End of Cyclic Testing (Worms 2005)

Sealant	Percent Load Drop					
	at +30°C		at 0°C		at -30°C	
	Tension	Compression	Tension	Compression	Tension	Compression
A	77.39	80.41	86.00 1	82.46	87.62 1	34.24
B	81.25	86.37	86.60 1	88.48	87.14 1	48.23
C	74.34	77.37	86.88	90.45	88.38 1	42.56
D	55.16	49.74	58.03	63.76	53.37	40.80
E	52.37	44.76	56.61	59.45	45.88	42.67
F	35.85	31.68	70.12	70.00	79.97 1	43.79
G	31.55	28.96	54.62	54.43	61.34	38.60
H	46.90	44.14	63.40	67.48	67.92	48.25

[1] Adhesion Failure Noted

The third criteria used for evaluating sealants performance was the surface stress. Due to the fact that the sealant shape is extending (or contracting) in a curved parabolic shape, the actual cross-sectional area of sealant sample is smaller (or larger) than the initial state before applying tensile (or compressive) load. This fact was not taken into account in the stress calculations. Lynch (1996) recommended a mathematical model, developed by Tons (1959), which accounted for the change in sealant's cross-sectional area based on conservation of volume. This mathematical model was used to compute the surface tensile stress. The average percent difference between theoretical and surface stresses was 12% at +30°C, 7.6% at 0°C, and 5.2% at -30°C. It can be noticed that the percent difference between the two stresses decreases with the decrease of test temperature. This condition can be due to the increase of sealant stiffness which leads to less change in sealant's cross-sectional area. Table 2.4 shows the classification of sealants according to the surface stresses. From Tables 2.3 and 2.4, there is a good agreement between using normal stress and surface stress in sealants classification.

TABLE 2.3: Classification of Sealants According to Normal Stress (Worms 2005)

Sealant	Sealant Performance		
	at +30°C	at 0°C	at -30°C
A	Poor	Poor	Poor
B	Poor	Poor	Poor
C	Satisfactory	Satisfactory	Poor
D	Satisfactory	Good	Good
E	Satisfactory	Good	Good
F	Good	Good	Satisfactory
G	Good	Good	Satisfactory
H	Good	Good	Satisfactory

TABLE 2.4: Classification of Sealants According to Surface Stress (Worms 2005)

Sealant	Sealant Performance		
	at +30°C	at 0°C	at -30°C
A	Poor	Poor	Poor
B	Poor	Poor	Poor
C	Satisfactory	Satisfactory	Poor
D	Satisfactory	Good	Good
E	Satisfactory	Good	Good
F	Good	Good	Satisfactory
G	Good	Good	Satisfactory
H	Good	Good	Satisfactory

2.5 Adopting Asphalt Binder Characterization Tests

2.5.1 Bending Beam Rheometer (BBR)

The BBR test comprises a 3 point bending apparatus. The applied load is 980 milliNewton (according to AASHTO standards T313) and the maximum deflection is limited to 5 mm. Al-Qadi et al. (2005) adopted the Bending Beam Rheometer (BBR) test for evaluating sealants and found that the standard BBR test is not appropriate for testing soft hot-pour sealants, where the measured deflection exceeded the permissible range of the apparatus. Al-Qadi et al. (2005) proposed doubling the thickness of the standard BBR specimen to reduce the specimen deflection. The proposed dimensions for the modified BBR specimen were 102mm in length, 12.7mm in thickness (replacing 6.35mm for the standard BBR specimen), and 12.7mm in width. The virtual work method was used to determine the effect of increasing beam thickness on the contribution of shear deflection to the total deflection. It was found that the shear deflection contribution increased from 1% to 4% by doubling the beam thickness. The repeatability of the test results was checked by testing ten different sealants at -40°C with a minimum of three replicates per each sealant. The coefficient of variation (COV) for all results was less than 19%, and almost 72% of results had a COV less than 10%.

Al-Qadi et al. (2006) adopted the modified BBR test to characterize the performance of eight hot-pour bituminous sealants. The eight sealants were divided into two groups with four sealants in each group: stiff sealants, and soft sealants. The stiff sealants were tested at -4°C and -10°C, while the soft sealants were tested at temperatures -28°C, -34°C, and -40°C.

Marasteanu and Anderson (2000) introduced an approach to verify the linear viscoelastic behaviour of asphalt binders tested with BBR. This approach is based on satisfying two conditions:

- The measured stiffness at different load levels remains constant.
- The summation of the measured responses to a sequence of loads should be equal to the measured response to the summation of these loads (the linear superposition principle).

This approach was adopted to verify the linearity of the viscoelastic behaviour of sealants. Sealants did not satisfy the second linearity condition at all test temperatures, this could be due to the differences between the composition of asphalt binder and bituminous sealants.

Creep stiffness at 240 seconds, rate of stiffness change (*m*-value), steady-state creep rate, and average creep rate were used to rank the tested sealants according to their performance at low temperature. Creep stiffness at 240 seconds was used for predicting sealant stiffness after 5 hours of loading according to time-temperature superposition principle. The 5 hours loading time was selected based on the daily temperature variation in two field test locations in United States during the winter. A good agreement was found among these different parameters.

Elseifi et al. (2006) developed a linear viscoelastic constitutive model for the stress-strain relationship of bituminous sealants at low temperatures ranging from -28°C to -40°C.

This model was developed by fitting the sealant creep compliance measured by the modified BBR test to the following mathematical viscoelastic model:

$$D(t) = D_{\circ} + \sum_{i=1}^{K} D_i \left(1 - e^{-t/\tau_i}\right) \tag{2.1}$$

Where:

$D(t)$ = Creep compliance at time t,

D_o = Glassy creep compliance,

D_i = Material constant,

τ_i = Relaxation time.

Finite element software (ABAQUS) was used to verify the results. A three-dimensional model of the modified BBR sample was analyzed, and the viscoelastic properties of sealant material were defined by creep compliance function. Shear relaxation modulus was used as a second viscoelastic function to define sealant material. Shear relaxation modulus was represented by Prony series expansion as follows:

$$G(t) = G_{\circ} - \sum_{i=1}^{K} G_i \left(1 - e^{-t/\tau_i}\right) \tag{2.2}$$

Where:

$G(t)$ = Shear relaxation modulus at time t,

G_o = Initial shear modulus,

G_i = Material constant,

τ_i = Relaxation time.

The creep compliance data, measured by modified BBR test, were converted to shear relaxation data using a built-in function in the finite element software with some assumptions. These data were used to fit the shear relaxation model and define the magnitude of its parameters.

A good agreement was found between the measured deflections and the deflections calculated from the finite element model. According to these results and given that the sealant behaviour in the linear viscoelastic region, the Prony-series expansion was suggested as an adequate mathematical model for the stress-strain relationship of bituminous sealants at low temperatures.

2.5.2 Dynamic Shear Rheometer (DSR)

Lynch and Janssen (1999) characterized the viscoelastic properties of silicone sealants by using Dynamic Shear Rheometer (DSR) test. The DSR test was conducted on six silicone sealants at temperatures ranging from -30°C to +50°C in 10°C increments and at different frequencies. The DSR data, complex shear modulus (G^*) and phase angle (δ), were used to construct master curves at selected temperatures. Prony series was used for modelling the viscoelastic behaviour of sealants. The shear modulus was represented according to Prony series as follows:

$$g_R(t) = \frac{G(t)}{G_o} = 1 - \sum_{i=1}^{N} g_i^p (1 - e^{-t/\tau_i}) \tag{2.3}$$

$$g_i^p = \frac{g_i}{G_o} \tag{2.4}$$

And, for a solid material

$$G_o = G_\infty + \sum_{i=1}^{N} g_i \tag{2.5}$$

Where:

$G(t)$ = Shear relaxation modulus at time t,

G_o = Initial shear modulus,

G_∞ = Shear relaxation modulus at infinity,

g_i = Material constant,

τ_i = Relaxation time.

A numerical analysis was conducted for a three-dimensional model for joint sealants. The model was analyzed by assuming plain strain conditions. The Prony series parameters estimated from the DSR test results were used to define the properties of sealant material that are used as input to the finite element software. For elongation up to 25 percent, it was found that the numerical model can give representative results to the true values.

Masson (2000) found that the rheological properties of bituminous sealant are related to sealant stiffness and stress relaxation ability at in-service temperatures. The DSR test was one of the recommended methods to address the rheological profile of sealants.

2.5.3 Direct Tension Tester (DTT)

Zhai and Salomon (2005) used direct tension tester (DTT) to evaluate the low-temperature performance of bituminous sealants. Six hot-pour and two cold-pour sealants were evaluated in this study. The DTT was conducted on each sealant in two different modes. The first test mode was the standard DTT for testing asphalt binders, where the failure stress and strain were measured at temperature -29° C. For sealants specimens that did not rupture during the test, the maximum stress was recorded as failure stress. In the second test mode, the thermal stress was measured by restraining the ends of the DTT specimen and the thermally-induced stresses due to a temperature drop from 5° C to -30°

C were recorded. This method simulates the Thermal Stress Restrained Specimen Test (TSRST). The TSRST is commonly used in evaluating the low-temperature cracking susceptibility of asphalt paving mixtures.

Thermal stress index (I_T) was used to rank sealants according to their resistance to temperature changes. Thermal stress index was calculated using the following expression:

$$I_T = \frac{\sigma_T}{\sigma_F} \times 100 \tag{2.6}$$

Where:

σ_T = the thermal stress at -29° C

σ_F = the failure stress at -29° C

Values of thermal stress index ranged from 0 to 100. The higher value of thermal stress index means the less ability of sealant to resist temperature change. The thermal stress index was used to rank hot-pour and cold-pour sealants.

2.5.4 Rotational Viscometer (RV)

Masson and Lacasse (2000) reported some of the mechanisms that explained the adhesion between bituminous crack sealants and asphalt concrete (AC). These mechanisms were affected by the sealant viscosity. Adsorption and mechanical interlock theories are examples of these mechanisms. In adsorption theory, the adhesion strength between a liquid and a solid is affected by the ability of the liquid to spread over the surface of the solid material which is function of the viscosity of the liquid material. In mechanical interlock mechanism, the interlocking is generated by the filling of micro-voids of the solid material with the liquid material which is strongly affected by the viscosity of the liquid material.

Masson et al. (2002) measured the viscosity of hot-pour bituminous sealants using a Bohlin Visco-88-BV viscometer at installation temperatures. The sealant was stirred for 30 minutes in a closed vessel before testing and the temperature was maintained constant at 185 ± 1° C. Results showed that sealants with viscosity less than 10 Pa.s were self leveling and were expected to have a good adhesion, while sealants with viscosity greater that 30 Pa.s were difficult to pour and expected to have a poor adhesion. A good agreement was found between the sealant viscosity and the field performance of sealant after one year of installation.

Al-Qadi et al. (2006) addressed the parameters that can affect the measured viscosity of hot-pour bituminous sealants with Brookfield rotational viscometer. These parameters were: spindle size, spindle speed, test temperature, and waiting time before taking viscosity measurements. Three sealants with low, medium, and high stiffness were selected for a set of pilot tests to determine the appropriate values of these parameters. These sealants were tested twice using two spindle sizes: SC4-29 (7.6mm diameter) and SC4-27 (11.76mm diameter). Results obtained by the SC4-27 spindle were more repeatable. To address the effect of spindle speed, the viscosity of the three sealants was measured with different spindle speeds. Spindle speed was changed in ascending and descending orders. Results showed that sealant viscosity decreases with the increase of spindle speed. A spindle speed of 60 rpm was recommend based on these results.

A waiting time was required to allow for the spindle to stabilize before taking viscosity measurements. The effect of the waiting time was examined by recording viscosity measurements at different waiting times. Results showed that the measured viscosity stabilized after 5 to 10 seconds of spindle rotation. A waiting time of 30 seconds was suggested to be used. The measured viscosity at significantly longer waiting times was found to be not representative of the field conditions.

Sealants were tested at the recommended temperature for installation. To address the effect of changing the installation temperature on sealant viscosity, six sealants were tested at installation temperature and at installation temperature $\pm$ 10°C. Results showed that the viscosity of some sealants are very sensitive to temperature changes. The viscosity of one sealant was increased by almost 40 percent due to a temperature increase of 10° C.

2.6 Dynamic Mechanical Analyzer (DMA)

Hot-pour bituminous sealants can be considered elastomeric polymers with viscoelastic behaviour. Stiffness modulus of a viscoelastic material changes with the change of temperature and time of loading. The stiffness-temperature behaviour of a bituminous sealant, as a viscoelastic material, can be divided into four regions (Aklonis et al. 1972, Rogers et al. 1999) as shown in Figure 2.2.

At very low temperatures, the sealant material is in the glassy region and is both stiff and brittle. The behaviour of sealant in this region is best approximated to that of an elastic solid. With the increase of temperature, the thermal energy increases until it becomes capable to overcome the potential energy restricting chain segments from rotation and translation. At this point, sealant modulus starts to decrease and moves toward the transition region. In the transition region, sealant modulus decreases rapidly and the viscous behaviour is noticeable. The width of the transition region varies from 5°C to more than 20°C. The temperature at which the state of sealant material changes from glassy (solid) to rubbery is called the glass transition temperature (Tg). With further increase in temperature, the material modulus enters the rubbery plateau region. In the rubbery plateau region, the modulus changes at a much slower rate compared to the rapid drop in the transition region. When the temperature reaches the melting temperature of the material, the sealant turns to a viscous liquid.

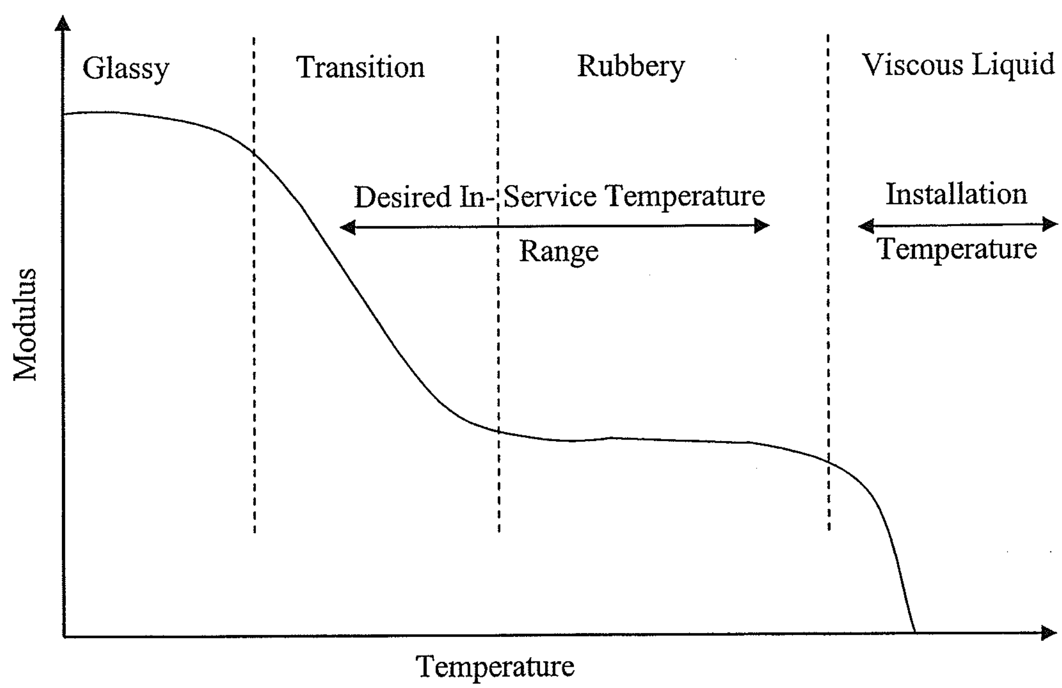


FIGURE 2.2: Stiffness-Temperature Behaviour of a Hot-Pour Sealant

The glass transition temperature (T_g) of a sealant is an important criteria in predicting its field performance. If the glass transition temperature of a sealant is higher than the in-service temperature, the sealant material will become very stiff (in the glassy region) and an increase in sealant cohesion and adhesion failures may occur.

The change of state from rubbery to solid state is associated with a distinct change in the coefficient of thermal expansion. Glass transition temperature can be considered as the temperature at which the change in the coefficient of thermal expansion occurs (Aklonis et al. 1972). Therefore, glass transition temperature of a material can be determined experimentally from the coefficient of thermal expansion test.

Glass transition temperature can also be determined from dynamic testing by measuring the complex modulus of the viscoelastic material at different temperatures. The complex modulus can be represented by two components: storage modulus (elastic component), and loss modulus (viscous component). The relationship between them is as follows (Figure 2.3):

$$E^* = E' + i E'' \tag{2.7}$$

$$\tan\delta = \frac{E''}{E'} \tag{2.8}$$

Where:

E^* = Complex modulus,

E' = Storage modulus,

E'' = Loss modulus,

δ = Phase angle,

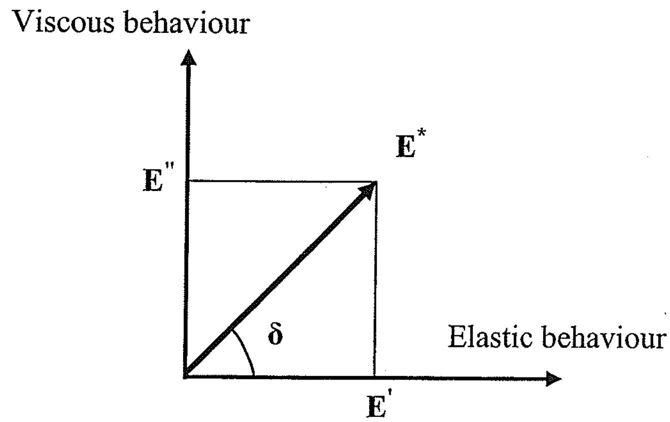


FIGURE 2.3: Viscoelastic Behaviour of Sealants

Dynamic mechanical analyzer (DMA) is one of the dynamic tests that can be used to determine glass transition temperature. Glass transition temperature can be determined from one of the following relationships (Figure 2.4): tan δ-temperature, loss modulus-temperature, or storage modulus-temperature. It can be considered as the temperature corresponding to (Aklonis et al. 1972):

- the peak of tan δ-temperature curve,
- the peak of loss modulus-temperature curve, or
- the intersection of tangents to storage modulus-temperature curve

2.6.1 DMA Test for Bituminous Sealants

Masson used the DMA test for studying the temperature effect on the viscoelastic properties of bituminous sealants (*11*). Two bituminous sealants were tested at a temperature range of -70° C to +50° C. For the first sealant, tan δ curve showed two peaks at -35° C and -20° C and the transition zone extended from temperature -10° C to temperature -40° C. For the second sealant, tan δ curve had only one peak at temperature -35° C with lower amplitude than that for the first sealant and the transition zone extended from temperature -20° C to temperature -50° C.

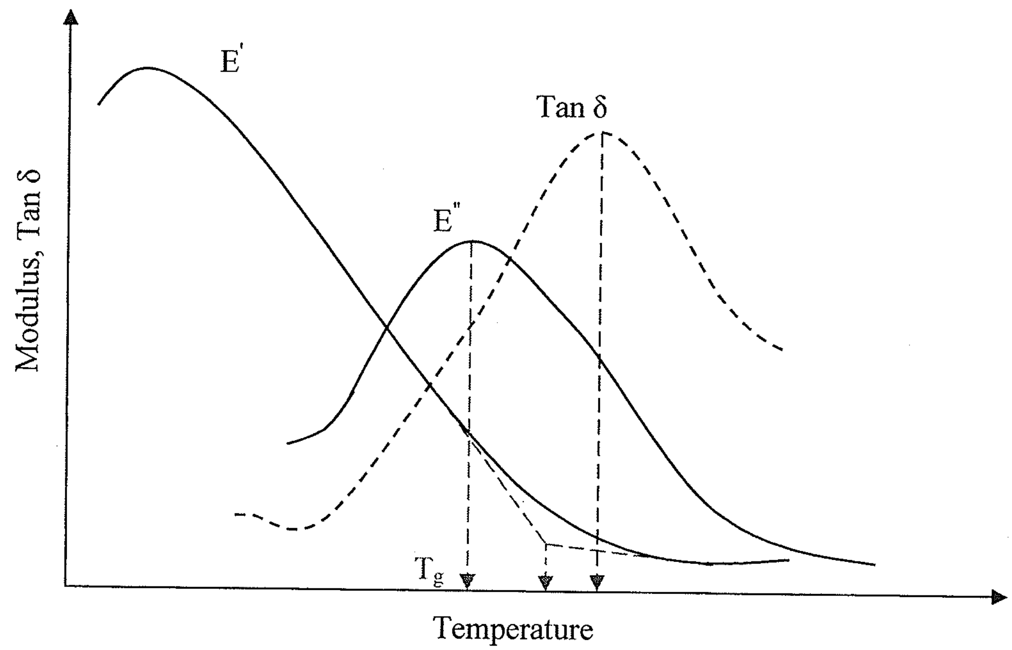


FIGURE 2.4: Determination of Glass Transition Temperature from Dynamic Testing

2.6.2 DMA Test for Polyurethane Sealants

Rogers et al. (1999) studied the effect of curing time on the glass transition temperature of polyurethane sealants. Two cold-pour, self-levelling, two-component polyurethane sealants were evaluated in this study using the DMA test. Sealant specimens, measuring 10 mm x 35 mm x 4 mm, were tested in a dual cantilever clamp. Specimens were subjected to oscillating strain at a frequency of 1 Hz and amplitude 10 μm. The two sealants were tested at two temperature ranges: from -70° C to +40° C and from -50° C to +40° C. Temperature was ramped at a rate of 2° C per min.

The tan δ-temperature curves for the two sealants showed one clear peak and the glass transition temperature of each sealant. For 24 hours curing time, the glass transition temperatures of sealants A and B were -21.4° C and -48.9° C, respectively.

3 Research Methodology

3.1 Introduction

In cold climate, joint and crack sealants are subjected to large annual temperature differential that may exceed +80°C (from -45°C to +35°C). Field evaluation studies conducted in Manitoba showed that most of sealant failures were adhesion failures. Adhesion between sealants and pavements is affected by sealant viscosity during installation (Masson and Lacasse 2000), the aggregate used in pavement mix (Fini et al. 2006), crack preparation (Evans et al. 1995), and installation procedures (Evans et al. 1999). Adhesion failure of sealants also can be due to the high stiffness of sealants and the sealants' inability to dissipate the tensile stresses generated at low temperature which lead to build-up of tensile stresses at the sealant-pavement interface. These properties are related to the fundamental rheological properties of sealants (Masson 2000).

The glass transition temperature is the boundary temperature between the rubbery and Glassy states of a viscoelastic material. The glass transition temperature (Tg) of a sealant is an important criteria in predicting its field performance. If the glass transition temperature of a sealant is higher than the in-service temperature, the sealant material will become very stiff and an increase in sealant failure may occur. The glass transition temperature of sealants can be evaluated from dynamic testing.

3.2 Tested Materials

Eight hot-pour bituminous sealants were evaluated in this study. Of the eight hot-pour sealants, two sealants fall under Type I and the remaining six under Type IV according to

ASTM standard D 6690. The two Type I sealants are labeled as sealant A and sealant B while the six Type IV sealants are labeled sealants C to H. Table 3.1 lists the material properties of Type I and Type IV sealants according to the datasheets received from each manufacturer.

TABLE 3.1: Properties of Sealants (Manufacturer's Datasheets)

Sealant	Penetration (1/10 mm)	Flow (mm)	Bond test	Resilience (%)	Asphalt compatibility	Max. Heating temp. (°C)	Application temp. (°C)
Type I Sealants (Bond test at -18°C)							
A	80	nil	Pass	N/A[a]	N/A	N/A	185-200
B	100	3 Max.	Pass	30%	Pass	204	188-199
Spec. limits	90 Max.	5 Max.	Pass 5 cycles @ 50% ext.	N/A	Pass	N/A	N/A
Type IV Sealants (Bond test at -29°C)							
C	103	nil	Pass	80%	Pass	N/A[a]	185-200
D	100-150	10	Pass	30-60%	Pass	204	193-204
E	130	3	N/A	30%	Pass	204	188-198
F	120	1	Pass	70%	Pass	200	170
G	120	3	Pass	54%	Pass	200	170
H	100-150	10	Pass	30-60%	Pass	210	193
Spec. limits	90-150	3 Max.	Pass 3 cycles @ 200% ext.	60% Min.	Pass	N/A	N/A

[a] N/A = not available

Sealant properties were verified by a certified third-party laboratory and the results of the verification tests are shown in Table 3.2. Datasheets received from manufacturers do not provide adequate information about the characteristics of the rubber and polymers added to these sealants.

TABLE 3.2: Properties of Type I and IV Hot-Pour Sealants (Verification Tests)

Sealant	Penetration (1/10 mm)	Flow (mm)	Bond test	Resilience (%)	Oven Aged Resilience (%)	Asphalt compatibility
Type I Sealants (Bond test at -18°C)						
A	67	2	Pass	82	65	Pass
B	95	1	Pass	67	63	Pass
Type IV Sealants (Bond test at -29°C)						
C	95	2	Pass	83	72	Pass
D	115	1	Pass	68	66	Pass
E	148	1	Pass	71	68	Pass
F	116	0	Pass	54	52	Pass
G	115	0	Pass	53	52	Pass
H	121	0	Pass	72	72	Pass

3.3 Laboratory Evaluation

Hot-pour bituminous sealants are viscoelastic material. The stiffness of sealants is temperature dependent. Sealants are subjected to a wide range of temperatures during installation and in-service (ranging from +180°C, or higher, during installation to -40°C, or less, during winter). Therefore, more than one test is required for characterizing the rheological properties of sealants.

The asphalt binder Performance Grade (PG) equipment was utilized in this research to characterize the rheological properties of hot-our bituminous sealants. Four laboratory tests were adopted in this research for full characterization of sealants, these tests are:

- Rotational Viscometer (RV) test: to evaluate the viscosity of sealants at the installation temperature.
- Dynamic Shear Rheometer (DSR) test: to characterize the rheological properties of sealants at moderate at high temperatures.
- Bending Beam Rheometer (BBR) test: to characterize the rheological properties at the minimum in-service temperature.
- Dynamic Mechanical Analyzer (DMA) test: to characterize the properties of sealants at low temperatures and evaluate the glass transition temperature (T_g).

The RV, DSR, BBR, and DMA tests can fully characterize the properties of hot-pour bituminous sealants over the range of temperatures that they are subjected to. Figure 3.1 shows the four tests adopted in this research and the range of temperature that each test covers.

Sample preparation, test procedures, and results of each test are discussed in chapters 4 to 6. The summary and discussion of these results are presented in chapter 7.

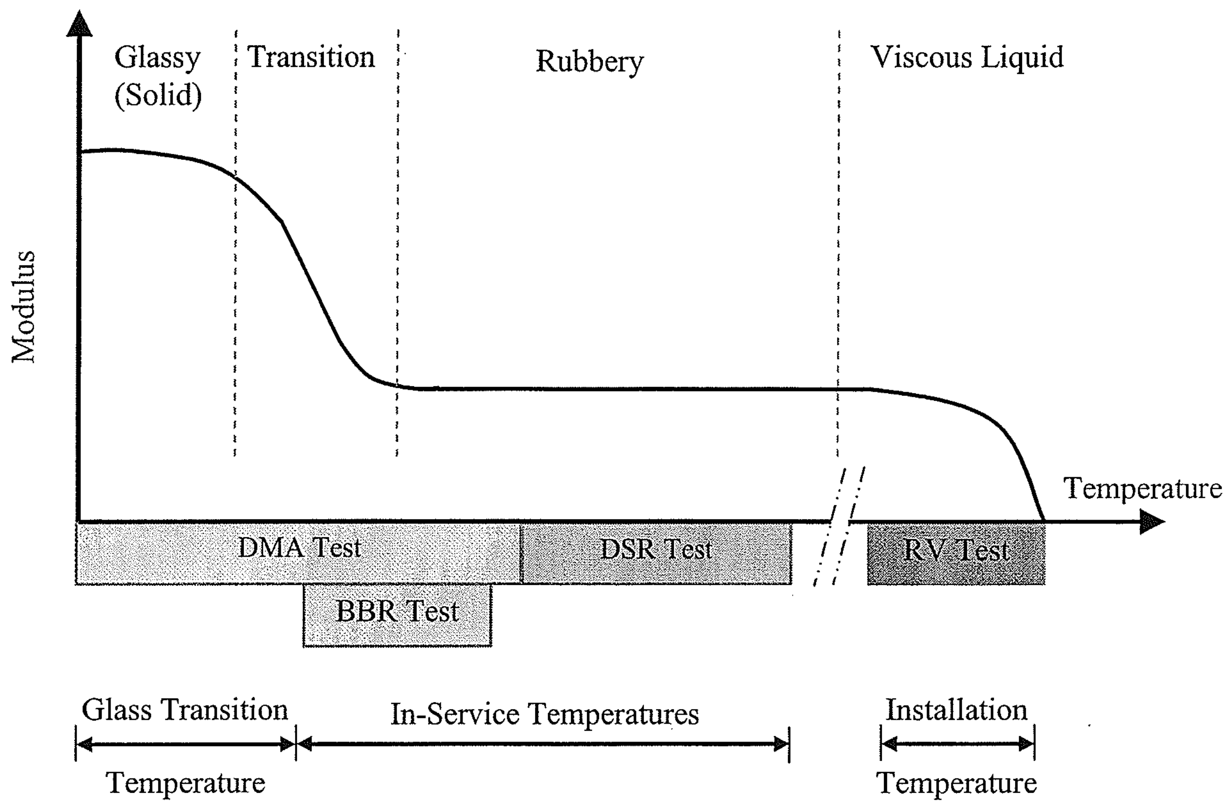


FIGURE 3.1: Laboratory Tests for Full Characterization of Sealants

4 Complex Shear Modulus (G^*) and Viscosity of Sealants

4.1 Introduction

Dynamic Shear Rheometer (DSR) test is originally developed for testing Performance Grades (PG) of asphalt binders based on complex shear modulus (G^*). DSR test is used for evaluating binder performance with respect to permanent deformation (rutting) and fatigue failure criteria. It is conducted on non-aged and Rotating Thin Film Oven (RTFO) aged binders to assess resistance to permanent deformation, and on Pressure Aging Vessel (PAV) aged binders to assess resistance to fatigue cracking.

The DSR test is adopted in this research to characterize rheological properties of sealants at moderate and high temperatures ranging from +5°C to +64°C.

Sealant Viscosity during installation is one of the factors that affect the adhesion between sealants and pavement walls (Masson and Lacasse 2000). Rotational Viscometer (RV) test is used for evaluating the viscosity of asphalt binders as one of the requirements of Performance Grade classification system. The rotational viscometer test is utilized in this study to measure the viscosity of sealants at the installation temperature recommended by the manufacturers.

This chapter introduces testing procedures and results of DSR and RV tests (Figure 4.1).

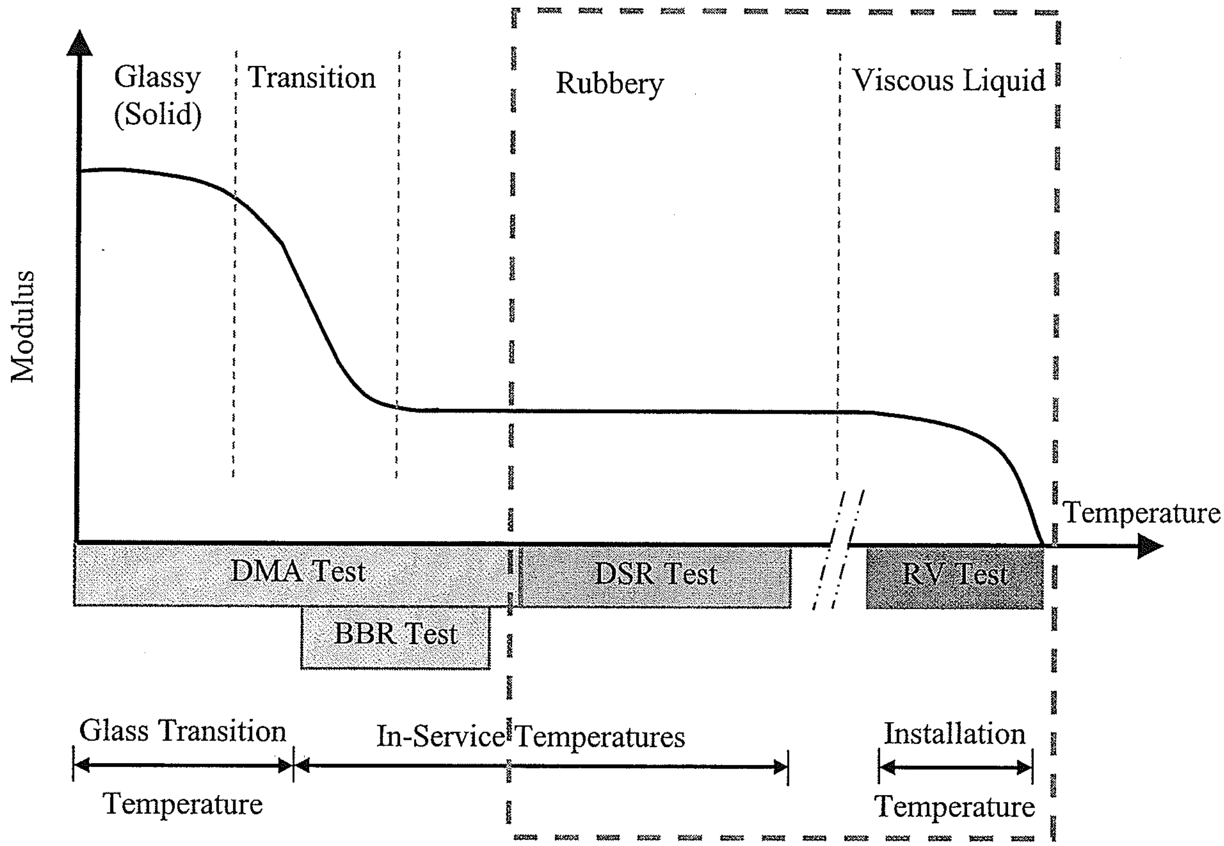


FIGURE 4.1: Evaluation of Sealants Using DSR and RV Tests

4.2 Dynamic Shear Rheometer (DSR) Test

4.2.1 Sample Preparation

The DSR test was conducted on the eight hot-pour sealants involved in this study. Representative samples were taken for each sealant by taking three top-to-bottom slices from three locations in each sealant block. The middle parts of these three slices were combined and heated to the installation temperature recommended by the manufacturers, and then used for preparing test specimens. The 25.0 mm diameter plate was adopted for

DSR test at all temperatures with a 1.0 mm gap. Figure 4.2 shows the Bohlin DSR apparatus used in this research.

Before placing the specimen in the DSR apparatus, the gap is adjusted to be 1.0 mm plus an extra 0.05 mm. The heated sealant is poured in a mold to form a sealant disk, and then this disk is placed between the fixed and oscillating plates of the DSR apparatus. The sealant specimen is trimmed to be flush with the plates' edge and the oscillating plate is lowered by 0.05 mm to get exactly 1.0 mm gap. The specimen shape is slightly bulged as indicated in Figure 4.3.

4.2.2 Verification of Linearity

A sinusoidal strain was applied to the sealant specimen with frequency 1.5 Hz. Pilot tests were conducted on selected sealants to select the appropriate strain amplitude . Sealants were subjected to different strain amplitudes and temperatures to verify that the measured complex shear modulus is in the linear viscoelastic region. The measured complex shear modulus is considered in the linear viscoelastic region if the drop in the shear modulus, with the increase of the strain amplitude, is less than 5% (Marasteanu and Anderson 2000).

Based on the pilot tests, the strain amplitude was selected to be 2% for test temperatures ranging from +5°C to +40°C and 4% for test temperatures ranging from +46°C to +64°C. Figure 4.4 shows an example of the results of pilot tests conducted to select the appropriate strain amplitudes.

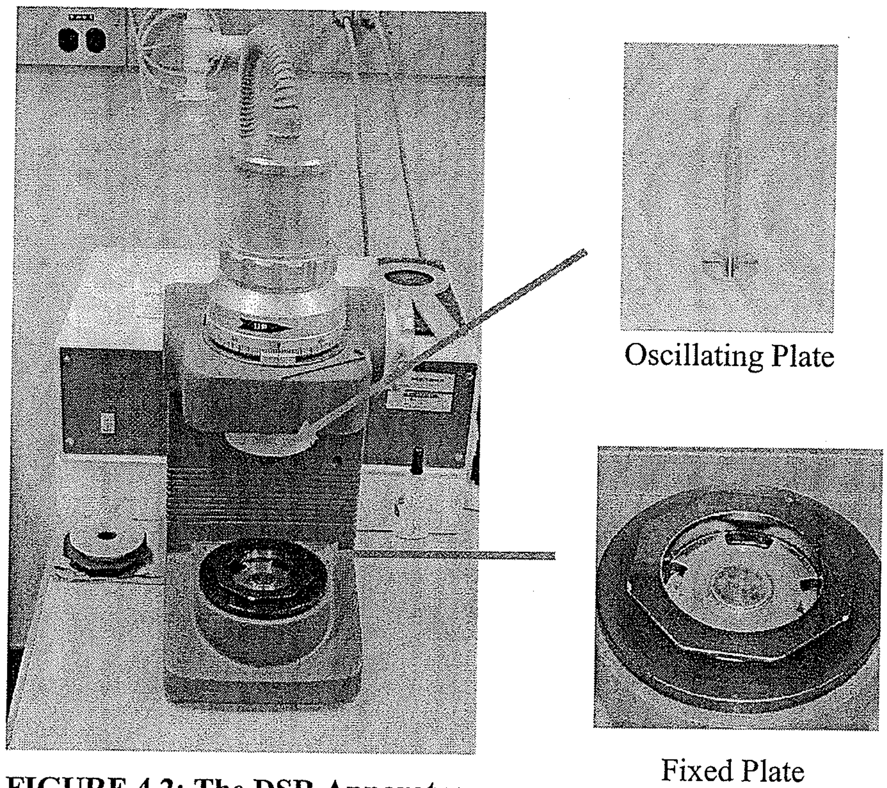


FIGURE 4.2: The DSR Apparatus

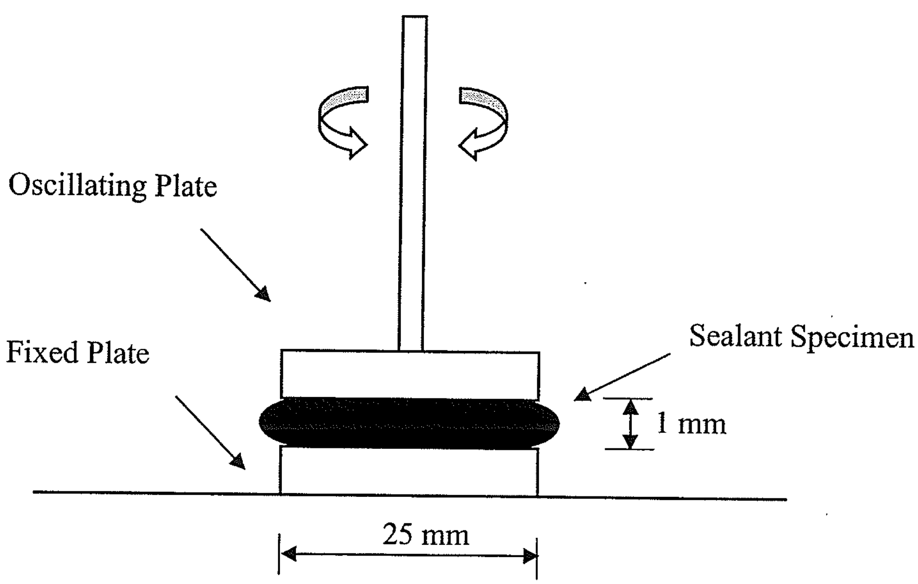


FIGURE 4.3: The Sealant Specimen for the DSR Test

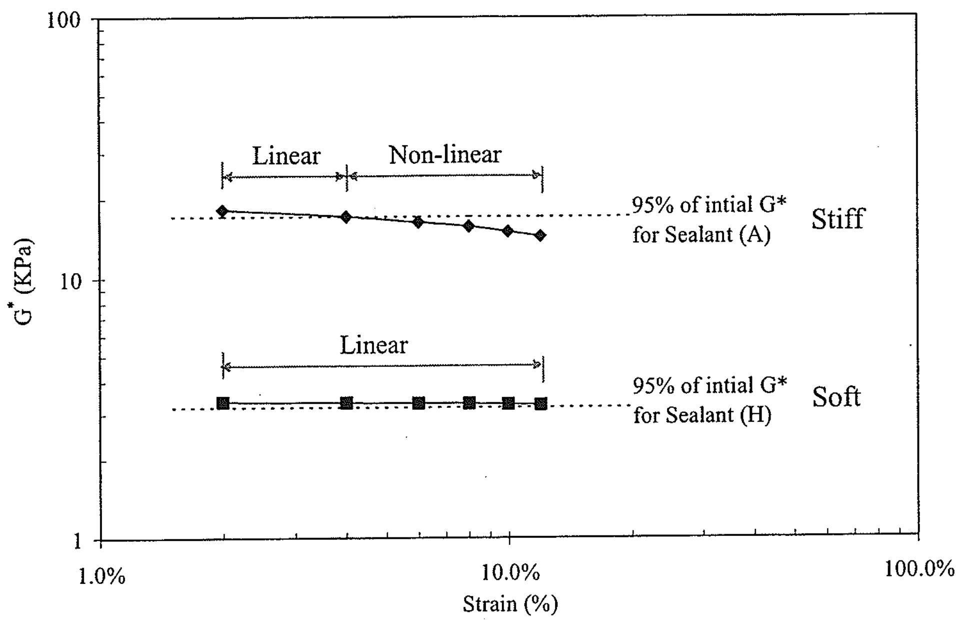


FIGURE 4.4: Linearity Check for Sealants (A) and (H) at +64°C

4.2.3 Complex Shear Modulus (G^*) and Phase Angle

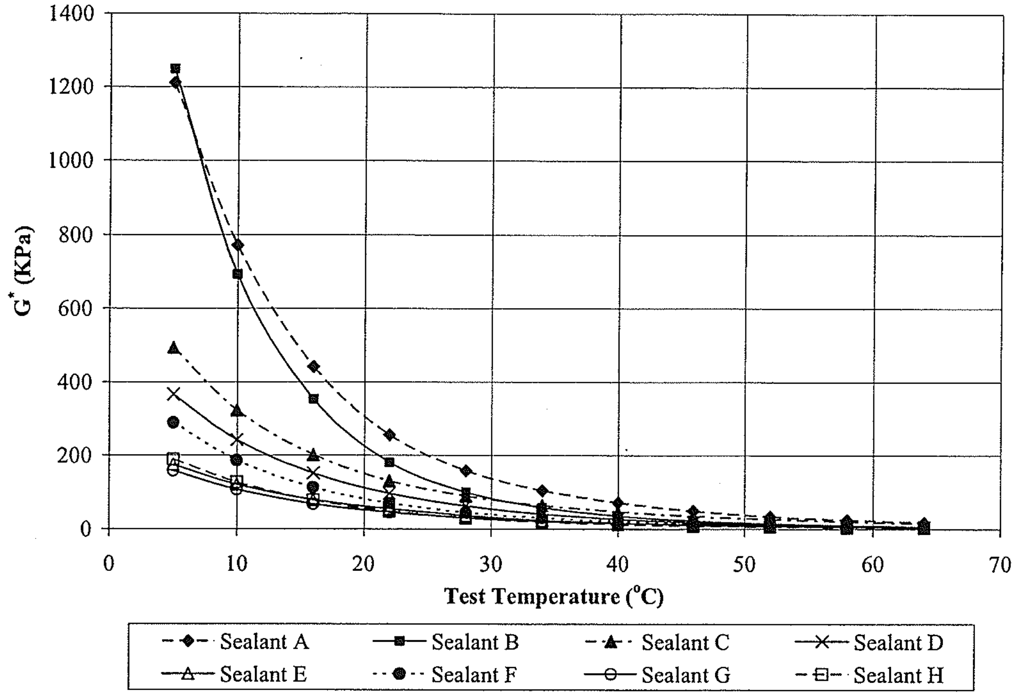
Sealants were tested at temperatures ranging from +5°C to +64°C. A water bath was used to control specimen temperature. Ten minutes waiting time was allowed for specimen temperature to equilibrate. At each test temperature, the sealant specimen was subjected to ten conditioning cycles followed by another ten cycles for obtaining test results. At temperatures ranging from +5°C to +16°C, the 2% strain could not be achieved for stiff sealants (A, B, and C), where the stress that can be applied by the DSR appartus to the 25 mm specimen is limited to 3228 Pa.

Figure 4.5 and Table 4.1 show the complex shear modulus (G^*) obtained from the DSR test. Sealants A and B have G^* higher than 1200 KPa at +5°C. At the same temperature, sealants H, G and E have G^* less than 200 KPa. Sealants A and B were more susceptible to temperature change than the other sealants. The shear modulus of sealants B and A dropped by 556.2 and 450.10 KPa, respectively, with the increase of temperature from +5°C to +10°C.

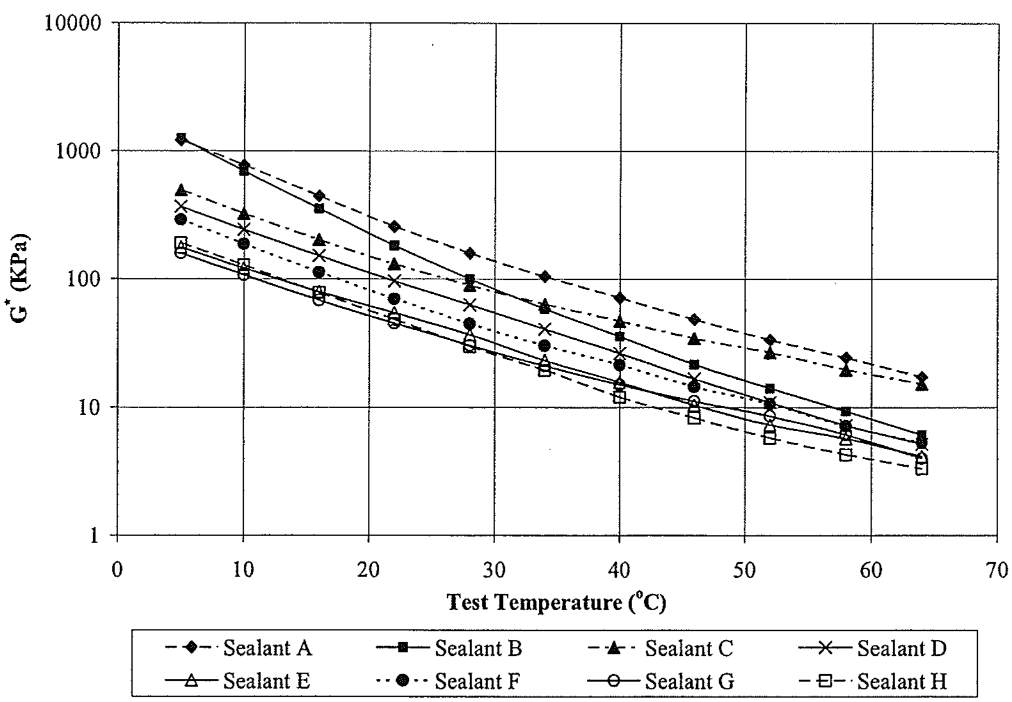
TABLE 4.1: Complex Shear Modulus (G*) for the Tested Sealants

Sealant	G^* (KPa)		
	+5°C	+34°C	+64°C
A	1211	104	17.1
B	1249	58.0	6.1
C	494	63.4	15.1
D	365.8	40.3	5.2
E	175.1	23.0	4.2
F	288.8	30.1	5.3
G	158.1	20.9	4.0
H	189.4	19.3	3.3

Figure 4.6 shows the measured phase angles for the tested group of sealants. The measured phase angles ranged from 23 to 48 degrees and they were not sensitive to the test temperature in the range of +5°C to +64°C.



a) Storage modulus (Normal scale)



b) Storage modulus (Logarithmic scale)

FIGURE 4.5: Complex Shear Modulus (G*) for the Tested Sealants (Normal and Logarithmic Scales)

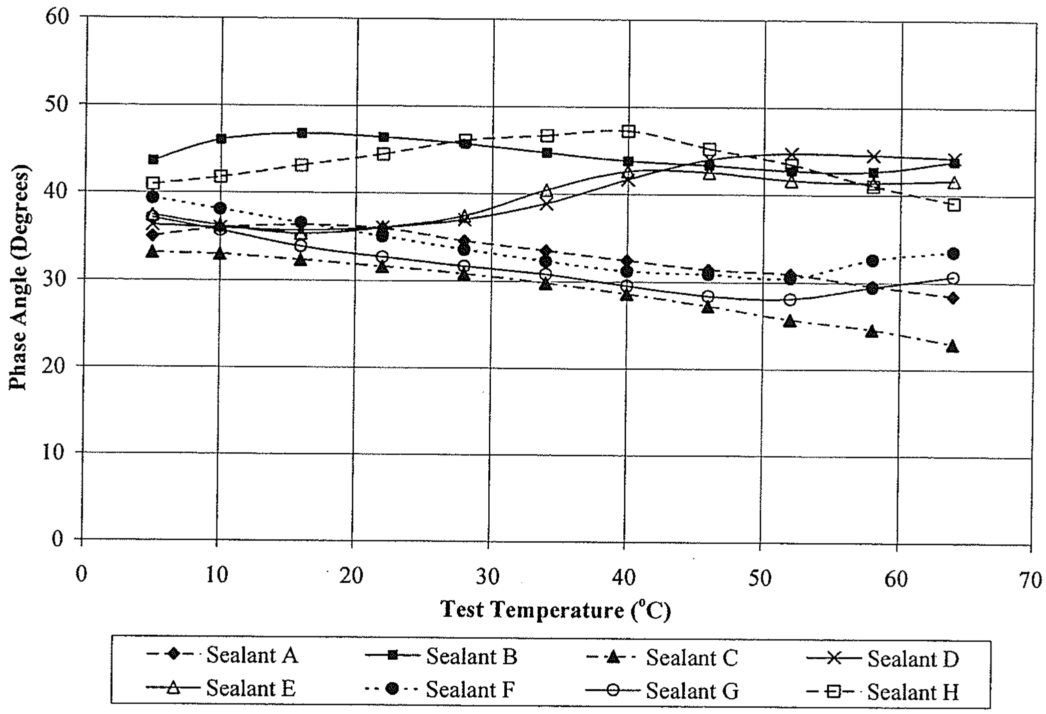


FIGURE 4.6: Phase Angle for the Tested Sealants

4.3 Rotational Viscometer (RV) Test

A Brookfield viscometer was used to measure the viscosity of sealants at the installation temperature. The viscosity of the test group of sealants was measured at +180°C. This temperature was selected based on the recommended application temperatures in the manufacturer datasheets. Tests were conducted using spindle number 27 with a speed of 50 rpm. The number 27 spindle and spindle speed of 50 rpm were found to give more repeatable results (Al-Qadi et al. 2006). AASHTO standards T316 were followed for the other test procedures. Figure 4.7 shows the Brookfield viscometer and the number 27 spindle.

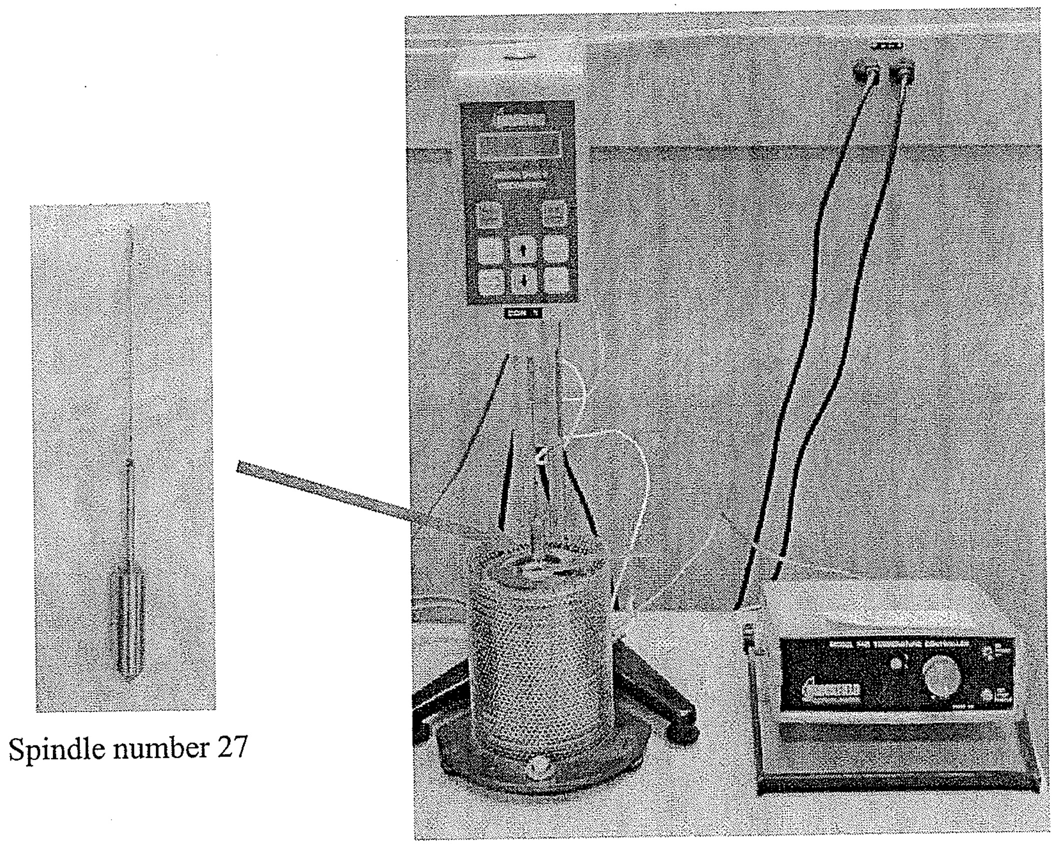


FIGURE 4.7: The Brookfield Viscometer

Table 4.2 shows the measured viscosity for the test sealants at +180°C. Form Table 4.2, sealants A and G have the highest and lowest viscosity (3.1 and 0.94 Pa.s), respectively. The viscosity of sealant A is more than three times the viscosity of sealant G. The tested sealants can be divided to three groups according to the clustering of their viscosity at the installation temperature:

- Sealants with low viscosity: sealants F, G, and H with viscosity ranging from 0.94 to 1.10 Pa.s

- Sealants with moderate viscosity: sealants B, C, D, and E with viscosity ranging from 1.53 to 1.97 Pa.s which represents 150% to 200% of the viscosity of the first group
- Sealants with high viscosity: sealant A with viscosity 3.1 Pa.s which represents 150% to 300% of the viscosity of the first and second groups.

TABLE 4.2: Viscosity at Installation Temperature (+180°C)

Sealant	Viscosity (Pa.s)
A	3.10
B	1.97
C	1.85
D	1.94
E	1.53
F	1.05
G	0.90
H	1.10

5 Creep Stiffness and Creep Rate at Low Temperatures

5.1 Introduction

Bending Beam Rheometer (BBR) test is originally developed for testing Performance Grades (PG) of asphalt binders. BBR test is used for evaluating low-temperature properties of binders based on creep stiffness (S) and creep rate (m-value). BBR test is adopted in this research to characterize the rheological properties of hot-pout bituminous sealants at low temperature. This chapter introduces testing procedures and results of the BBR test conducted on sealants (Figure 5.1). The correlation between BBR test results and field performance is discussed in chapter 7.

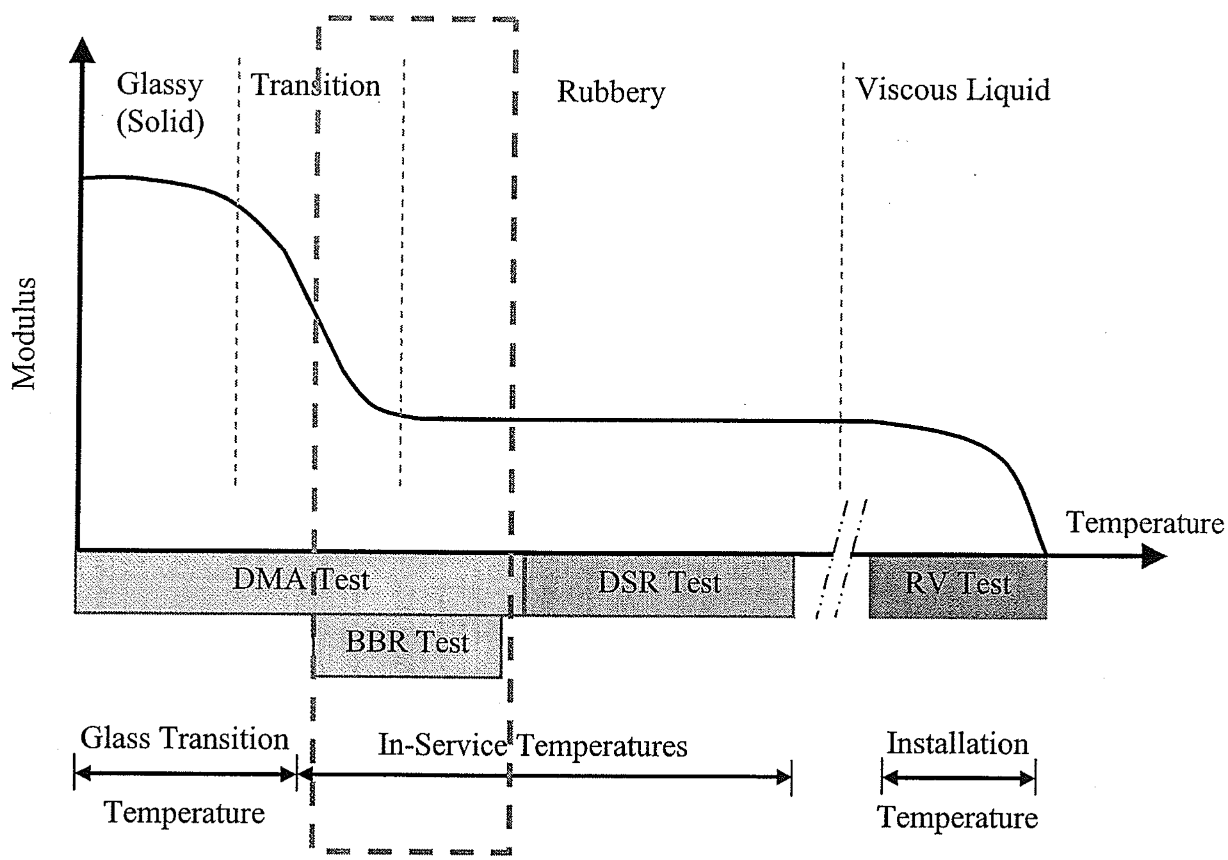


FIGURE 5.1: Evaluation of Sealants Using BBR Test

5.2 Tested Materials and specimen Preparation

The eight hot-pour sealants, involved in this study, were evaluated using BBR test. Representative samples were taken for each sealant, by taking three top-to-bottom slices from three locations in each sealant block. The middle parts of these three slices were combined and heated to the installation temperature recommended by the manufacturers, and then used for preparing the BBR test specimens. The heated sealant was poured in a rectangular aluminium mold. Mold sides and base were covered with wax paper. A mix of glycerine and talc was used as a releasing agent for the end pieces of the mold. The excess sealant material was removed using a hot knife to obtain a smooth and leveled surface. The final dimensions of the sealant specimen were 12.5 mm in width, 125 mm in length, and 6.25 mm in thickness. Before demolding the sealant specimen, the mold was placed in a freezer for 10 minutes. Figure 5.2 shows the BBR test specimen.

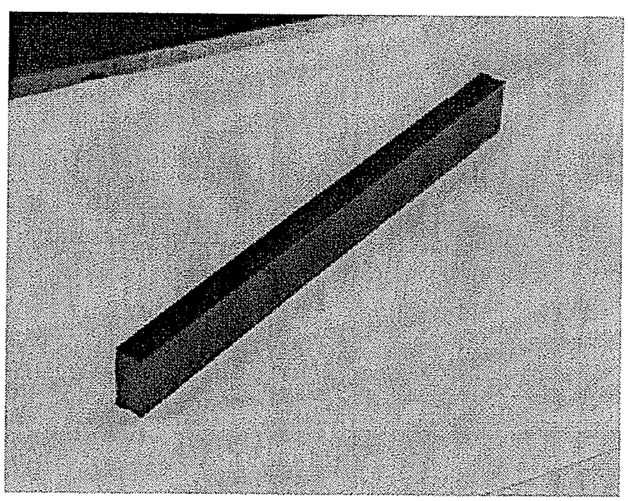


FIGURE 5.2: BBR Test Specimen

5.3 Test Setup

Canon instruments BBR apparatus is used in this study. The BBR apparatus was calibrated using a reference specimen before starting the actual tests. Alcohol cooling bath was used for controlling specimen temperature and reaching temperatures lower than 0°C. The BBR test was conducted at -30°C for all sealing materials. Sealant beams were conditioned in the BBR cooling bath for 60 minutes at -30°C prior to testing. Figures 5.3 and 5.4 show the BBR apparatus and the conditioning of sealant beams.

The test load is applied to the midpoint of the simply-supported sealant beam. The distance between beam supports is 102 mm (Figure 5.5). The sealant beam is subjected to a series of conditioning loads before applying the test load. First, sealant beam is subjected to a 30 mN (milliNewton) preload to ensure a firm contact between the beam and the supports. A seating load of 980 mN is then automatically applied by BBR software for one second. After this step, the load is reduced to the preload level for a recovery period of 20 seconds. The actual test load is applied after the 20 seconds recovery period.

The BBR apparatus is limited to a maximum applied load of 3900 mN and a maximum stroke of 5 mm. Due to the softness of tested sealants, the standard test load (980 mN) was replaced by a lower load level. A 500 mN load was used to test sealants A, B, C, and F while a 200 mN load was used to test sealants D, E, G, and H. These values were chosen based on pilot tests on each type of sealants with the aim to maintain a smooth time-deflection curve with a maximum deflection less than 5 mm and ensure the repeatability of the test results.

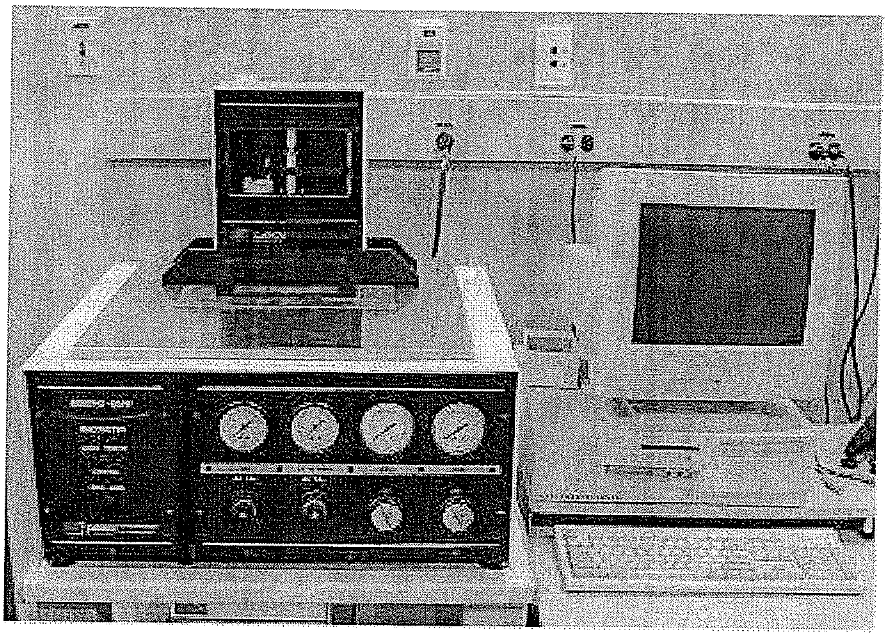


FIGURE 5.3: Canon Instruments BBR Apparatus

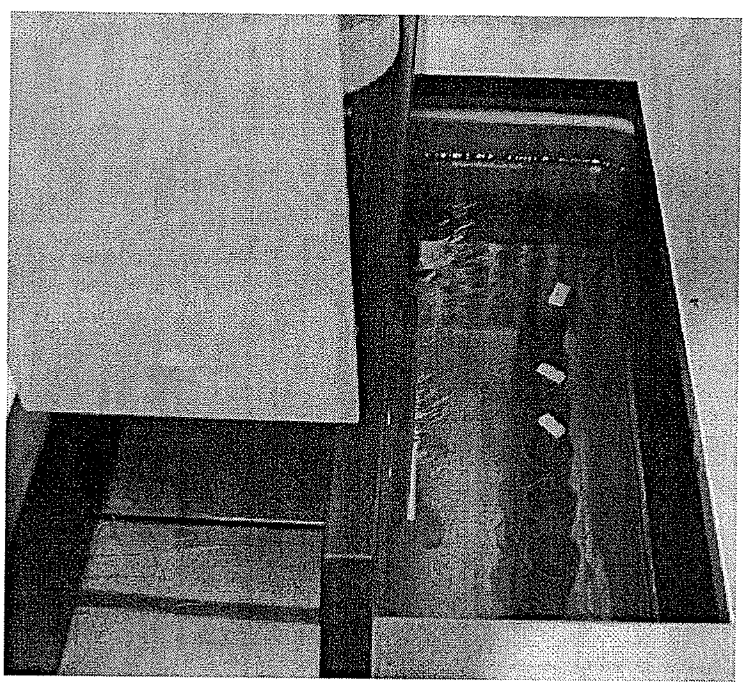
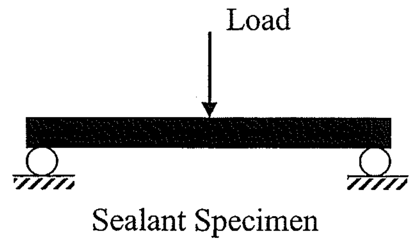


FIGURE 5.4: Conditioning of Sealant Beams

Sealant Specimen Dimensions (mm)	Load Amplitude	Test Temperature
12.5 in Width 6.25 in Thickness 125 in Length	500 mN (stiff sealants)	-30°C
	200 mN (soft sealants)	



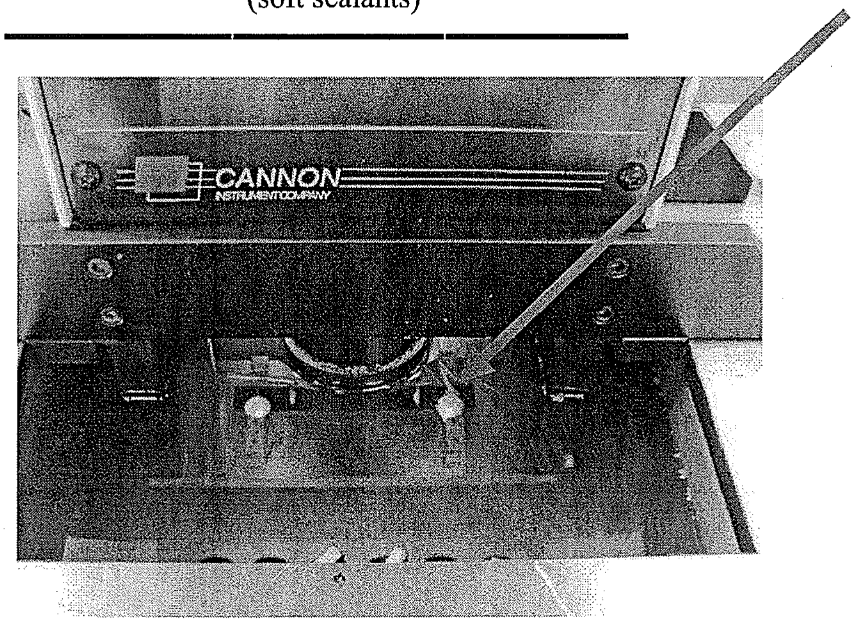
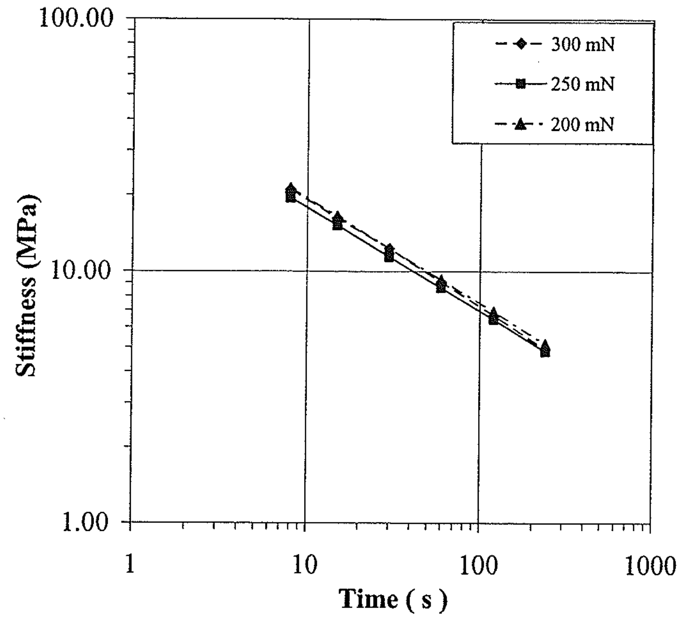
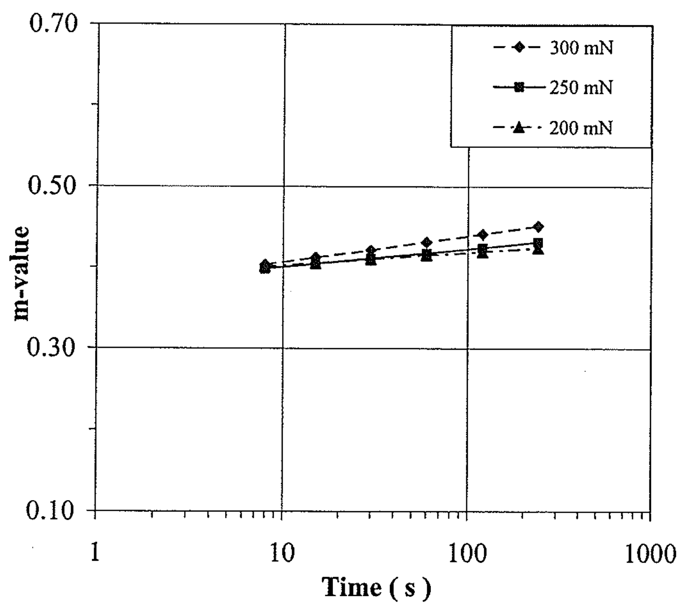


FIGURE 5.5: BBR Test Setup

The load dependency of sealants was checked by applying multiple load levels to test the same sealant. Figures 5.6 (a) and (b) show the creep stiffness (S) and m-value, respectively, for sealant G obtained from BBR test at three load levels (200 mN, 250 mN, and 300 mN). The 300 mN load represents 150% of the load level used in testing sealant G. Figure 5.6 (a) shows that the creep stiffness is independent of load level within this range of loading, and Figure 5.6 (b) indicates that m-value remained within the linear region for the load level used in the test.



a) Creep Stiffness at Three Load Levels



b) *m*-value at Three Load Levels

FIGURE 5.6: Creep Stiffness and *m*-value for Sealant G from BBR Test at Three Load Levels

5.4 Test Results

Four replicates were tested for each sealant at -30°C. The BBR software calculates creep stiffness (*S*) and creep rate (*m*-value) at different time steps from the measured deflections. Figures 5.7 to 5.14 show creep stiffness versus time on log scale for the tested group of sealants.

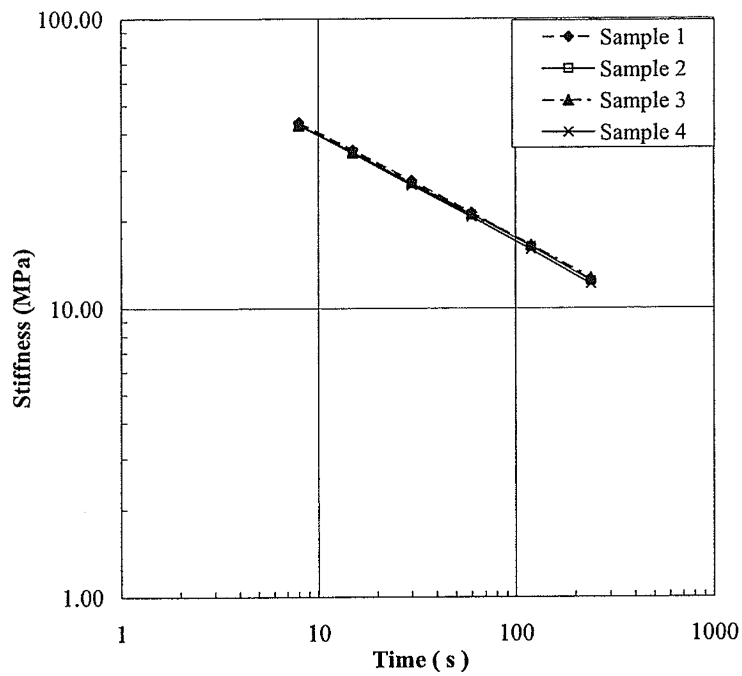


FIGURE 5.7: Creep Stiffness versus Time for Sealant (A)

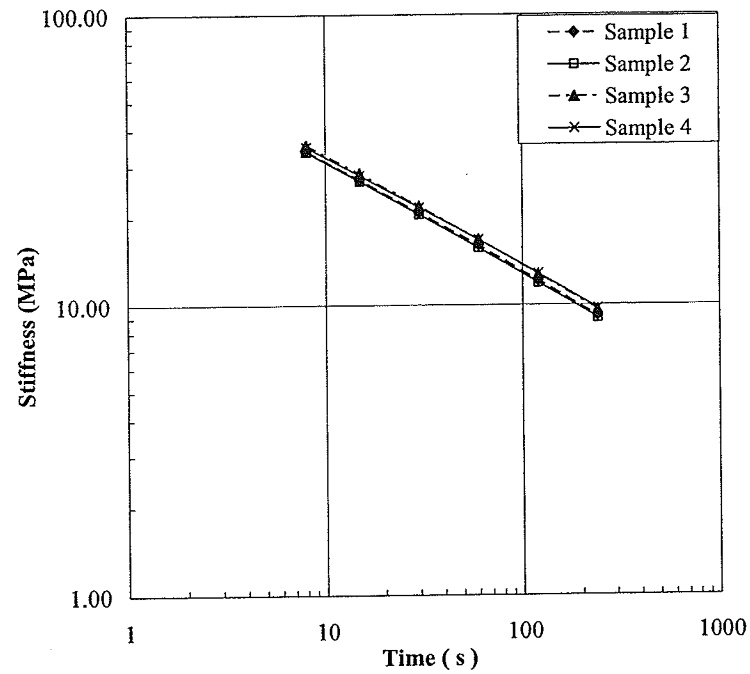


FIGURE 5.8: Creep Stiffness versus Time for Sealant (B)

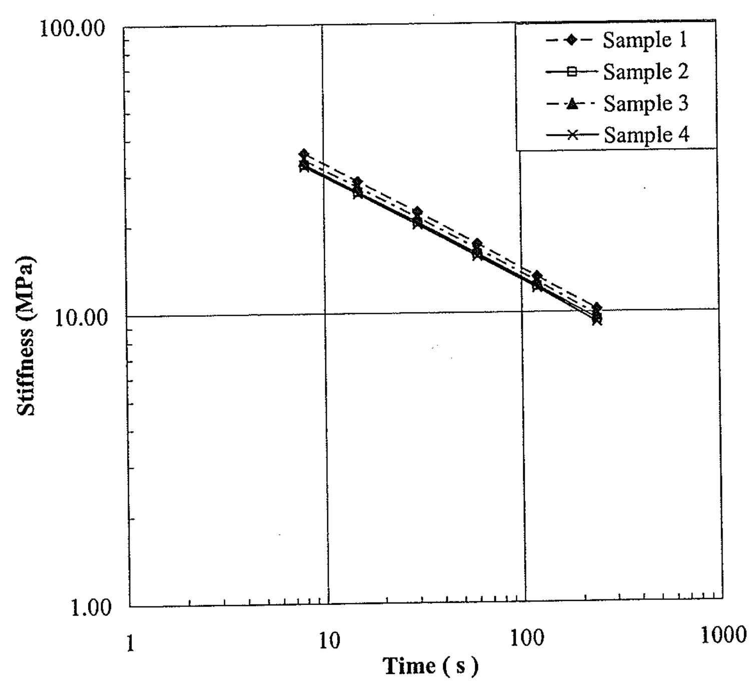


FIGURE 5.9: Creep Stiffness versus Time for Sealant (C)

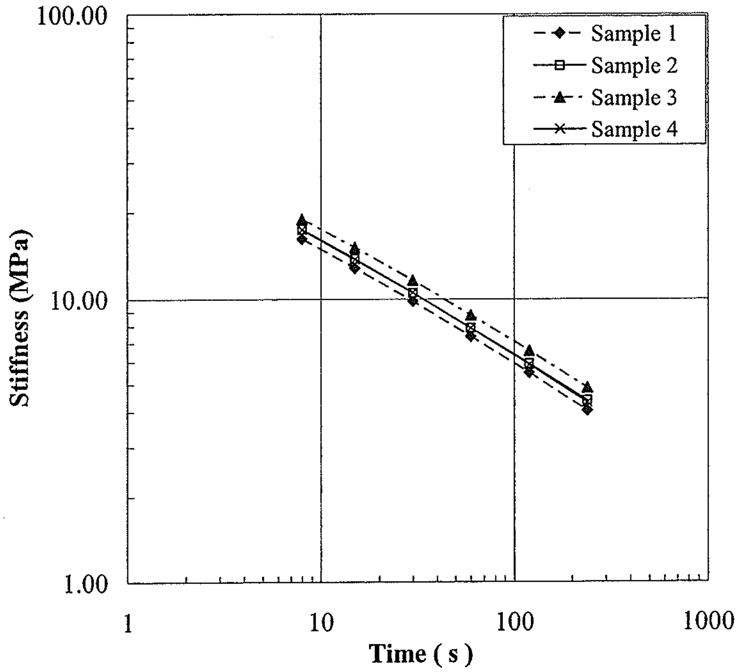


FIGURE 5.10: Creep Stiffness versus Time for Sealant (D)

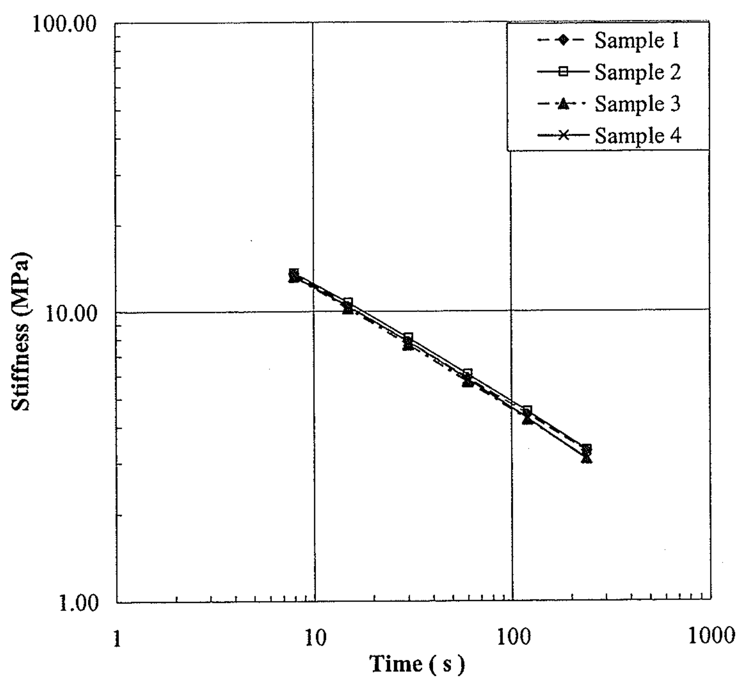


FIGURE 5.11: Creep Stiffness versus Time for Sealant (E)

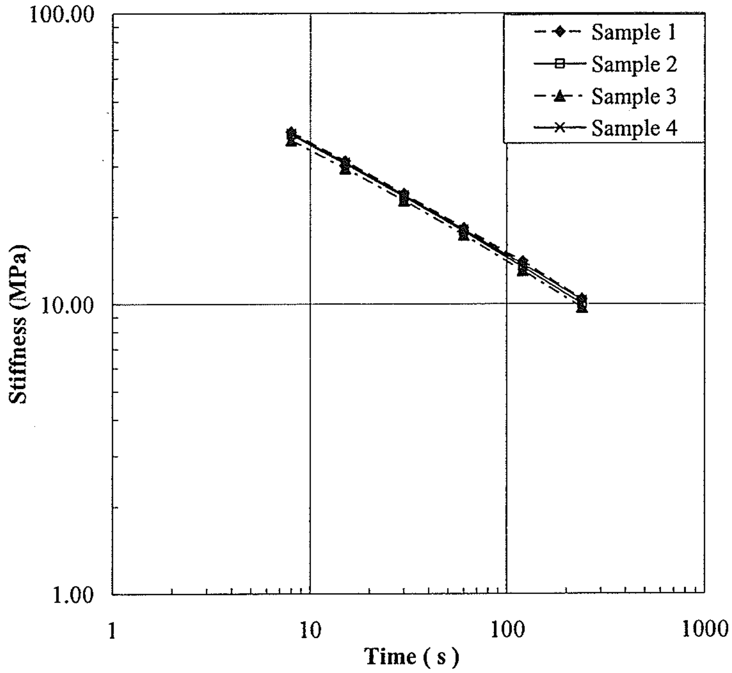


FIGURE 5.12: Creep Stiffness versus Time for Sealant (F)

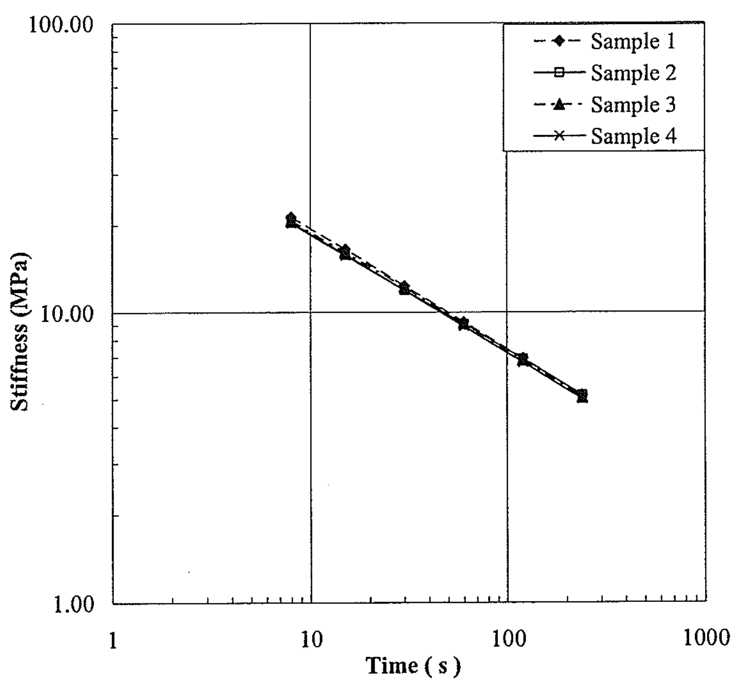


FIGURE 5.13: Creep Stiffness versus Time for Sealant (G)

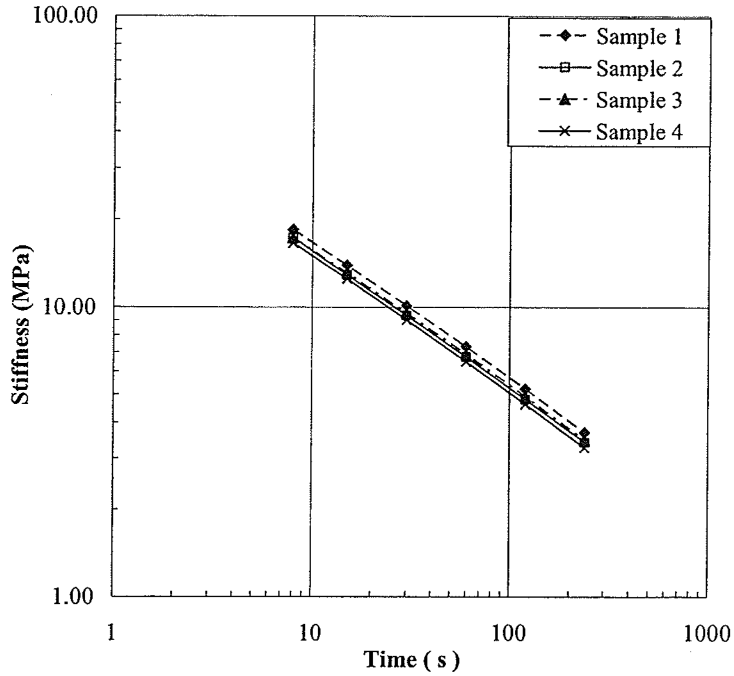


FIGURE 5.14: Creep Stiffness versus Time for Sealant (H)

5.5 Analysis of Results

The mean value was recorded for S and m at 60 seconds (S_{60} and m_{60}). Table 5.1 shows the average values of S_{60} and m_{60} and their coefficient of variation (COV) for each sealant. From Table 5.1, the COV for creep stiffness is less than 5% for all sealants except sealant D. The COV for m-value is less than 2% for all sealants.

To maintain adequate adhesion at low temperatures, sealants should not have high stiffness and should be able to dissipate the tensile stresses. These criteria can be evaluated by using S_{60} and m_{60} values from BBR test. Sealants with good performance should have low S_{60} and high m_{60} values, and vice versa.

Figure 5.15 compares the creep stiffness of all sealants. From Figure 5.14, the eight tested sealants are divided to two groups according to creep stiffness S_{60}. The first group of sealants (D, E, G, and H) has low creep stiffness, while the second group of sealants (A, B, C, and F) has much higher creep stiffness. A threshold value of S_{60} equals to 10 MPa can be used to distinguish between the two groups of sealants.

TABLE 5.1: Creep Stiffness and *m*-Value after 60 Seconds at -30°C

Sealant	S_{60} (MPa)		m_{60}	
	Mean Value	COV (%)	Mean Value	COV (%)
A	21.00	1.40	0.369	1.83
B	16.17	3.52	0.391	0.26
C	16.53	3.94	0.371	0.47
D	7.98	7.17	0.414	1.12
E	5.88	3.15	0.422	1.55
F	17.88	2.43	0.397	0.73
G	9.00	2.77	0.414	1.51
H	6.86	5.00	0.480	0.45

COV = Coefficient of variation

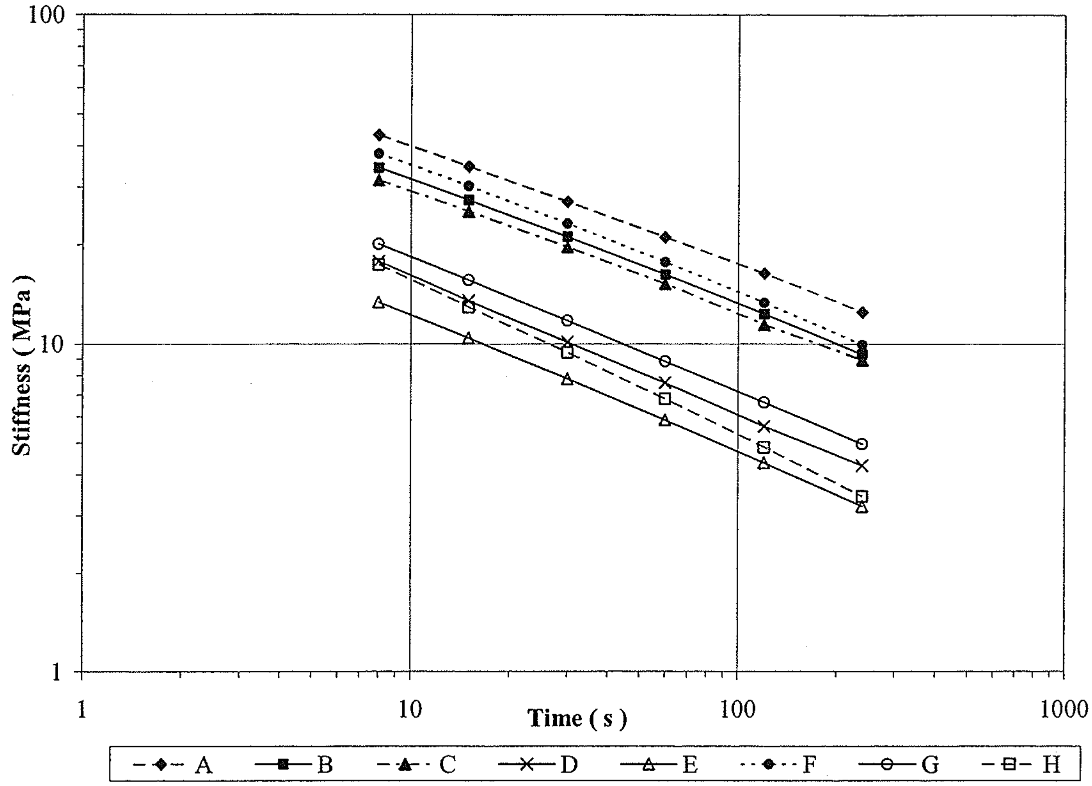


FIGURE 5.15: Creep Stiffness for the Tested Sealants Obtained from BBR Test

6 Glass Transition Temperature and Dynamic Modulus

6.1 Introduction

The glass transition temperature (Tg) of a sealant is an important criteria in predicting its field performance. If the glass transition temperature of a sealant is higher than the in-service temperature, the sealant material will become very stiff (in the glassy region) and an increase in sealant cohesion and adhesion failures may occur. The change of state from rubbery to solid state is associated with a distinct change in the coefficient of thermal expansion. Glass transition temperature can be considered as the temperature at which the change in the coefficient of thermal expansion occurs. Therefore, glass transition temperature of a material can be determined experimentally from the coefficient of thermal expansion test.

Glass transition temperature can also be determined from dynamic testing by measuring the complex modulus of the viscoelastic material at different temperatures. Dynamic mechanical analyzer (DMA) is a dynamic test that is used for characterizing the viscoelastic properties of polymers at temperatures ranging from -100°C to +100°C. DMA test is adopted in this research to evaluate the glass transition temperature and low-temperature stiffness of sealants.

The correlation between DMA test results and field performance is discussed in chapter 7.

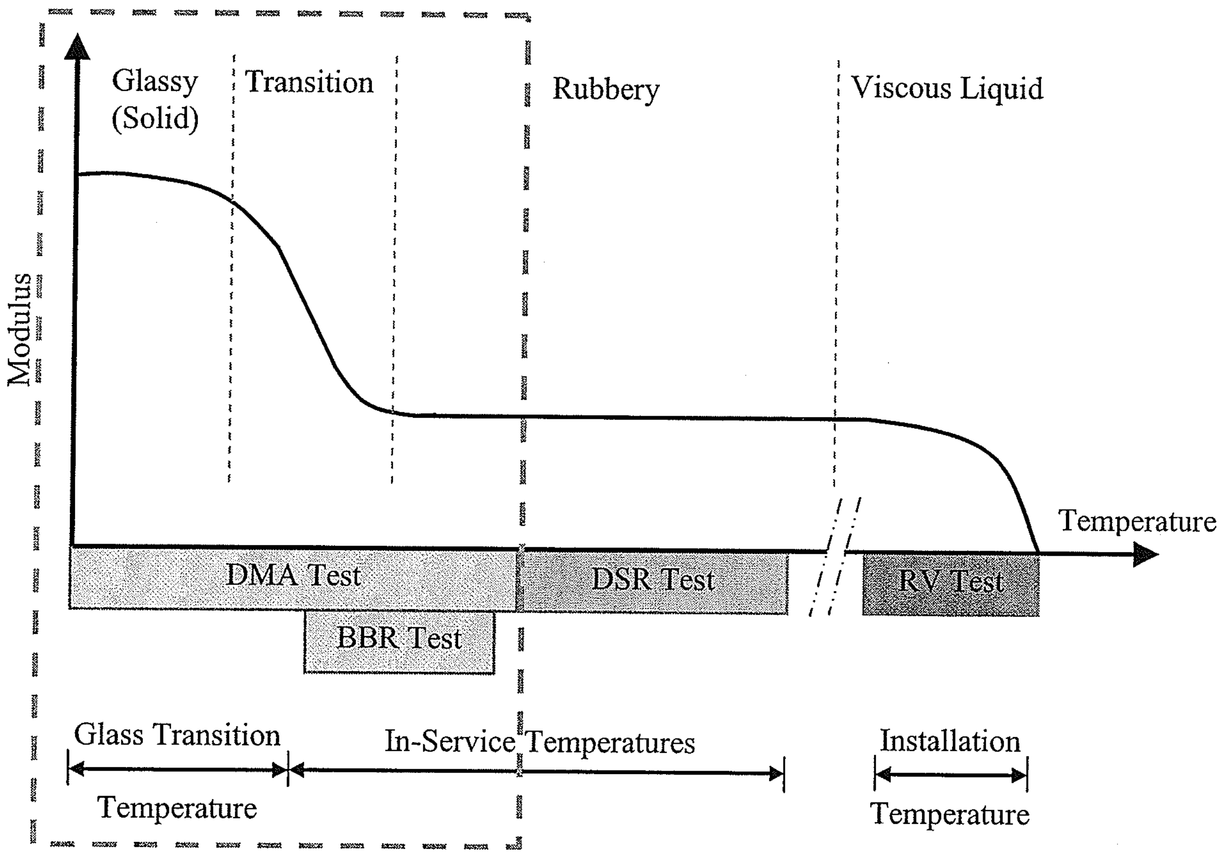


FIGURE 6.1: Evaluation of Sealants Using DMA Test

6.2 Tested Materials and specimen Preparation

A representative sample was taken from each sealant block and heated in an oil bath to the installation temperature recommended by the manufacturer. The dimensions of the sealant specimen were 10 mm in width, 60 mm in length, and 4 mm in thickness. Figure 6.2 shows the dimensions of the mold and Figure 6.3 shows the assembled mold before and after pouring the heated sealant. Wax paper and a mix of glycerine and talc powder were used for preventing sealant from bonding to the mold. The excess sealant material was removed by a hot knife to obtain a smooth and leveled top surface. Before demolding the sealant specimen, the mold was cooled to a temperature of -20°C to avoid tearing or deforming the specimen.

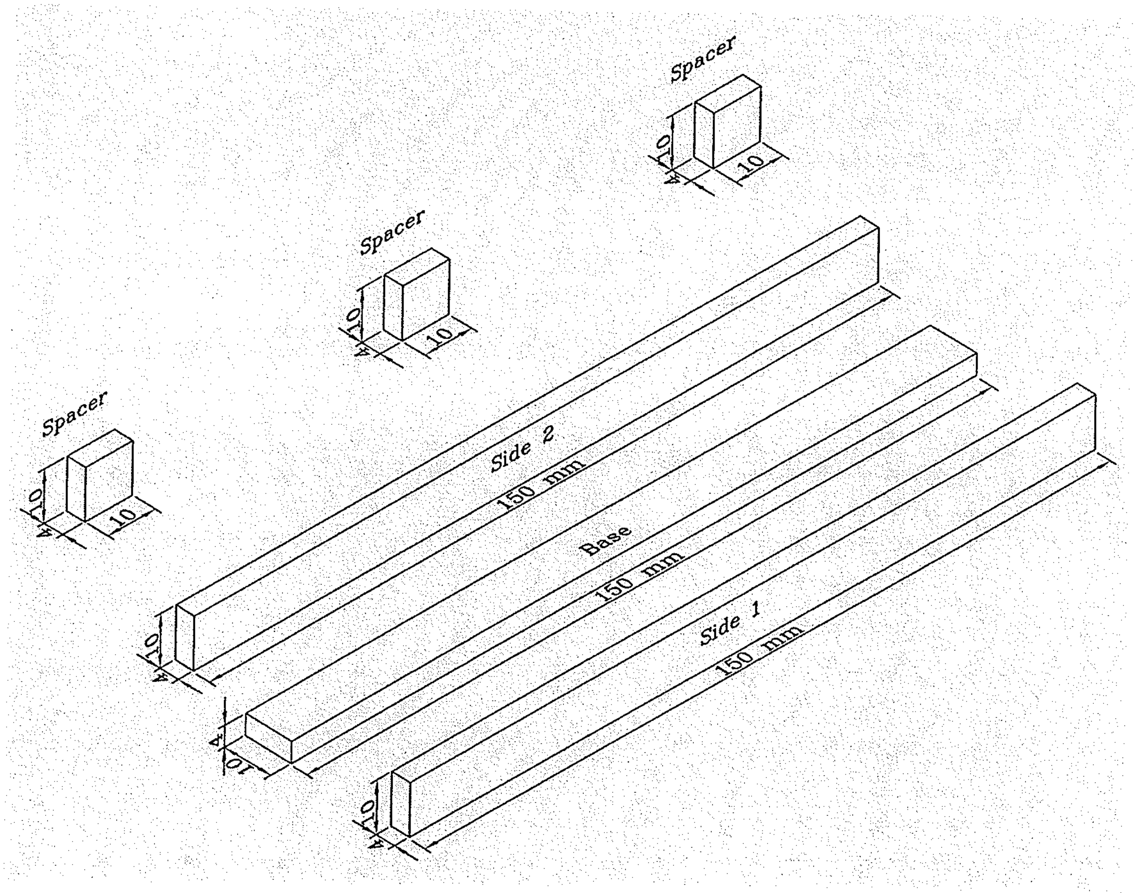
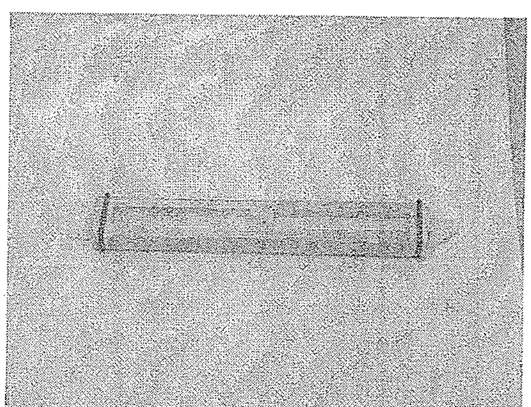
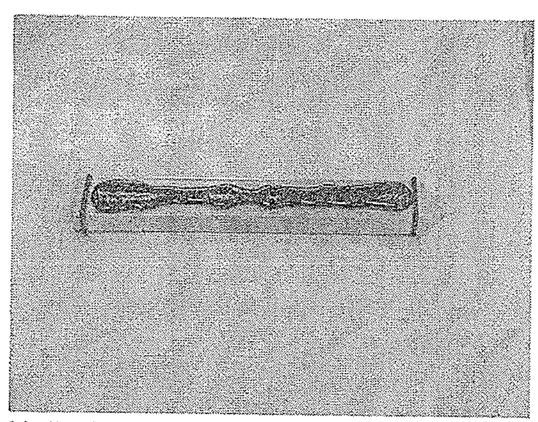


FIGURE 6.2: Dimensions of the Specimen Mold



a) The assembled mold before pouring sealant



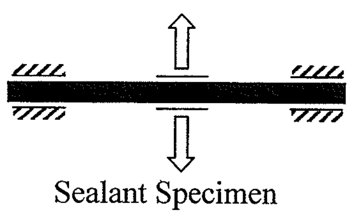
b) Sealants poured into the mold

FIGURE 6.3: Preparation of Sealant Specimen

6.3 Test Setup

The DMA test was conducted using a *TA instruments DMA 2980* apparatus and a dual cantilever loading clamp. Before testing, a calibration was conducted for the clamp mass and modulus using a standard metal specimen made from the same clamp material. Liquid nitrogen was used for cooling the specimen below room temperature. Figure 6.4 shows the calibration process of the clamp and the test setup.

Sealant specimens were subjected to a sinusoidal strain with amplitude of 5 μm and frequency of 1 Hz. The DMA test was conducted in the temperature-sweep mode with a temperature ranging from -70°C to 0°C and a ramp rate of 2°C/minute. Each specimen was conditioned first to a temperature of -50°C before mounting it to the clamp.



Sealant Specimen Dimensions (mm)	Strain Amplitude	Frequency	Test Temperature
10 in Width 4 in Thickness 60 in Length	5 μm	1 Hz	-70°C to 0°C

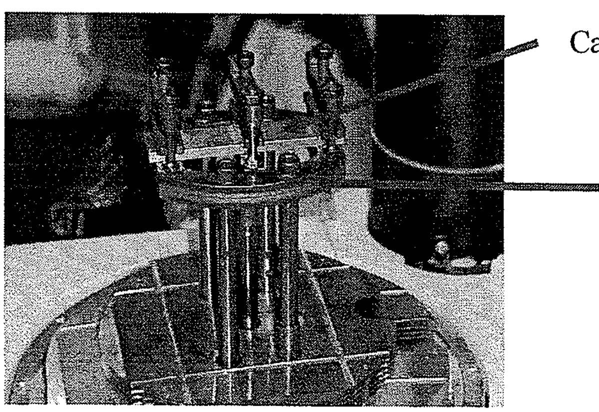


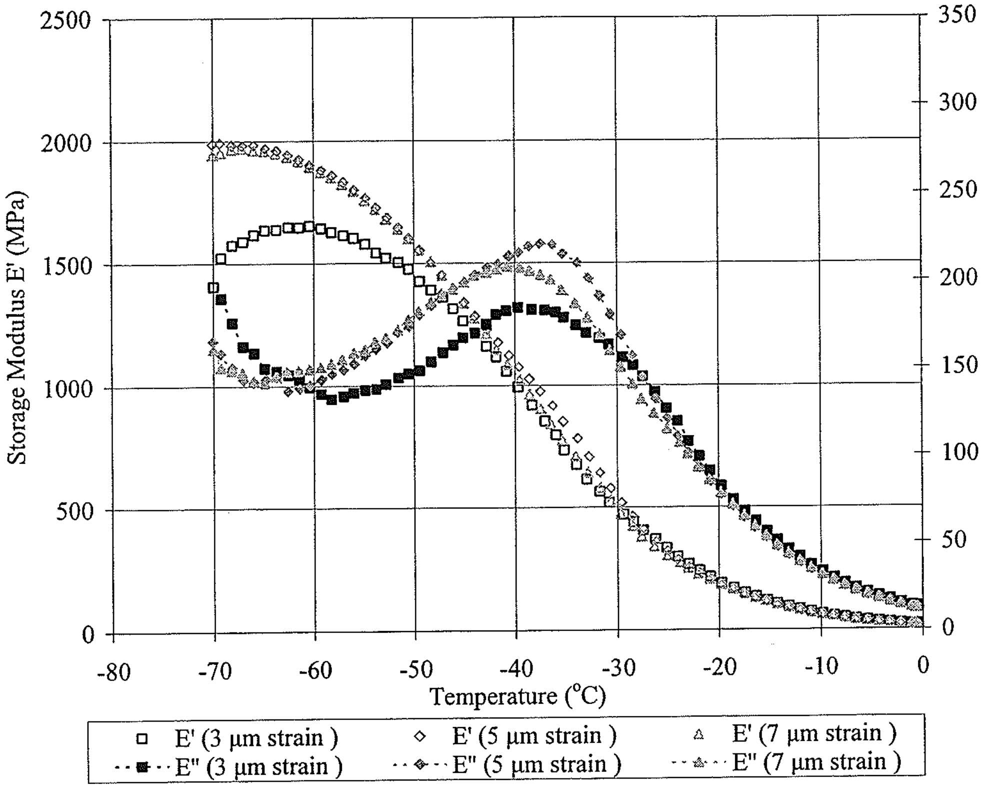
FIGURE 6.4: Dual Cantilever Clamp and Test Setup

A waiting time of 7 minutes was allowed for specimen temperature to equilibrate before starting the test. Two specimens were tested for each sealant and the results of both specimens were averaged.

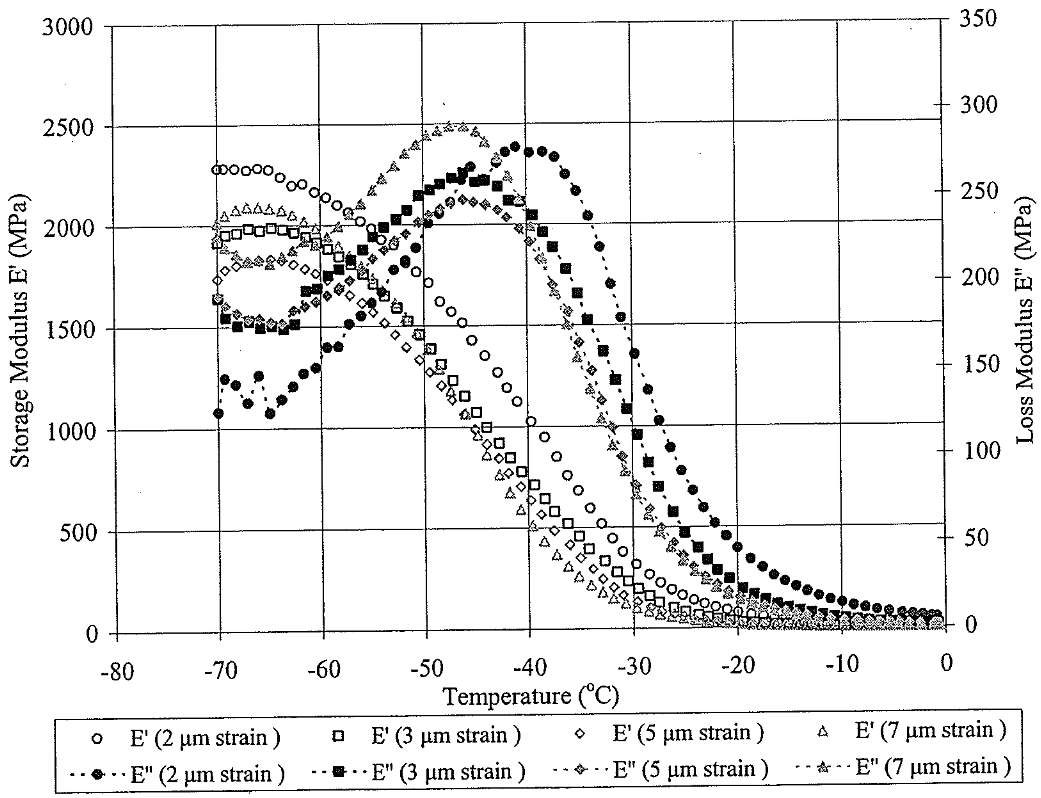
The criteria for setting the strain amplitude (5 μm) were based on ensuring that all materials remained in the linear viscoelastic zone throughout the tests. Sealant E (soft sealant) and Sealant A (stiff sealant) were tested at different strain amplitudes ranging from 2 μm to 10 μm. Results of these tests showed that the 5 μm strain amplitude is adequate for both stiff and soft sealants. Figures 6.5a and 6.5b show the storage modulus (E') and loss modulus (E") for sealant A and E, respectively, measured at different strain amplitudes; while Figures 6.6a and 6.6b show tan δ (E"/ E') for both sealants. Test results showed higher noise at strain amplitudes lower than 3 μm.

6.4 Test Results

Storage modulus (E'), loss modulus (E"), and tan δ (E"/ E') were recorded at temperatures ranging from -70°C to 0°C for each of the tested sealants. Data was collected at a rate of 1 reading per second. For each sealant, the results of the two tested specimens were averaged. Figures 6.7 to 6.13 show storage modulus, loss modulus, and tan δ for the tested sealants.



a) Sealant A



b) Sealant E

FIGURE 6.5: Storage and Loss Moduli at Different Strain Amplitudes

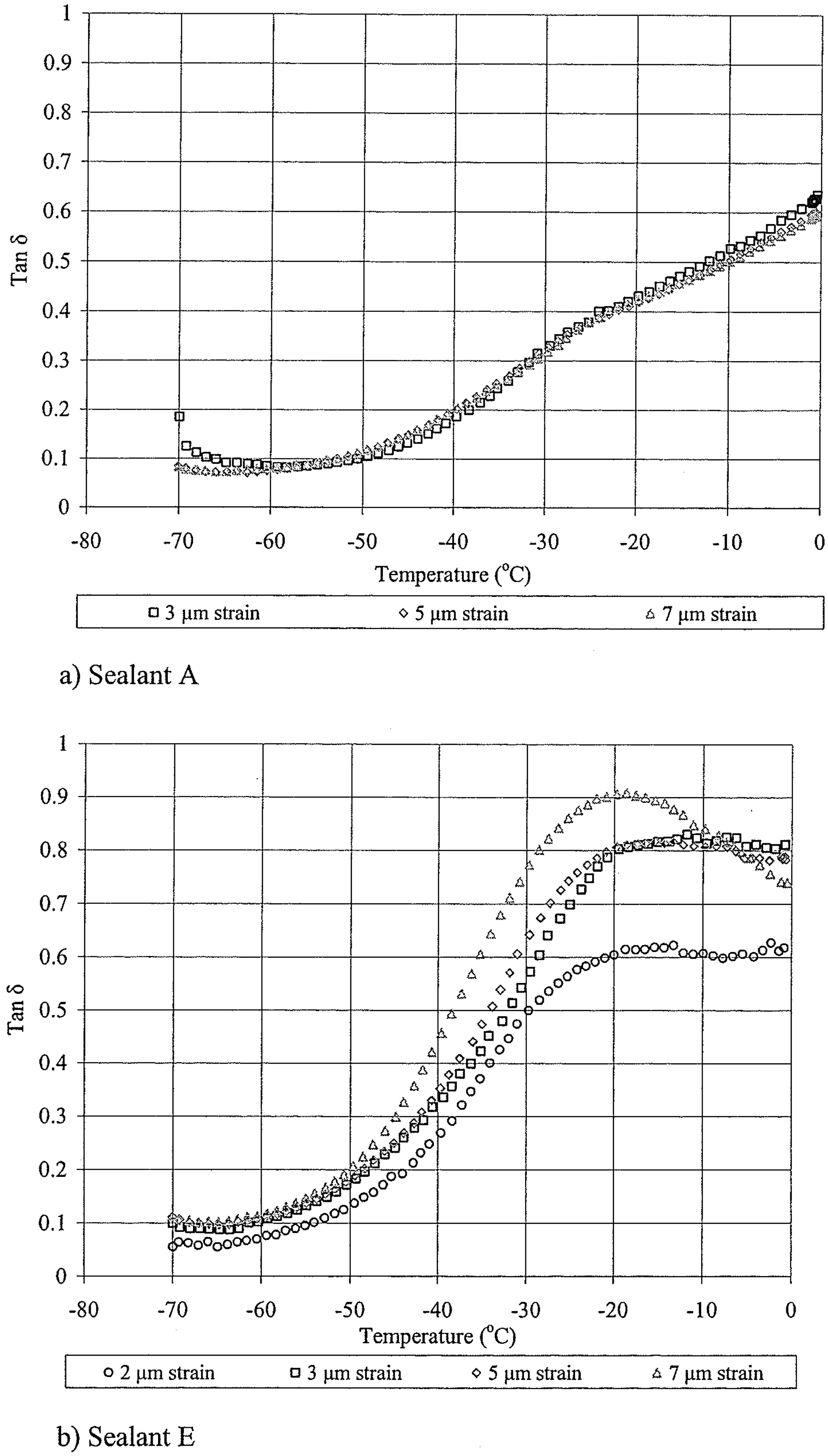
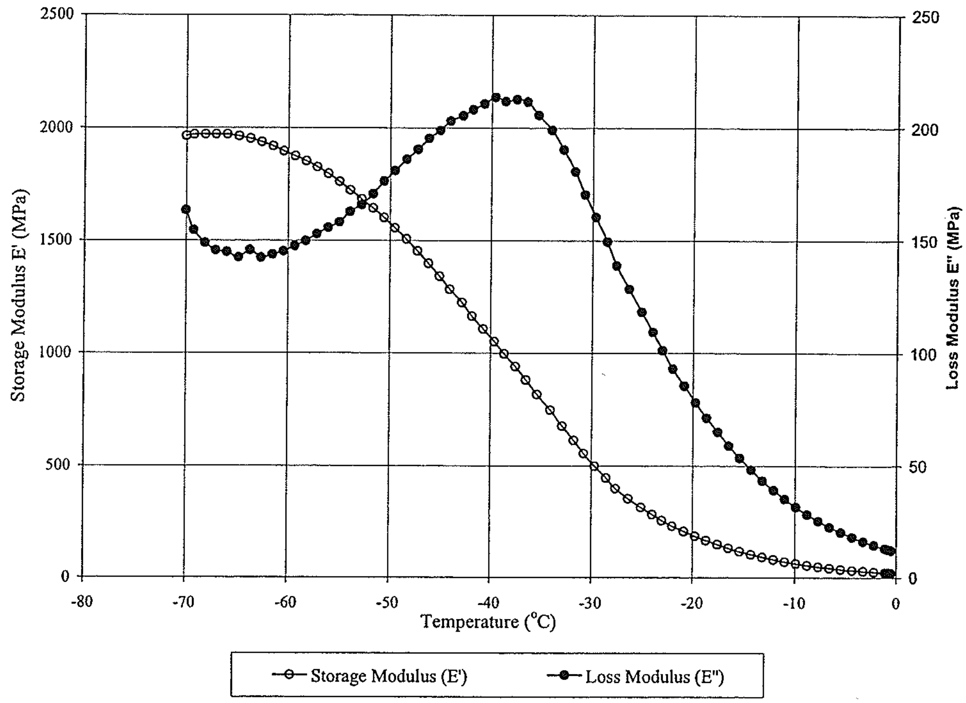
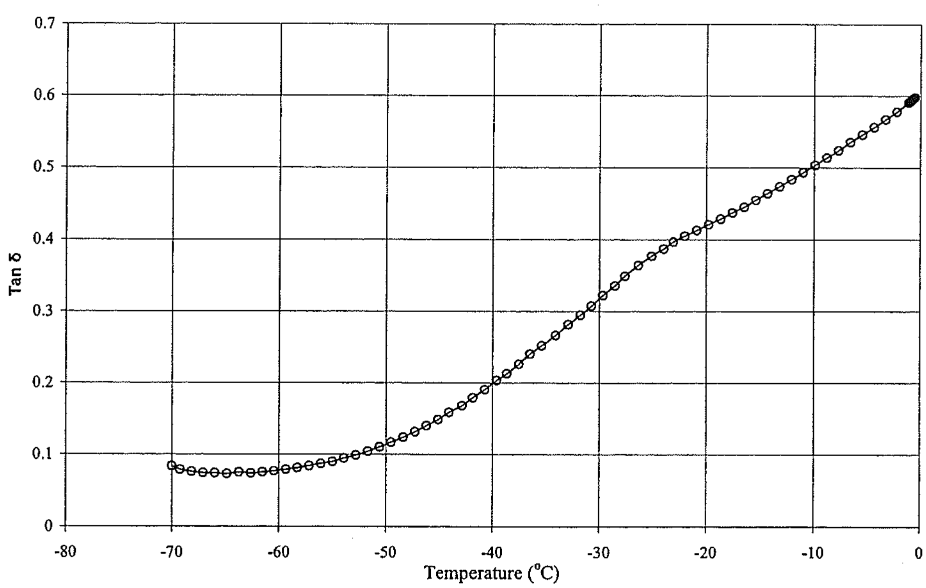


FIGURE 6.6: Tan δ at Different Strain Amplitudes

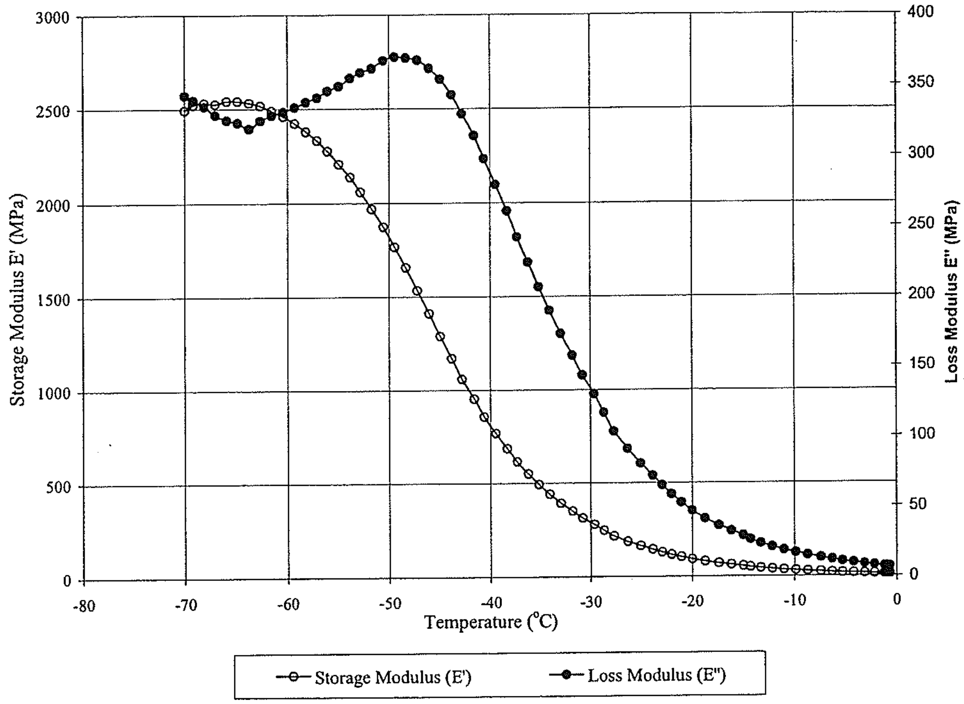


a) Storage and Loss Moduli for Sealant (A)

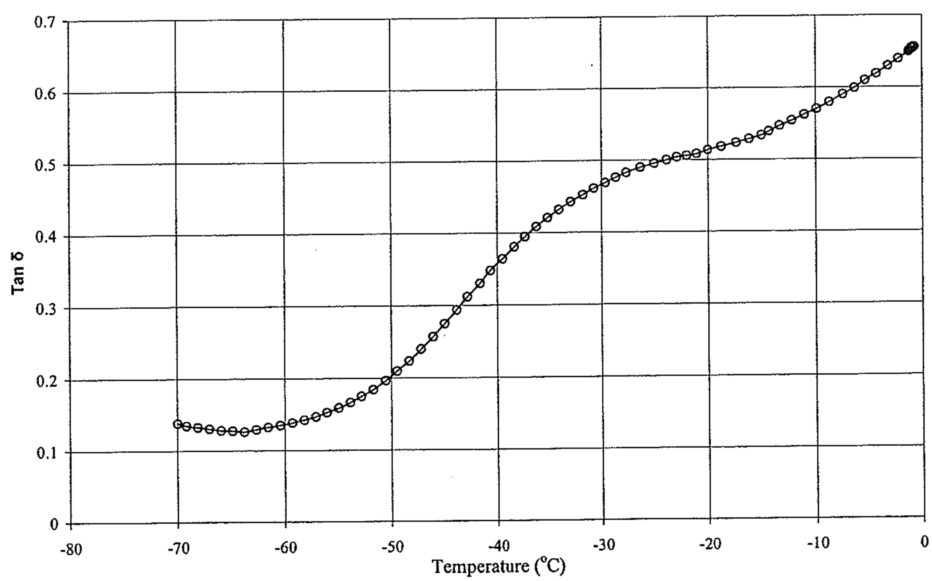


b) Tan δ for Sealant (A)

FIGURE 6.7: DMA Test Results for Sealant (A)

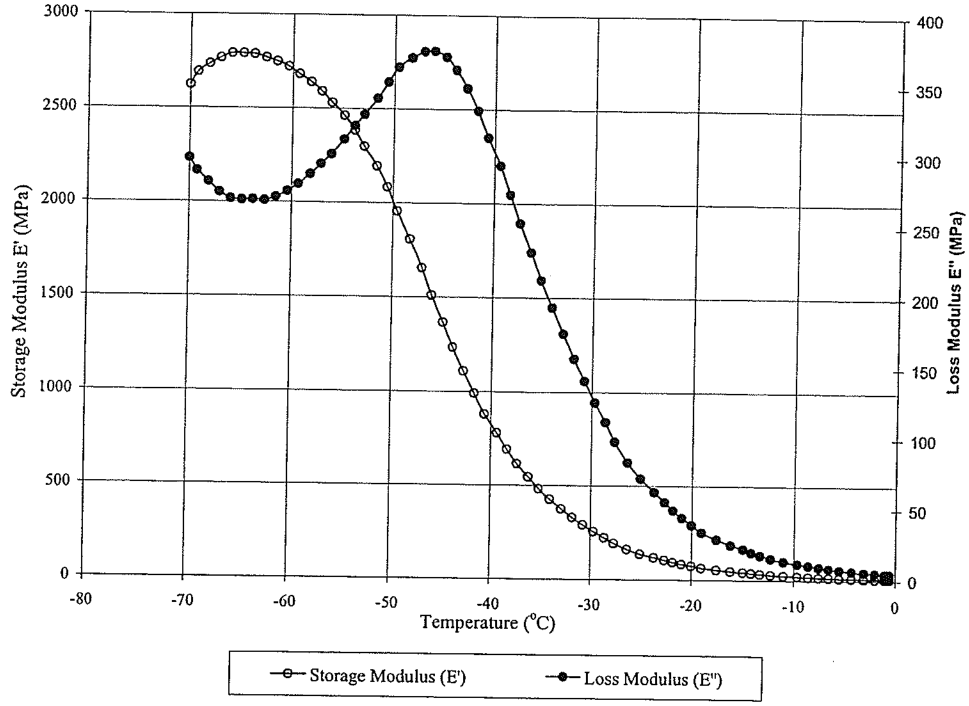


a) Storage and Loss Moduli for Sealant (B)

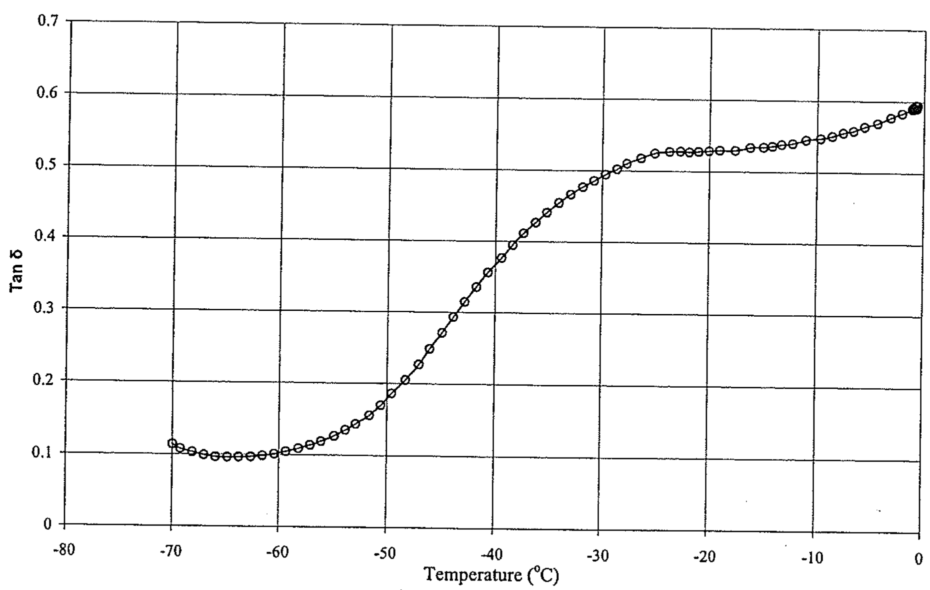


b) Tan δ for Sealant (B)

FIGURE 6.8: DMA Test Results for Sealant (B)

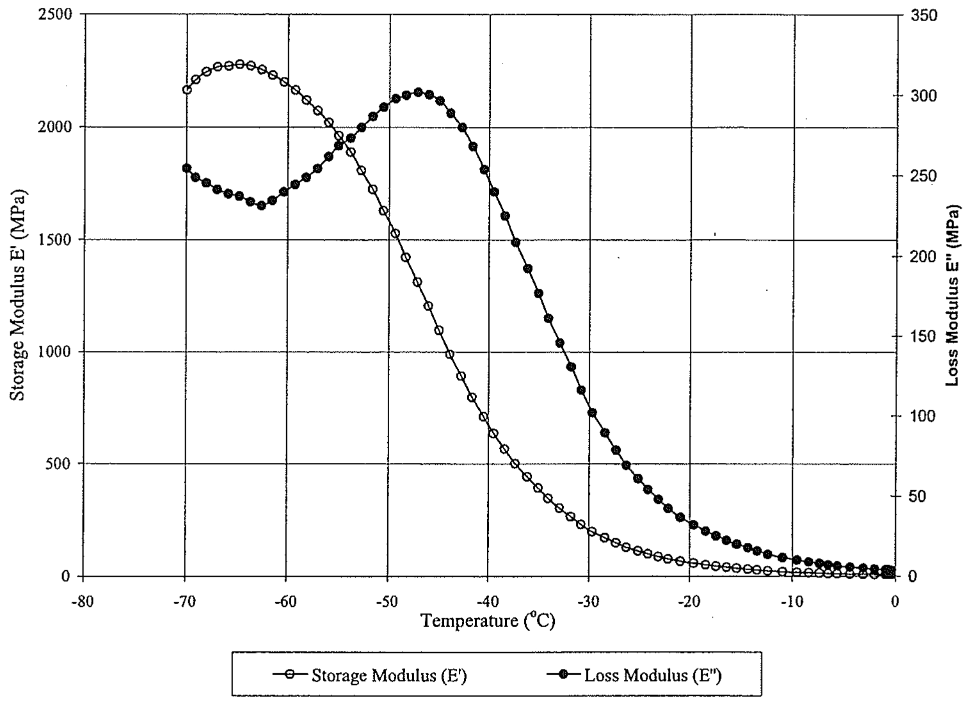


a) Storage and Loss Moduli for Sealant (C)

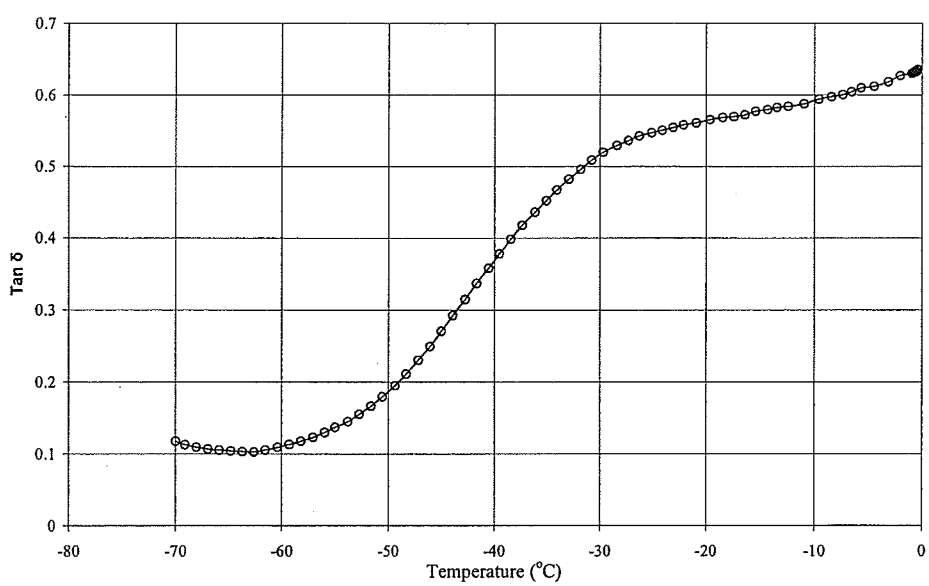


b) Tan δ for Sealant (C)

FIGURE 6.9: DMA Test Results for Sealant (C)

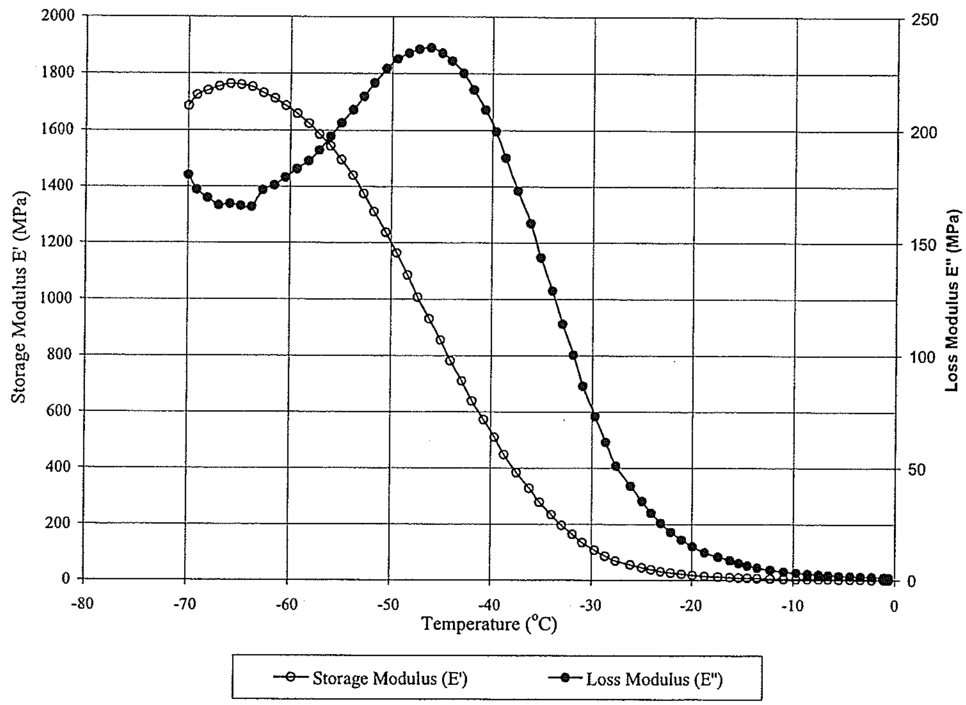


a) Storage and Loss Moduli for Sealant (D)

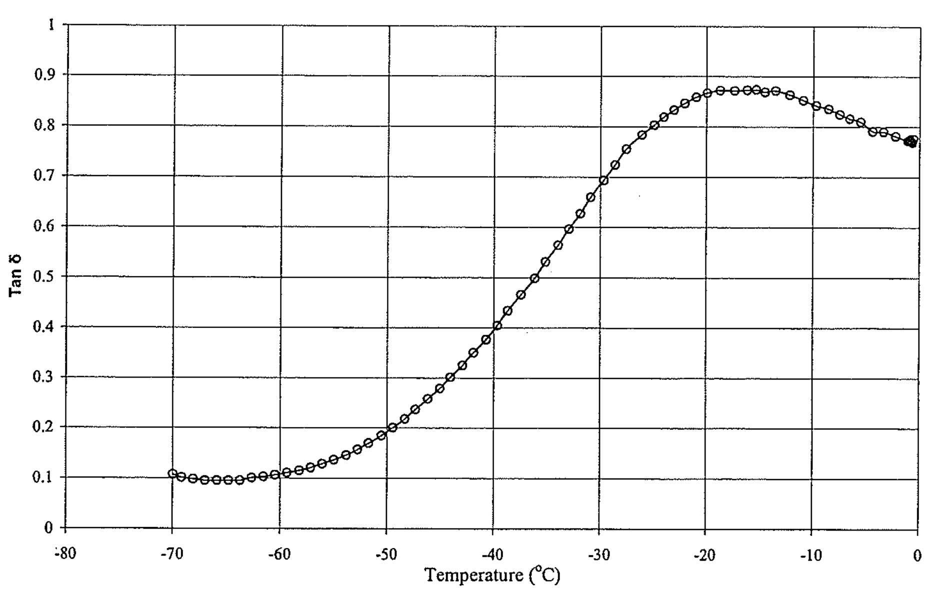


b) Tan δ for Sealant (D)

FIGURE 6.10: DMA Test Results for Sealant (D)

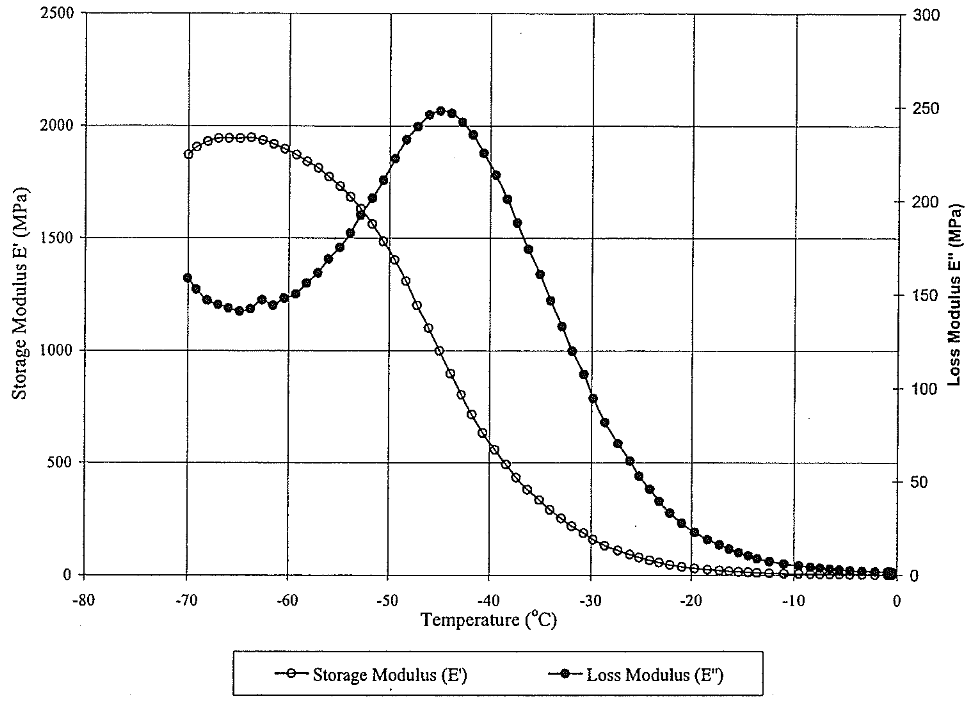


a) Storage and Loss Moduli for Sealant (E)

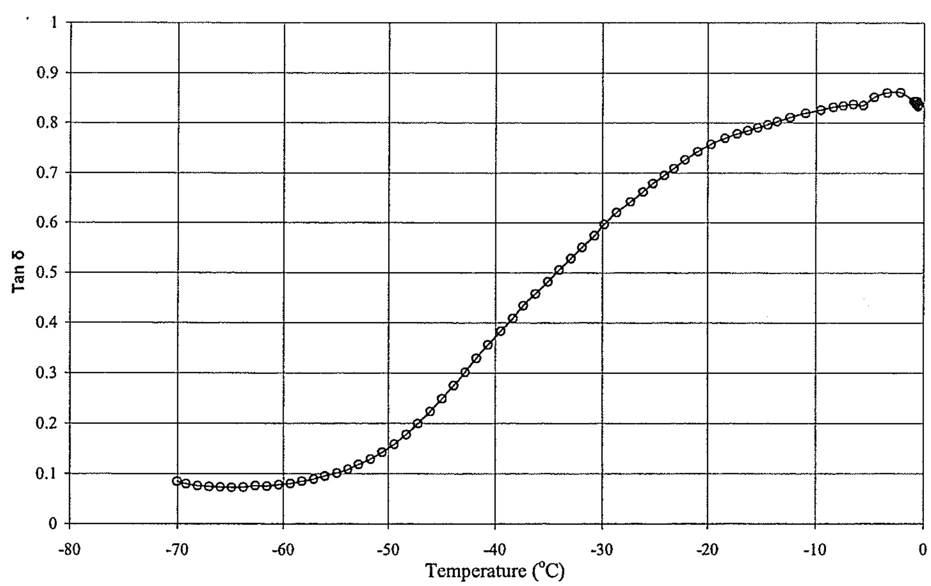


b) Tan δ for Sealant (E)

FIGURE 6.11: DMA Test Results for Sealant (E)

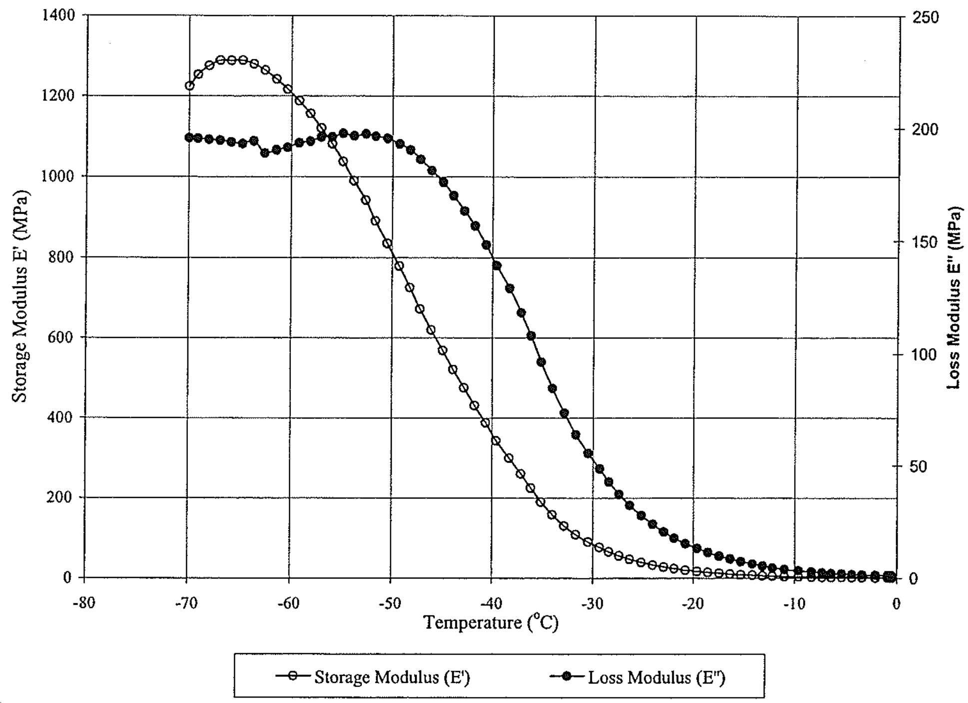


a) Storage and Loss Moduli for Sealant (G)

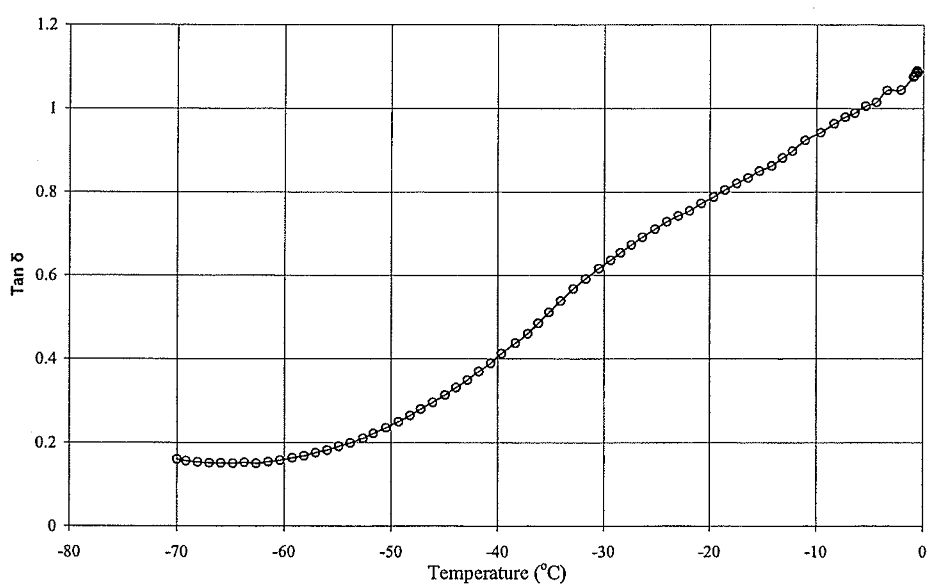


b) Tan δ for Sealant (G)

FIGURE 6.12: DMA Test Results for Sealant (G)



a) Storage and Loss Moduli for Sealant (H)



b) Tan δ for Sealant (H)

FIGURE 6.13: DMA Test Results for Sealant (H)

6.5 Analysis of Results

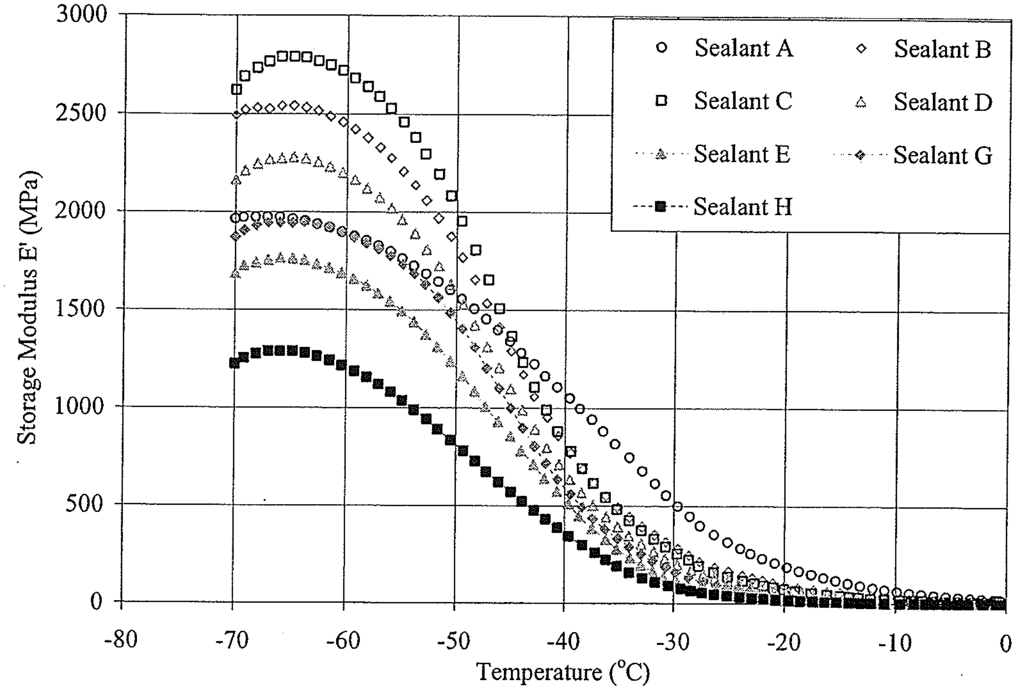
6.5.1 Storage Modulus (E')

Figure 6.14 shows the storage modulus for the tested sealants drawn on normal and logarithmic scales. Although the modulus values are low at temperatures ranging from -30°C to 0°C, the variability of the modulus spans multiple orders of magnitude. Sealants C and B showed the highest rates of the increase in modulus with the decrease of temperature while Sealant H had the lowest.

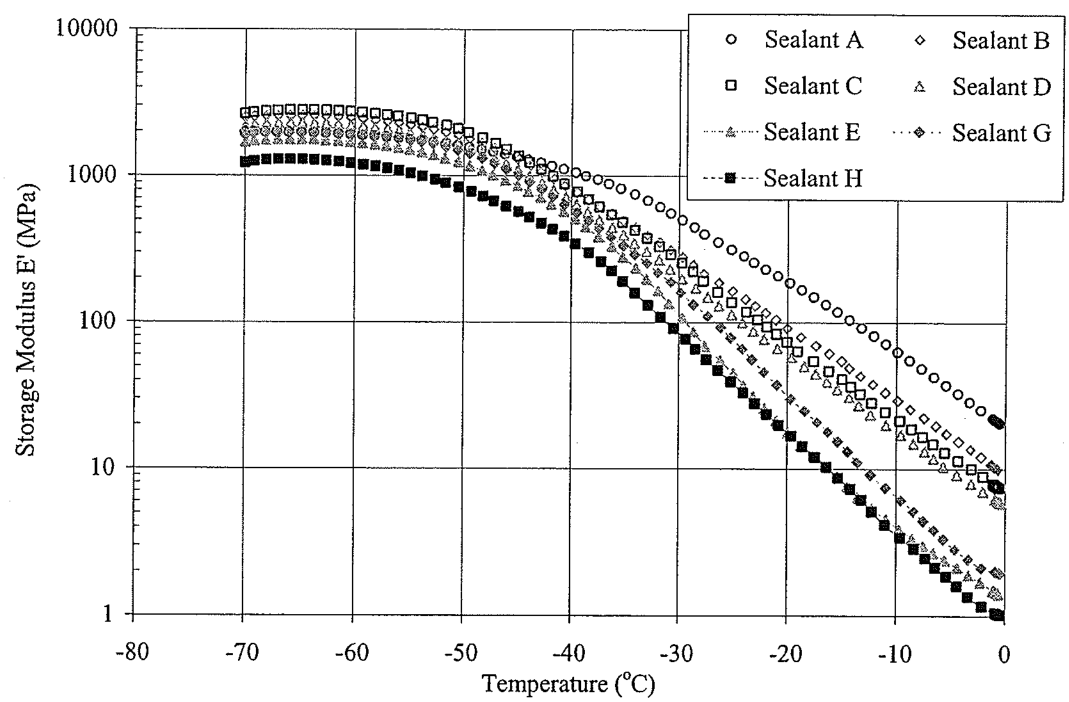
The storage modulus (E') was adopted as an indicator for low-temperature stiffness of sealants. For temperatures ranging from -40°C to 0°C, Sealants A and H have the highest and lowest storage modulus, respectively. Table 6.1 shows storage modulus at temperature -40°C for the tested sealants. The ability of a sealant to dissipate stresses is governed by the low-temperature stiffness (Marasteanu 2004). Therefore, Sealant H is expected to be more capable of dissipating stresses than Sealant A and to have better field performance at low temperatures.

TABLE 6.1: Storage Modulus at -40°C

Sealant	E' (MPa)
A	1075
B	810
C	830
D	670
E	595
G	540
H	365



a) Storage modulus (Normal scale)



b) Storage modulus (Logarithmic scale)

FIGURE 6.14: Storage Modulus for the Tested Sealants (Normal and Logarithmic Scales)

6.5.2 Loss Modulus (E")

The glass transition temperature (T_g) of a sealant can be estimated from the loss modulus-temperature curve, where it is the temperature corresponding to the peak of the loss modulus (Aklonis et al. 1972). Figure 6.15 shows the loss modulus for the tested sealants drawn on normal and logarithmic scales. Table 6.2 shows the glass transition temperature of the tested sealants estimated from the loss modulus-temperature curve. Sealants A and H have the highest and lowest glass transition temperature (-39.5 and -55°C), respectively, while the glass transition temperature for the remaining sealants ranged from -45°C to -48.5°C.

The glass transition temperature is an important factor in determining the compatibility of a sealant to certain climatic conditions. A sealant should not be expected to perform adequately in cold climates at temperatures close to or lower than its glass transition temperature. According to T_g values in Table 6.2, Sealant H is expected to have a better field performance in cold climates than other sealants.

Due to the complex composition of hot-pour bituminous sealants which are a blend of several viscoelastic materials that have different T_gs, the glass transition temperature obtained from DMA test is not considered an exact value. Sealants should be selected such that the glass transition temperature is lower than the minimum in-service temperature by a range of 10°C to 15°C.

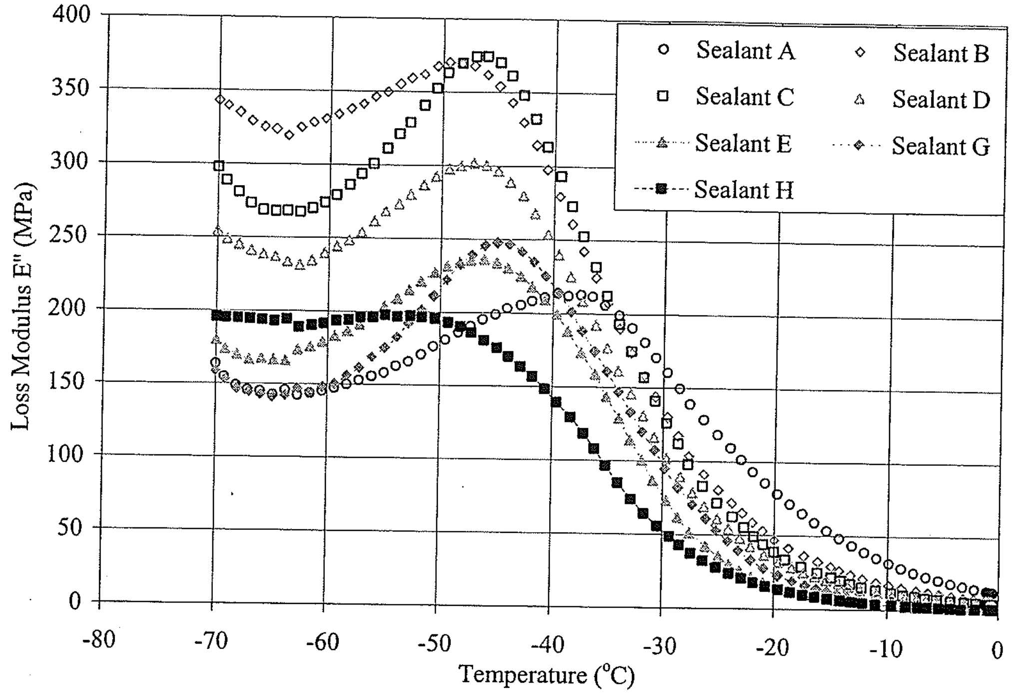
TABLE 6.2: Glass Transition Temperature (T_g)

Sealant	T_g (°C)
A	-39.5
B	-48.5
C	-46.0
D	-47.0
E	-46.0
G	-45.0
H	-55.0

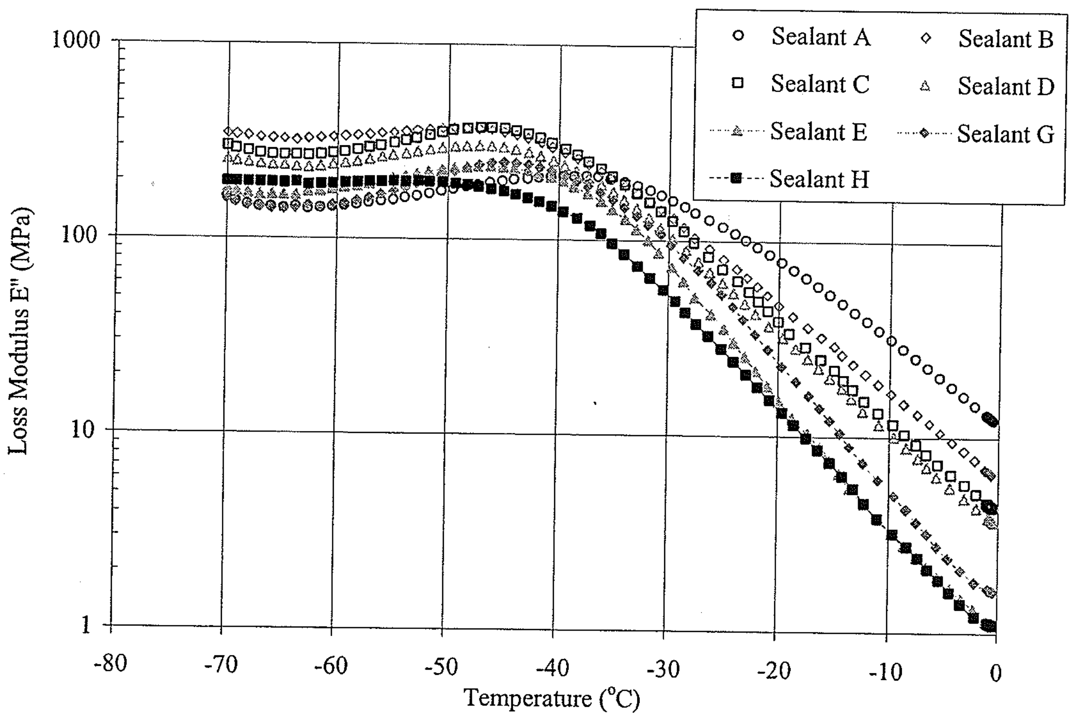
6.5.3 Phase Angle (δ)

Figure 6.16 shows the phase angles for the tested sealants calculated from tan δ (E"/E'). The glass transition temperature could not be estimated from tan δ-temperature curve for several sealants because no distinct peaks were detected. This condition can be attributed to the complex composition of hot-pour bituminous sealants which are a blend of several viscoelastic materials that have different T_gs.

The phase angle of sealant A at -40°C is 11°, while the phase angle for the remaining sealants ranged from 20° to 22°. Sealant A maintained a lower phase angle than other sealants with the increase of temperature. With the increase of temperature, the phase angles of sealants E, G, and H increase at a higher rate than the remaining sealants. The phase angle of sealant H is 47.5° at 0°C.



a) Loss modulus (Normal scale)



b) Loss modulus (Logarithmic scale)

FIGURE 6.15: Loss Modulus for the Tested Sealants (Normal and Logarithmic Scales)

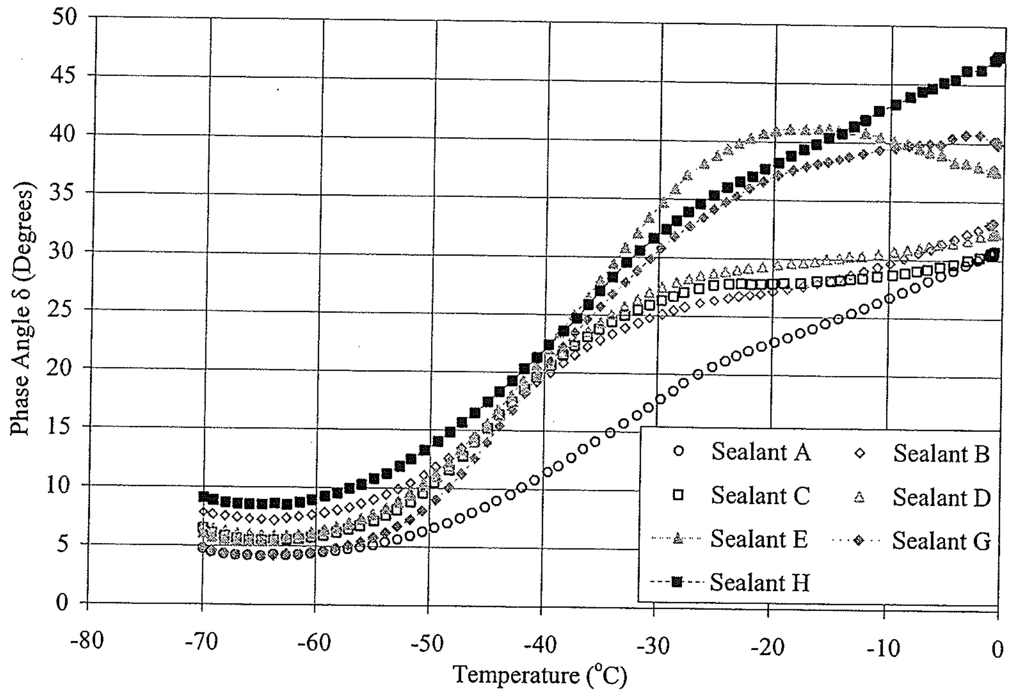


FIGURE 6.16: Phase angle (δ) for the Tested Sealants

7 Characterization of Sealants and Selection Indicators

7.1 Proposed Test Protocol for Full Characterization of Sealants

Sealants are subjected to a wide range of temperatures during installation and in-service. This range of temperatures could vary from +180°C, or higher, (average installation temperature of sealants) to -40°C, or less, (in-service temperature during winter). The field performance of sealants is affected by their rheological properties at installation and in-service temperatures (Masson 2000). The state of sealant material changes with temperature variation from a viscous liquid at high temperatures to an elastic solid at very cold temperatures. Therefore, more than one testing mode is required to evaluate the rheological properties of sealants at installation and in-service temperatures.

Figure 7.1 shows the proposed test protocol for full laboratory characterization of sealants performance. Four testing methods were adopted in this test protocol:

- Rotational Viscometer (RV): to characterize the rheological properties of sealants at installation temperature
- Dynamic Shear Rheometer (DSR): to characterize the rheological properties of sealants at moderate in-service temperatures.
- Bending Beam Rheometer (BBR): to characterize the rheological properties of sealants at low in-service temperatures (minimum in-service temperature).
- Dynamic Mechanical Analyzer (DMA): to characterize the rheological properties of sealants at low in-service temperatures.

The major advantage of using these testing methods is that the equipment is readily available in asphalt binder and polymer characterization laboratories and the procedures are familiar to laboratory technicians. Table 7.1 shows a comparison of testing procedures for RV, DSR, BBR, and DMA tests.

7.2 Laboratory-Based Performance Indicators for Sealants

7.2.1 Viscosity at Installation Temperature

Viscosity of sealants at installation temperature is one of the factors that affect the adhesion strength between sealants and pavements (Masson and Lacasse 2000). Adsorption and mechanical interlock theories are two examples of the mechanisms that explain the adhesion between bituminous sealants and asphalt concrete pavements. In adsorption theory, the adhesion strength is affected by the ability of the sealant to spread over the surface of the pavement joint (crack). In mechanical interlock mechanism, the interlocking is generated by the filling of micro-voids of the pavement joint (crack) with the sealant. In both mechanisms, the adhesion strength is strongly affected by the viscosity of the sealant at installation temperature.

Figure 7.2 shows the viscosity of the eight sealants at +180°C measured by Rotational Viscometer. Sealants A and G have the highest and lowest viscosity (3.1 and 0.94 Pa.s), respectively. The viscosity of sealant A is more than three times the viscosity of sealant G. The tested sealants can be divided to three groups according to the clustering of their viscosity at the installation temperature:

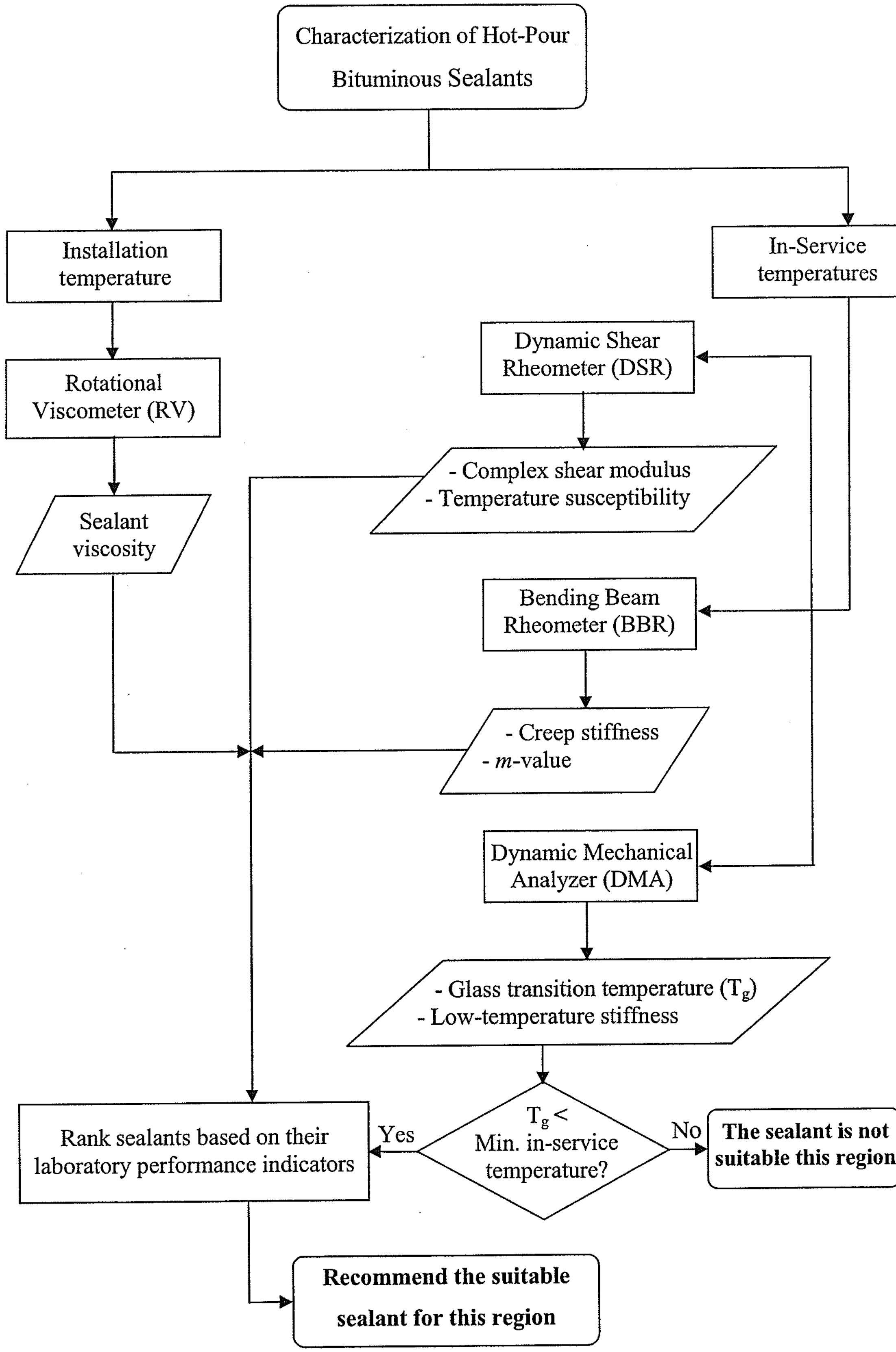
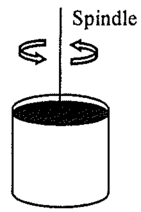
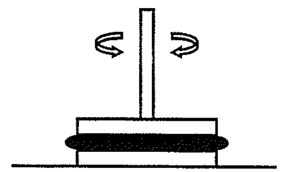
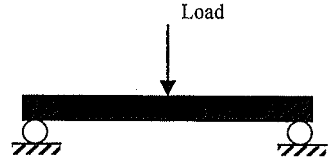
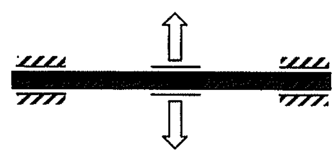


FIGURE 7.1: Proposed Test Protocol for Full Characterization of Sealants

TABLE 7.1: Comparison of Testing Procedures for RV, DSR, BBR, and DMA Tests

Test	Specimen Shape	Specimen Dimensions (mm)	Test Setup	Loading Mode	Load / Strain Amplitude	Frequency	Test Temperature
RV	Liquid	N/A [a]		Constant shear strain	Spindle speed 50 rpm	N/A [a]	+180°C
DSR	Circular disc	25 in Diameter 1 in Thickness		Oscillating shear strain	2%	1.5 Hz	+5°C to +40°C
					4%	1.5 Hz	+46°C to 64°C
BBR	Rectangular beam	12.5 in Width 6.25 in Thickness 125 in Length		Creep under constant load	500 mN (stiff sealants) 200 mN (soft sealants)	N/A [a]	-30°C
DMA	Rectangular beam	10 in Width 4 in Thickness 60 in Length		Oscillating bending strain	5 μm	1 Hz	-70°C to 0°C

[a] N/A = Not Applicable

- Sealants with low viscosity: sealants F, G, and H with viscosity ranging from 0.94 to 1.10 Pa.s
- Sealants with moderate viscosity: sealants B, C, D, and E with viscosity ranging from 1.53 to 1.97 Pa.s which represents 150% to 200% of the viscosity of the first group
- Sealants with high viscosity: sealant A with viscosity 3.1 Pa.s which represents 150% to 300% of the viscosity of the first and second groups.

Based on viscosity only and neglecting the effect of other factors, the first group of sealants (F, G, and H) is expected to have better adhesion to pavement than the other two groups.

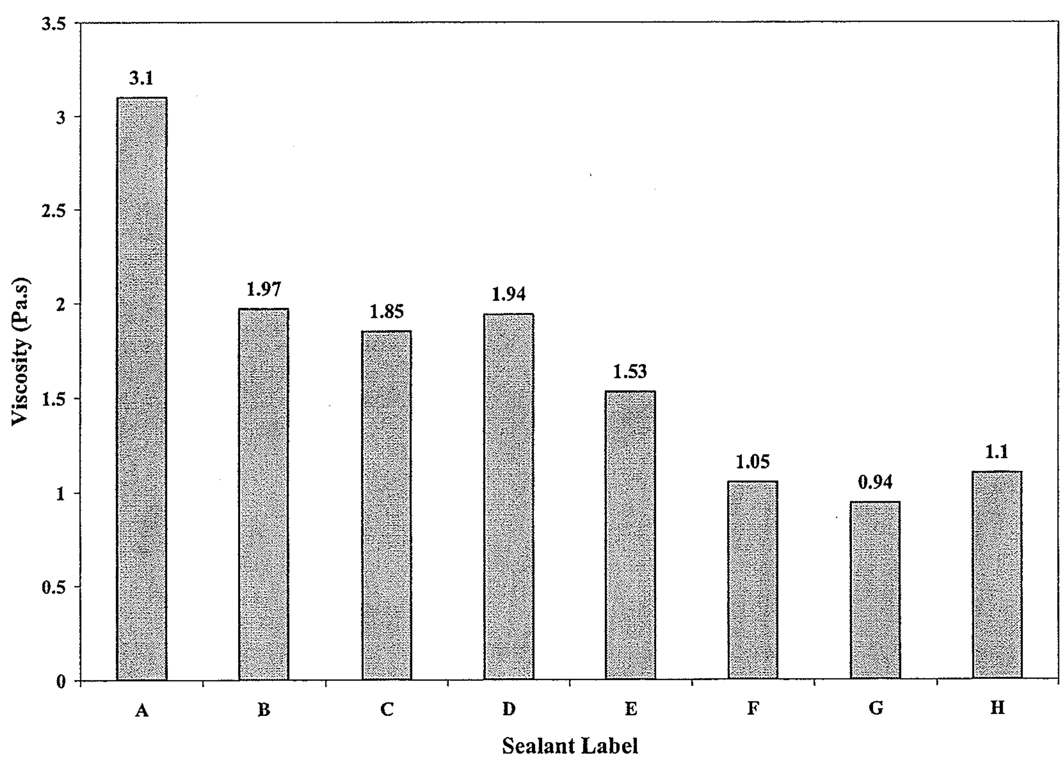


FIGURE 7.2: The Viscosity of the Tested Group of Sealants at +180°C

7.2.2 Creep Stiffness and *m*-value

Adhesion failure of sealant can be due to high stiffness of sealant and the sealant's inability to dissipate the tensile stresses generated at low temperature which lead to build-up of tensile stresses at the sealant-pavement interface. These properties are related to the fundamental rheological properties of sealants at low temperatures which can be evaluated by the BBR test (Masson 2000). The BBR test is used for determining creep stiffness and rate of change in creep stiffness (*m*-value). Creep stiffness and *m*-value are correlated to the ability of asphalt binders (bituminous material) to dissipate thermal stress (Marasteanu 2004).

Creep stiffness (S) and *m*-value were measured at -30°C. It is assumed that S at temperature T after two hours equal to S at temperature (T+10) after 60 seconds, according to the time-temperature superposition principle (Asphalt Institute 1995). Therefore, S_{60} and m_{60} can be taken to represent creep stiffness and *m*-value after two hours of loading at -40°C. Figures 7.3 and 7.4 show S_{60} and m_{60}, respectively, for the tested group of sealants. The eight tested sealants are divided into two groups according to creep stiffness S_{60}. The first group of sealants (D, E, G, and H) has low creep stiffness, while the second group of sealants (A, B, C, and F) has much higher creep stiffness. A threshold value of S60 equals to 10 MPa can be used to distinguish between the two groups of sealants.

The tested sealants can be divided to three groups according to the clustering of their m_{60} values:

- Sealants H with m_{60} equals to 0.48
- Sealants D, E, and G with m_{60} ranging from 0.414 to 0.422
- Sealants A, B, C, and F m_{60} ranging from 0.369 to 0.397.

Sealants with low creep stiffness and high m-value are expected to be capable of dissipating thermal stresses and have good field performance.

Al-Qadi et al. (2006) used creep stiffness at 240 seconds (S_{240}), m-value at 240 seconds (m_{240}), creep rate at 240 seconds, and average creep rate to evaluate sealant performance at low temperature. Creep stiffness at 240 seconds was used to predict sealant stiffness after 5 hours of loading, according to time-temperature superposition principle, where the daily temperature variation in the two locations involved in this study during the winter showed that sealants may be subjected to tensile strain for 6 to 10 hours during the day.

Table 7.2 shows the values of S_{240}, m_{240}, average creep rate, and creep rate at 240 seconds for all sealants calculated from the BBR results compared with the BBR parameters used in this paper (S_{60} and m_{60}). It was assumed that the creep rate at 240 seconds equals to the secondary (steady state) creep rate and its value will not be affected by using different testing loads. Figure 7.5 (a) shows a comparison between S_{60} and S_{240}, while Figure 7.5 (b) shows a comparison among the different parameters that can be used for evaluating creep rate. In Figure 7.5, each parameter was normalized by the maximum value obtained for this parameter for the tested group of sealants. Figure 7.5 illustrates that there is a good agreement between the BBR parameters used in this study (S_{60} and m_{60}) and the parameters proposed by Al-Qadi et al. (2006) which are S_{240} and m_{240}.

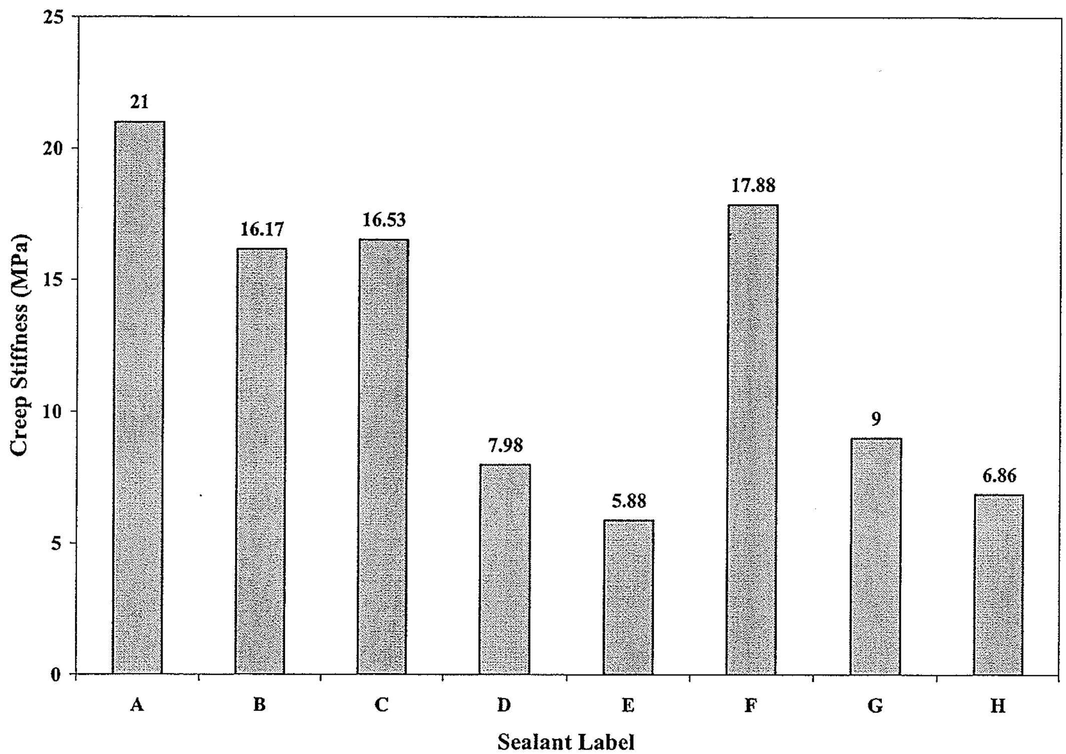


FIGURE 7.3: Creep Stiffness after 60 Seconds of Loading at -30°C

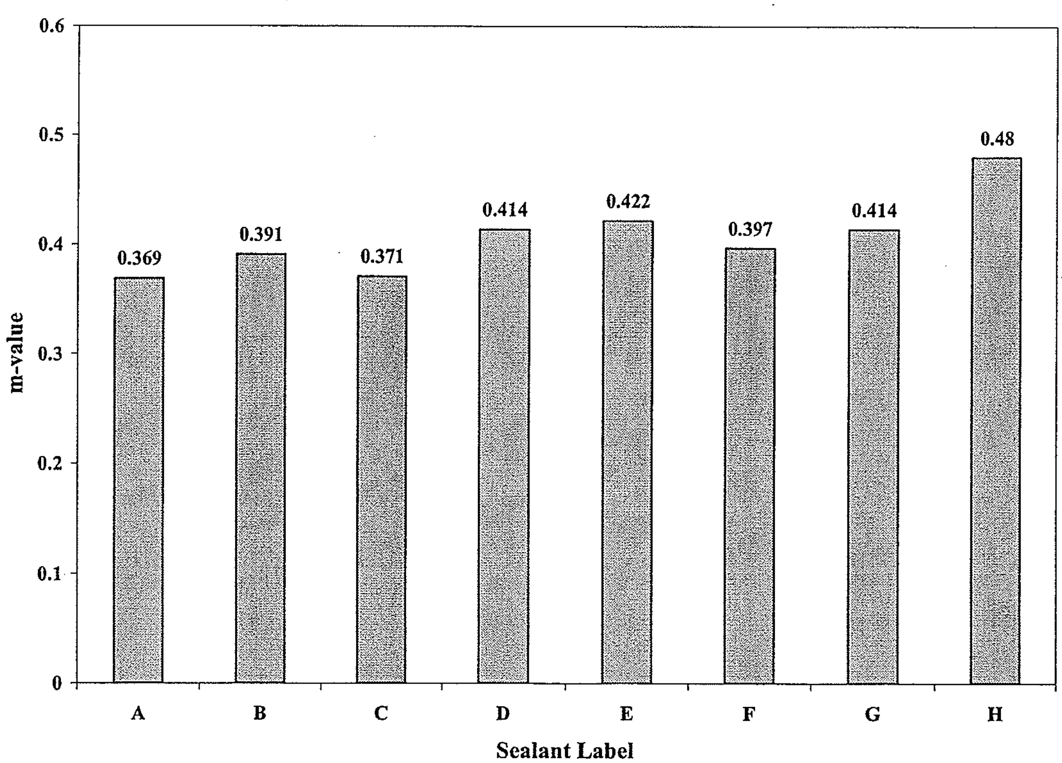
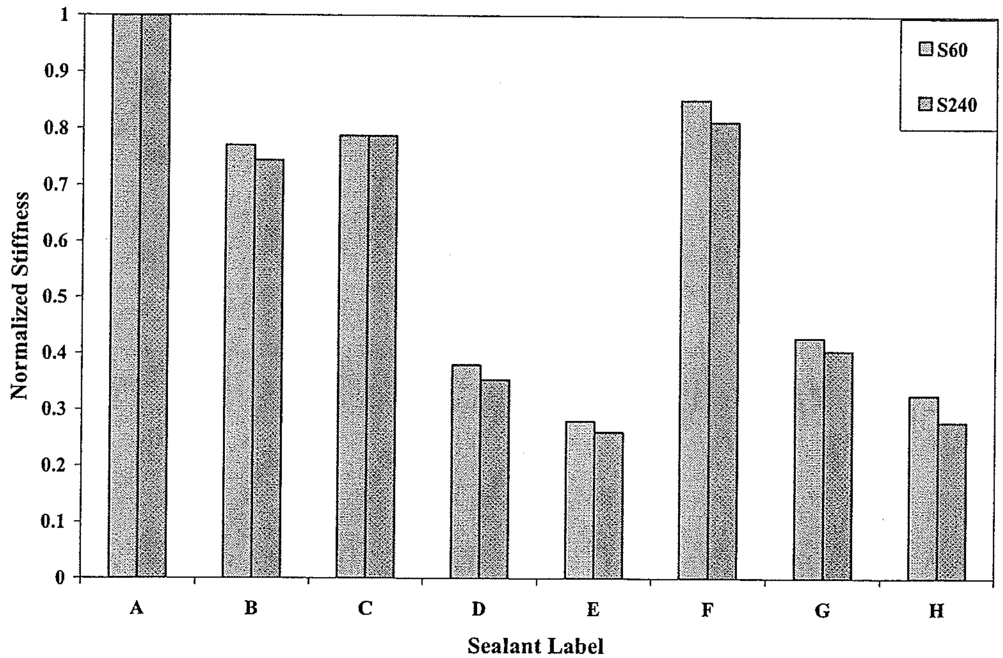


FIGURE 7.4: *m*-value after 60 Seconds of Loading at -30°C

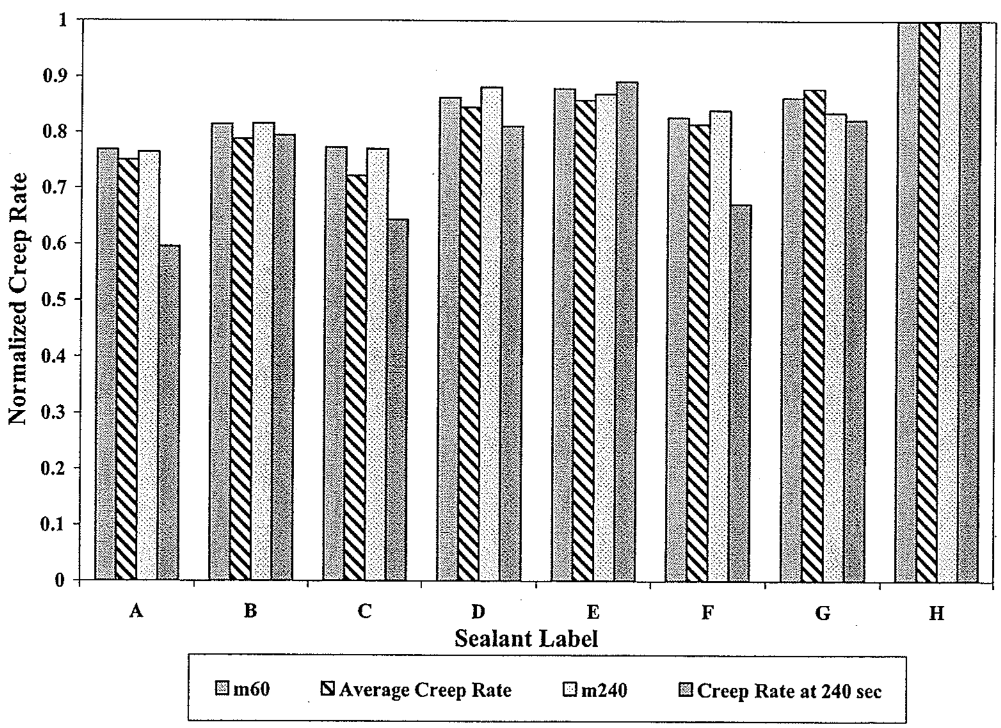
Marasteanu (2004) found that for asphalt binder there is a non-linear relationship between m-value and rate of stress relaxation when m-value is greater than 0.30 and that a higher m-value binder may perform better in cold climates when the temperature remains low for extended periods, which is the case at the test site. Further research need to be conducted to study the relationship between m-value and rate of stress relaxation for sealants.

TABLE 7.2: Stiffness at 240 sec, *m*-value at 240sec, Average Creep Rate, and Creep Rate at 240 sec for All Sealants

Sealant	S_{240} (MPa)	m_{240}	Average Creep Rate	Creep Rate at 240 sec (mm/Sec) x 10^{-3}
A	12.43	0.386	0.364	5.48
B	9.25	0.412	0.382	7.31
C	9.78	0.389	0.350	5.92
D	4.40	0.445	0.410	7.47
E	3.24	0.439	0.416	8.20
F	10.10	0.424	0.395	6.18
G	5.04	0.422	0.426	7.57
H	3.47	0.505	0.485	9.20



a) **Normalized Creep Stiffness at 60 and 240 Seconds**



b) **Normalized Creep Rates**

FIGURE 7.5: Comparison of Evaluation Parameters Obtained from BBR Test

7.2.3 Glass Transition Temperature (T_g) and Low-Temperature Stiffness

The glass transition temperature is an important factor in determining the adequacy of a sealant to certain climatic conditions. A sealant should not be expected to perform adequately in cold climates with temperatures lower than its glass transition temperatures. Where at these temperatures, the sealant material will become very stiff (in the glassy region) and an increase in sealant cohesion and adhesion failures may occur.

Figure 7.6 shows the glass transition temperature for the tested sealants. Sealants A and H have the highest and lowest glass transition temperature (-39.5 and -55°C) respectively, while, the glass transition temperature for the other sealants ranged from -45°C to -48.5°C. According to the values of T_g in Figure 7.6, Sealant H is expected to have a better field performance in cold climates than the other sealants.

Due to the complex composition of hot-pour bituminous sealants which are a blend of several viscoelastic materials that have different T_gs, the glass transition temperature obtained from DMA test can not be considered a precise value. Therefore, it is important to ensure that the minimum in-service temperature is 10°C to 15°C higher (warmer) than the glass transition temperature. This difference guarantees a good field performance. The T_g for Sealant H is lower than the in-service temperature by 14°C and it is the only sealant that has a good field performance (2% failure rate). The T_gs for the other sealants are either lower than the in-service temperature by 4°C to 7.4°C or higher than the in-service temperature by 1.5°C, and they have fair to poor field performance (29% to 70% failure rate).

Figure 7.7 shows the storage modulus at -40°C for the tested sealants. Sealants A and H have the highest and lowest storage modulus. The ability of a sealant to dissipate thermal stresses is governed by its low-temperature stiffness (Marasteanu 2004). Therefore, Sealant H is expected to be more capable of dissipating stresses than the other sealants and to have better field performance at low temperatures.

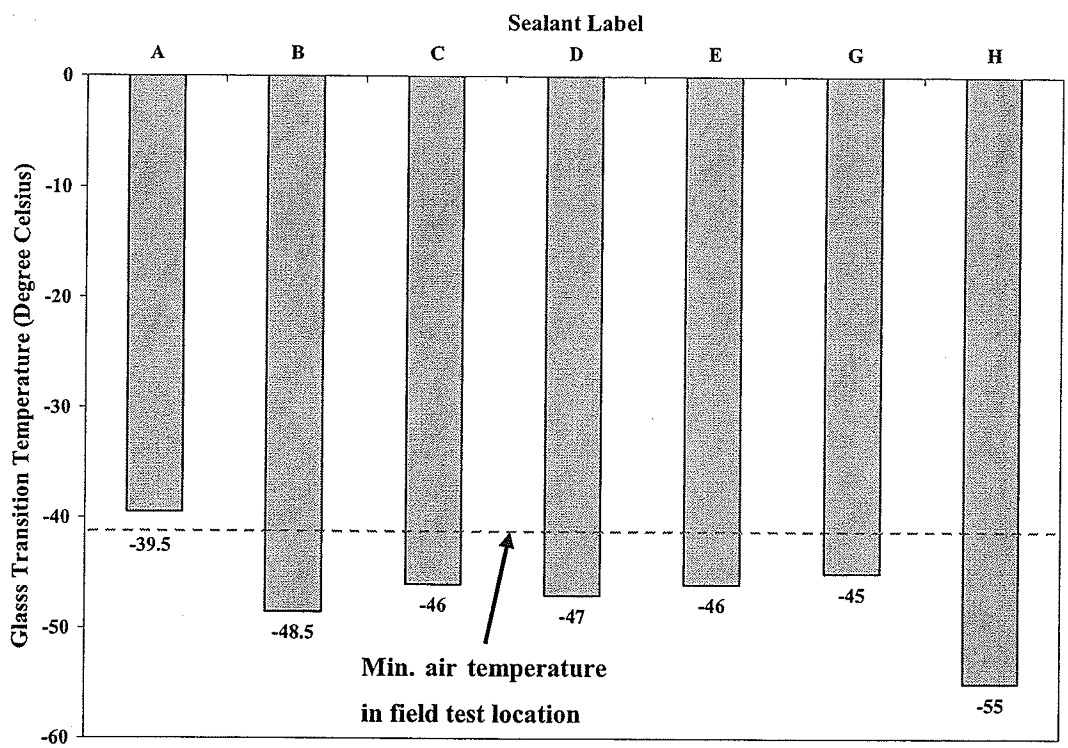


FIGURE 7.6: Glass Transition Temperature (T_g)

7.2.4 Stiffness at Moderate Temperatures and Temperature Susceptibility

The complex shear modulus (G^*) was evaluated for the tested group of sealants using DSR test at temperatures ranging from +5°C to +64°C. Figure 7.8 shows the complex shear modulus at +5°C, +34°C, and +64°C. The complex shear modulus at +5°C can be used as an indicator for sealant stiffness at moderate temperatures.

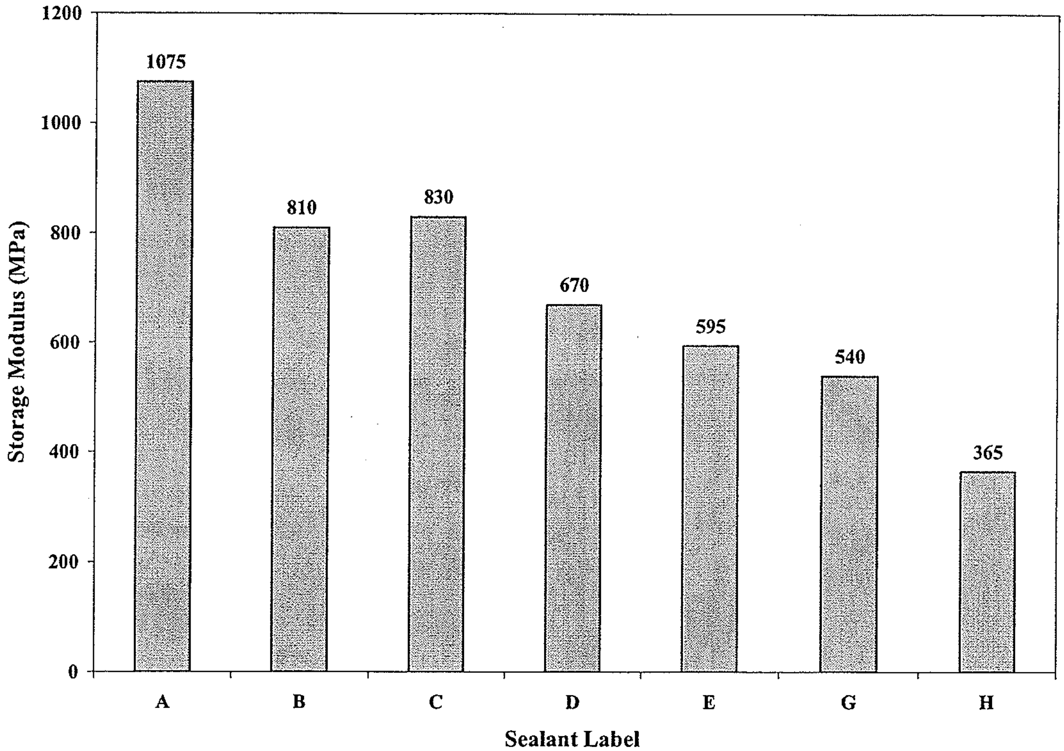


FIGURE 7.7: Storage Modulus at -40°C

Sealants can be classified to three groups according to the clustering of their shear modulus values at +5°C:

- Sealants with low stiffness: sealants E, G, and H with shear modulus ranging from 158.1 KPa and 189.4 KPa
- Sealants with moderate stiffness: sealants C, D, and F with shear modulus ranging from 288.8 to 494 KPa which represents 152% to 312% of the shear modulus of the first group
- Sealants with high stiffness: sealants A and B with complex shear modulus equal to 1211 KPa and 1249 KPa, respectively, which represents 800% of the shear modulus of the first group

Temperature susceptibility (*TS*) indicates the sensitivity of sealant stiffness to temperature change. Figure 7.9 shows the absolute values of the slope of the tangent to G^*-Temperature curve at temperature +7.5°C, where this slope presents the average rate of decrease in sealant stiffness due to increase in temperature (*TS*). The average rate of decrease in sealant stiffness was calculated at +7.5°C to represent the susceptibility of sealants to temperature variation during spring and fall seasons as pavements are subjected to high temperature variation during the day.

Sealants A and B were significantly more susceptible to temperature variation than other sealants. Sealants can be classified to three groups according to the clustering of their shear modulus values at +5°C:

- Sealants with low temperature susceptibility: sealants E, G, and H with an average rate of decrease in shear stiffness ranging from 10.20 to 12.50 KPa / °C
- Sealants with moderate temperature susceptibility: sealants C, D, and F with an average rate of decrease in shear stiffness ranging from 20.30 to 33.7 KPa / °C
- Sealants with high temperature susceptibility: sealants A and B with an average rate of decrease in shear stiffness equal to 88 and 108 KPa / °C, respectively.

Neglecting the effect of other factors, Sealants with low temperature susceptibility are expected to have better field performance.

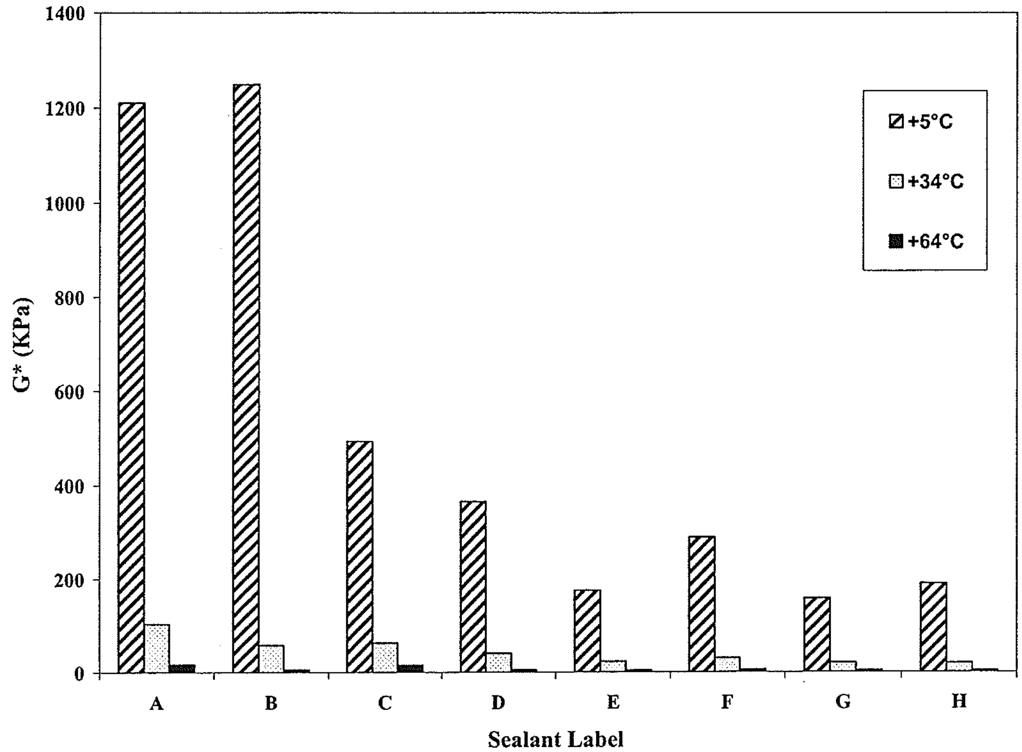


FIGURE 7.8: Complex Shear Modulus (G*) for the Tested Sealants

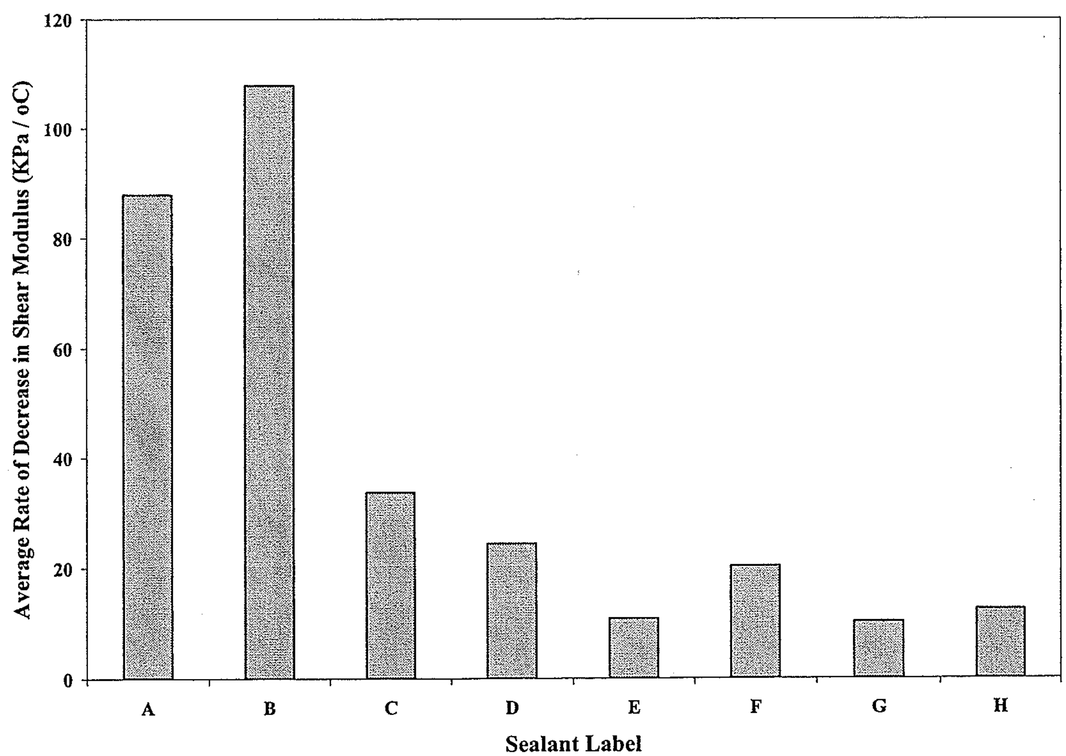


FIGURE 7.9: Average Rate of Decrease in Complex Shear Modulus at +7.5°C

7.3 Correlation between Laboratory-Based Performance Indicators and Field Performance

Manitoba Transportation and Government Services (MTGS) started a field study in 2004 to evaluate the field performance of joint and crack sealants (Worms 2005). The test section was located in an asphalt concrete (AC) major highway. The pavement in the test section consists of a 100 mm thick, four years old AC overlay placed over a 76 mm existing AC pavement on granular base. The total pavement width, including paved shoulders, is 9.20 m. Pavement in test location is subject to severe temperature changes, where the maximum and the minimum air temperatures during the last five years were +36° C and -41° C respectively. The laboratory performance of the sealants used in this study should be evaluated at these temperatures, since they represent the extreme temperatures that sealant experiences during the year.

Sealants were applied to longitudinal and transverse cracks of the test section in 2004. Sealants were inspected in 2005 after one year, and in 2006 after two years. Inspections of transverse and longitudinal sealed cracks were made in early spring when adhesion failures can be visually observed. The rate of failure was calculated for each sealant by dividing the length of failed cracks by the total length of sealed cracks with this sealant. Figure 7.10 shows the rate of sealants failure after two years of sealants application. The rate of failure was calculated for transverse cracks only. Transverse cracks are mainly caused by thermal contraction of pavements and they are subjected to movement of pavements due to contraction and expansion. Sealant A showed the highest failure rate (70%), while, sealant H showed the lowest failure rate (2%).

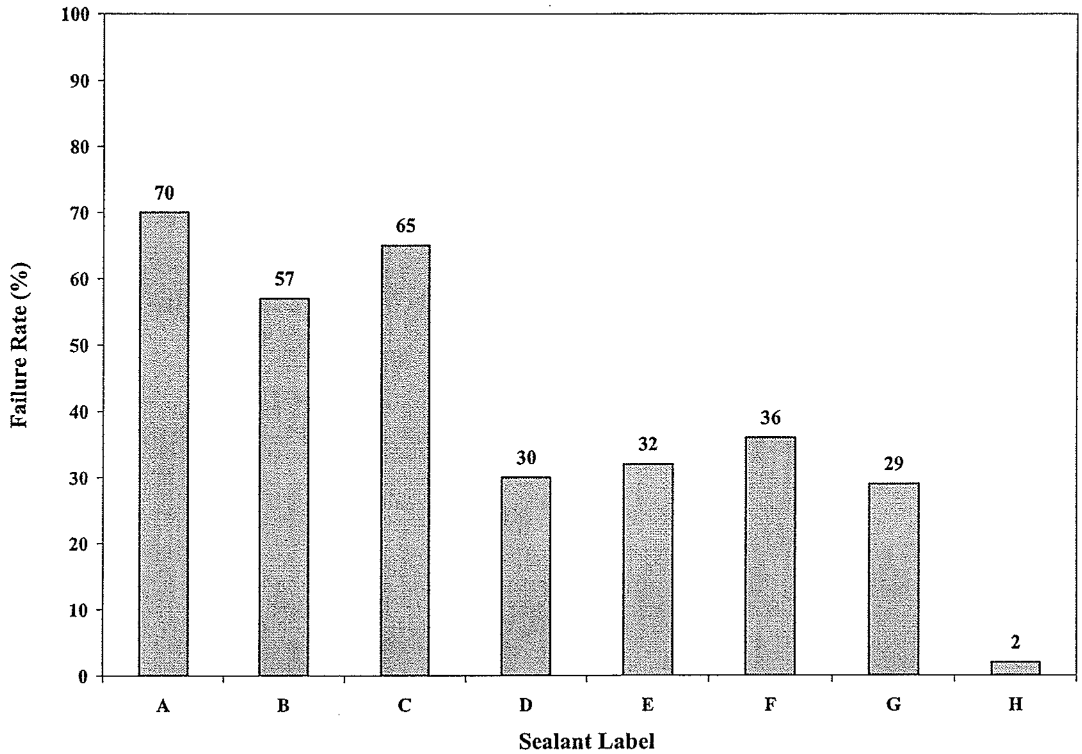


FIGURE 7.10: Rate of Sealants Failure for Transverse Cracks Only

A rating system was developed in SHRP project H-106 (SHRP 1991) to rank sealants according to the percent of sealant failures. In this rating system, sealants were classified to five groups (from excellent to very poor performance), and a 35% failure rate was the boundary between fair and poor performance.

Sealants were classified to three groups according to their failure rates in field, shown in Figure 7.10, after two years:

- Group 1: Good performance, percent transverse failure less than 10%
- Group 2: Satisfactory performance, percent transverse failure from 10% to 35%
- Group 3: Poor performance, percent transverse failure greater than 35%

The 10% failure rate was selected based on MTGS requirements, where, MTGS requires that the failure rate of crack sealant does not exceed 7% after one year of sealant application, and 10% after two years. The 35% failure rate was adopted to distinguish between satisfactory and poor performance based on the SHRP rating system (SHRP 1991).

Using the same three performance groups used for field evaluation, sealants can be classified to three groups according to the clustering of the laboratory-based performance indicators value:

- Group 1: Good performance
- Group 2: Satisfactory performance
- Group 3: Poor performance

Table 7.3 shows the classification of sealants according to their filed performance and laboratory-based performance indicators. From Table 7.3, it is clear that there is a good correlation between laboratory and field evaluation of sealants. There is an agreement between all laboratory-based performance indicators and field performance for Sealant A and H which have the highest and lowest failure rates, respectively, in the field study. Stiffness at -40°C, m_{60}, S_{60}, and T_g have better correlation to field performance than the other laboratory performance indicators.

The number of sealants involved in this research is insufficient to recommend a selection limits for the laboratory-based performance indicators. A threshold value can be developed for each performance indicator when more field performance data is available.

TABLE 7.3: Classification of Tested Sealants According to Field and Laboratory Performance

Sealant	Field Evaluation	Laboratory Evaluation						
		RV	BBR		DMA		DSR	
		Viscosity	S_{60}	m_{60}	T_g	Stiffness at -40°C	G^* at +5°C	*TS* at +7.5°C
A	3	3	3	3	3	3	3	3
B	3	2	3	3	2	3	3	3
C	3	2	3	3	2	3	2	2
D	2	2	2	2	2	2	2	2
E	2	2	1	2	2	2	1	1
F	3	1	3	3	N/A [a]	N/A [a]	2	2
G	2	1	2	2	2	2	1	1
H	1	1	1	1	1	1	1	1

[a] N/A = Not Available

8 Conclusions and Recommendations

8.1 Summary

Pavements in Canada experience large temperature variations resulting in large deformation and cracking. Sealing pavement joints and cracks is one of the essential pavement maintenance practices to protect subsurface layers from the ingress of moisture and debris. Hot-pour sealants, consisting of bitumen modified with polymers, are the most prevalent type of sealants. The type of polymer can affect the performance of sealants. Development of a reliable characterization method for crack sealants has been a challenging process in the last decade. Currently, field studies are the most reliable method to evaluate sealants performance in cold climates which is not a cost-effective method. Field studies are commonly repeated on a 10 years cycle, which does not provide a timely response to progressive market changes and the availability of new products.

In this thesis, a test protocol is proposed for full characterization of hot-pour bituminous sealants. Four test methods are adopted in this test protocol: Rotational Viscometer (RV), Dynamic Shear Rheometer (DSR), Bending Beam Rheometer (BBR), and Dynamic Mechanical Analyzer (DMA). These four tests characterize the rheological properties of sealants over the wide range of temperatures that they experience, starting from installation temperature and ending with the minimum in-service temperature. The major advantage of using these methods is that the equipment is readily available in asphalt binder and polymer characterization laboratories; and the procedures are familiar to laboratory technicians.

Eight hot-pour bituminous sealants were evaluated according to the proposed test protocol. Two sealants are classified as Type I and the remaining six as Type IV according to ASTM Standards D6690. RV test was conducted at +180°C, which is the average installation temperature for the tested sealants, and a spindle speed of 50 rpm. The standard BBR test load (980 mN) was reduced to be 500 mN for stiff sealants and 200 mN for soft sealants. The BBR test was conducted at a temperature of -30°C. The 25-mm diameter specimen was adopted for DSR test at all temperatures to capture the complex composition of the hot-pour sealants. DSR test was conducted in the strain controlled mode with strain amplitude of 2% for temperatures ranging from +5°C to +40°C, and 4% for temperatures ranging from +46°C to +64°C. The dual cantilever clamp was adopted for DMA test. Sealant specimens were subjected to oscillating strain with amplitude of 5 μm and frequency 1 Hz. DMA test was conducted at temperatures ranging from -70°C to 0°C.

Seven parameters were selected as laboratory-based indicators for sealant performance. For each sealant, the values of these performance indicators were evaluated. Sealants were ranked according to their laboratory performance indicators. Moreover, sealants were ranked according to their failure rate after two years of application in the field. The results of field evaluation and laboratory evaluation were compared together to study the correlation between them.

8.2 Conclusions

The proposed test protocol in this thesis provides a full characterization of sealants over the range of temperatures that they experience during installation and in-service. Asphalt binder test protocols, with some modification, are utilized in the proposed method to characterize the rheological properties of sealants. The proposed test protocol can be considered a cost-effective and a rapid alternative to field performance studies of hot-pour bituminous sealants.

The results of the laboratory and field evaluation of the tested group of sealants led to the following findings:

- The creep stiffness (S_{60}) is directly proportional to sealant field performance, and a creep stiffness of 10 MPa at -30°C is recommended as a threshold value to distinguish between sealants with poor performance and sealants with good or satisfactory performance.
- The creep rate (*m*-value) is inversely proportional to sealant field performance and a strong correlation was found between them.
- The glass transition temperature is best estimated from the loss modulus-temperature curve. The glass transition temperature could not be estimated from tan δ-temperature curve because no distinct peaks were detected for several sealants. This condition can be attributed to the complex composition of hot-pour bituminous sealants which are a blend of several viscoelastic materials that have different glass transition temperatures.
- The glass transition temperature obtained from DMA test is not a well defined value due to the complex composition of hot-pour bituminous sealants.

- Sealants should be selected such that the glass transition temperature is lower than the minimum in-service temperature by a range of 10°C to 15°C to guarantee a good field performance.
- The low-temperature stiffness (storage modulus) at the minimum in-service temperature is directly proportional to sealant field performance and a strong correlation was found between them.
- The viscosity at installation temperature is directly proportional to sealant field performance and a good correlation was found between them.
- A good correlation was found among the complex shear modulus at +5°C, temperature susceptibility at +7.5°C, and the field performance of sealants.
- The phase angle, obtained from the DSR test, for the tested sealants ranged between 23 and 48.0 degrees, and was not sensitive to temperature changes in the range of +5°C to +64°C. These values are lower than typical phase angle values for asphalt binders at this range of temperatures.
- Current ASTM specification for hot-pour bituminous sealants evaluates the bond strength between sealants and pavements at low temperatures but it does not characterize the low-temperature properties of sealants. Glass transition temperature, low-temperature stiffness, creep stiffness, and m-value provide fundamental information about the low-temperature properties that can be correlated to the field performance of sealants.
- The performance of sealants can not be evaluated with one test only; more than one test is required.

- Asphalt binder tests (RV, DSR, and BBR) can be used for characterizing sealants performance providing that some modifications should be done in the testing procedures.

8.3 Recommendations for Future Work

Selection limits can be developed for the proposed laboratory-based performance indicators by comparing their values to the actual field performance of sealants obtained from field studies in other cold regions. The number of sealants tested in this study is insufficient to recommend these selection limits.

In the literature, the relationship between m-value and rate of stress relaxation was found to be non-linear when m-value is greater than 0.30 (Marasteanu 2004). Further research is needed to study the relationship among m-value, creep stiffness, and rate of stress relaxation for sealants.

The relationship between the glass transition temperature and the minimum in-service temperature can be developed further through a testing program. Results of this testing could suggest a minimum acceptable difference (factor of safety) between the two temperatures to guarantee good field performance.

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