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Low Temperature Rheological and Mechanistic Characteristics of Three Different Types of Asphalt Binders

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Abstract: This study investigates the rheological and mechanistic characteristics of unmodified, polymer modified asphalt binder and epoxy asphalt binder at low temperatures. The dynamic shear rheometer (DSR) test and lap shear strength test were conducted to examine the rheological and mechanistic properties of the three types of asphalt binders. The results indicate that the complex modulus and lap shear strength of modified asphalt binder were higher than unmodified asphalt binder. Epoxy asphalt showed the highest complex modulus at lower temperatures. The phase angle of modified asphalt binder was low, and it increased slightly with the increasing temperature. The results of lap shear testing agree with the DSR test results. The lap shear bonds method is a reliable test method for evaluating asphalt performance at low temperatures quickly.

Key words: *Adhesive; Asphalt; Lap shear bond; Strain rate; Temperature.*

Introduction

To achieve better pavement performance, polymer modified asphalt has been widely used as road paving materials worldwide in the last two decades. However, testing methods and specifications have not been well established for polymer modified asphalt [1-3]. In fact, the conventional test methods for original asphalt specifications, such as penetration, ring and ball point, and viscosity, are not sufficient in characterizing polymer modified asphalt. It has been suggested that rheological tests be used to characterize polymer modified asphalt, and some authors have correlated rheological data like complex shear modulus (G*), phase angle (δ), creep stiffness (S) and creep stiffness change rate (m) with high and low temperature performances. Dynamic shear rheometer (DSR) and bending beam rheometer (BBR) were used to evaluate the high and low temperature performance of asphalt, respectively [4-5]. Rheological tests generally provide more reliable information about the asphalt binders. In the dynamic shear rheometer test, the shearing frequencies used in the sample characterization can be correlated to traffic conditions [10-13]. In these studies, frequencies between 10^{-1} and 10^2 rad/s were used to simulate normal vehicle traffic on pavements. Two important parameters obtained from rheological tests in dynamic mode are the complex modulus (G*) and the phase angle (δ). The former can be related to the material strength, and the latter provides information about the ratio between elastic and viscous responses during the shearing process.

Creep compliance is a fundamental material property that represents the rheological behavior of viscoelastic materials. Accurate determination of the creep compliance of asphalt is crucial in evaluating the time/frequency dependent stress and strain response. Under small strain levels, the unmodified asphalt exhibits linear rheological behavior so it can be generally characterized as a linear viscoelastic material. At low temperatures (or under short loading durations), the behavior of asphalt approaches the behavior of elastic solids. However, a weak viscoelastic behavior is observed as the temperature increases. Two different geometric configurations of test specimens is required for asphalt binders tested using the DSR test and the BBR test. It would be very beneficial to select or develop a test method that evaluates asphalt performances over wide temperature ranges.

In fact, the adhesion between asphalt and aggregate affects asphalt concrete strength. Strong adhesion between asphalt and aggregate is a key factor in achieving good pavement performance and durability [6-7]. Since the 1920s, researches and evaluations have been conducted to understand and resolve moisture damage or simply the stripping of asphalt pavement [8-10]. However, most of these studies concentrated only on the mechanistic properties of the mixtures, and few focused on the adhesion behaviors between the aggregate and binder. The separation between asphalt binder and aggregate may be attributed to the adhesion and cohesion of binders. Adhesive failure occurs when the bond between asphalt binder and aggregate is broken. The most significant outcome of stripping research is the identification of chemical components of the asphalt binder and aggregate that affect adhesion between asphalt and aggregate. However, there is little research on how the chemical components of asphalt binder affect the adhesion between the binder and aggregate, because chemical compositions of asphalt binder from different crudes are difficult to analyze. Furthermore, different modifiers may influence asphalt pavement performance differently [11-13]. Therefore, the effect of polymer modifiers on the adhesion strength of asphalt binders and how that influences the pavement needs further study.

In this study, the rheological properties of three different types of asphalt binders— virgin asphalt binder, polymer asphalt binder, and epoxy asphalt binder— are evaluated at low temperatures. The lap shear bond method is commonly used in the adhesives industry to

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evaluate different adhesives. Asphalt in pavement applications can be treated as an adhesive that binds aggregates together. Thus, the lap shear bond method was chosen to evaluate the low temperature properties of asphalt binders in this study. Complex modulus and the phase angle of asphalt binders were evaluated between temperature ranges of -10°C to 45°C, and the conventional indirect tensile strength was also determined. It was thought that the lap shear bond method would simulate perfectly the adhesions between aggregate and aggregate or between aggregate and asphalt binder. This research also evaluates whether the lap shear bond method is an appropriate and effective method in evaluating asphalt binder properties at low temperatures.

Experimental Program

Materials

Three types of asphalt binders, including virgin unmodified binder, polymer modified asphalt binder, and epoxy asphalt binder, with different performance tracks were evaluated in this study. Unmodified asphalt yielded a penetration of 77.8 mm at 25°C, a ductility of 200 cm at 15°C, a softening point of 44.8°C, and a viscosity of 0.45 Pa.s at 135°C. The polymer used in this study was Styrene-Butadiene-Styrene (SBS) triblock copolymer, grade 1301, and was manufactured by the Yueyang Petrochemical Co., Ltd., in China. The grade 1301 SBS polymer was a linear-like material, containing 30% of styrene by weight with an-average molecular weight of 120,000 g/mol. The 4.5 wt% SBS modified asphalt binder had the following properties: a penetration of 69 mm at 25°C, a ductility of 45 cm at 5°C, and a softening point of 81.8°C. The epoxy resin used to produce epoxy asphalt in this study was diglycidyl ether of bisphenol A with an epoxy value of 0.52 mol/100g. It was obtained from Shanghai Xinhua Resin Co., Ltd, in Shanghai, China. The methyl tetrahydro phthalic anhydride (MTHPA) curing agent was provided by Jiaxing Fine Chemical Co., Ltd., in the Zhejiang province of China. The epoxy asphalt with 30 wt% epoxy resin was prepared. All materials were commercially available in China and were used as received.

In preparation of the SBS modified asphalt samples, the asphalt binder was heated to 175°C±5°C in an oil-bath heating container until it flowed freely. The 4.5 wt% SBS was mixed into the asphalt under a 5000 rpm rotation speed for about 60 minutes to ensure that the mixture became essentially homogeneous. For epoxy asphalt samples, the asphalt binder was heated to 150°C±5°C in an oil-bath heating container until it flowed freely. Appropriate amounts of curing agent were then added into the blend and mixed for 30 minutes with a lab mixer at fast speed (usually 500 rpm) to create a small vortex without whipping excessive air into the sample. The mixtures were cooled down to 120°C. The epoxy asphalt was obtained when the desired amount of epoxy resin (preheated to 120°C) was added into the mix, and the mixer continued to stir the mix for 5 minutes under the same mixing condition. The 30 wt% epoxy resin was added into the asphalt to prepare the epoxy asphalt in this study.

Test Methods

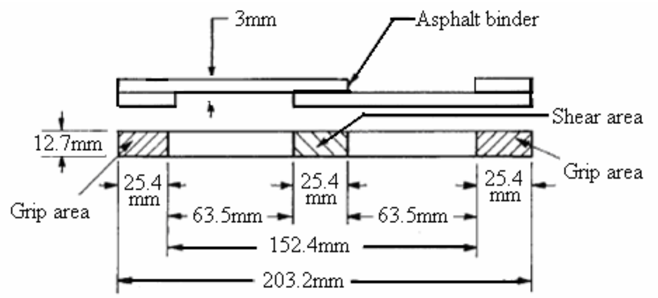

Fig.1 Specimen Geometry and Dimensions of Lap Shear Test.

The conventional physical properties of bitumen (empirical rheological tests), including penetration at 25°C, softening point, ductility at 15°C and elastic recovery at 25°C, were determined in accordance with standard test methods ASTM D5, ASTM D36, ASTM D113 and ASTM D6084, respectively. The penetration is an empirical test used to measure the consistency of bitumen. In this test, a container of bitumen is brought to the standard test temperature in a thermostatically- controlled water bath. The sample is placed under a needle of prescribed dimensions. The needle is loaded with a 100 g weight and allowed to penetrate the bitumen sample for 5 seconds. The depth of penetration is measured in units of 0.1 mm and is reported in penetration units. The softening point is measured by the ring and ball (R&B) method. The test consists of taking a brass ring filled with bitumen and suspending it in a breaker filled with water. A steel ball of specified dimensions and weight is placed in the center of the sample. The bath is heated at a controlled rate of 5°C/min. When the bitumen softens, the ball and bitumen sink toward the bottom of the breaker. The temperature is recorded at the instant when the softened bitumen sinks the prescribed distance and touches the bottom plate. The ductility test measures the distance in centimeter that a standard briquette of bitumen will stretch before breaking.

The lap shear bond method was used in this study to evaluate the adhesion property of the three types of asphalt binders. As shown in Fig. 1, the metal-metal (steel with 3.0 mm thickness) configuration was used for lap shear adhesive tests [14]. The overlapping zone was 322.58 mm^2 in area (25.4 mm×12.7 mm) for each case. The thickness of the asphalt binder within the overlapping area was 0.03 mm. Before applying asphalt binders onto the adherent surfaces, the surfaces were ground with emery paper and washed with trichloroethylene to remove grease and organic oil materials, then dried at 100°C. The asphalt binders were painted onto the metal surfaces as a layer between 0.2 mm to 0.3 mm thick, which was formed using a simple device made from distance wires. The subsequent curing reaction proceeded at room temperature. The metal-metal joints were fixed in a special press to maintain a constant position and cohesion of the film during hardening. A pressure of 0.3 MPa was applied, and two steel shims was utilized to adjust the desired thickness of the adhesive. The extra asphalt binder on the edge was removed using a hot steel spatula. The specimen's geometry and the dimensions of the lap shear test are shown in Fig. 1. The testing of the effect of temperature on the adhesive strength of asphalt binder with metal-metal joints was done subsequently.

Fourier Transform Infrared (FTIR) Spectroscopy (NEXUS, Thermo Nicolet, USA) was used to obtain the IR spectra of asphalt binder samples. Infrared spectroscopy is one of the most important analytical techniques available in today's researchers. It can study samples in virtually any state. Liquids, gases, solid, solutions, powders, and surfaces can all be tested with a judicious choice of sampling technique. Infrared spectroscopy is a technique based on the vibrations of a molecule's atoms. An infrared spectrum is commonly obtained by passing infrared radiation through a sample and determining what fraction of the incident radiation is absorbed at a particular energy. The energy of any peak in an absorption spectrum corresponds to the frequency of a vibration of a part of the sample molecule. A sample was prepared by casting film onto a potassium bromide (KBr) thin plate, and the spectra were obtained with a 4 cm^{-1} resolution.

As has been well documented, asphalt binders' behavior is dependent on temperature and time of loading. Unlike steel, which is considered a completely elastic material that can store energy indefinitely, asphalt binders cannot sustain a load without showing time-dependent deformation known as creep [15-18]. Additionally, some of the input energy dissipates in the material and results in a permanent deformation. This is referred to as viscoelastic behavior. Samples exhibiting viscoelastic behavior are well suited for rheological characterization. At high temperatures or long periods of loading, asphalt binders act like high-viscosity flowable fluids [19-22].

Viscosity is defined as the resistance to flow. As the viscosity decreases, a sample will flow more readily under the same conditions of temperature and load. At low temperatures or short loading periods, asphalt binders act like elastic solids. Elastic solids approach the response of steel, where all the input energy is recoverable. Traditional tests used to characterize asphalt binders were either penetration or simple one-point viscosity tests using a capillary device held at constant temperature. Both of these measurements have shortcomings and provide only limited insight into the overall flow behavior and ultimate end use performance of an asphalt binder. Often these traditional tests provide misleading results in regard to the effect of additives, modifiers, and environmental conditions.

Dynamic shear properties were measured with Physical MCR 101 dynamic shear rheometer (Anton Paar Inc., Austria) in a parallel plate with a 2 mm gap and an 8 mm diameter. A DSR temperature sweep test was performed under the strain controlled mode at a constant frequency of 10 rad/s. The applied strain was kept low enough so that all tests were performed within the linear viscoelastic range. Test temperatures varied from -10°C to 45°C with a 2°C increment per minute. The complex modulus and phase angle at different temperatures were recorded automatically during the test.

A bending beams rheometer (BBR), manufactured by Cannon Instrument Company, was used to conduct low temperature creep tests. The asphalt binder beams (125 mm×12.5 mm×6.25 mm) were prepared in an aluminum mold. In preparation, the asphalt binder was heated to a fluid state, poured into the mold, and allowed to cool to room temperature for approximately 90 minutes. The sample was then cooled to approximately -5°C for 1 minute and de-molded. Plastic strips were used to release the beam from the mold. Sets of the sample beams were submerged in a methanol bath with a constant temperature of -10°C or -18°C. After storage for different length of time, the rectangular beam was placed on two stainless steel supports (102 mm apart) and loaded with a 100-g load. The deflection of the centre was measured continuously with a linear variable differential transformer (LVDT). The creep stiffness (S) and creep rate (m) of the binders were determined at loading time 60 s.

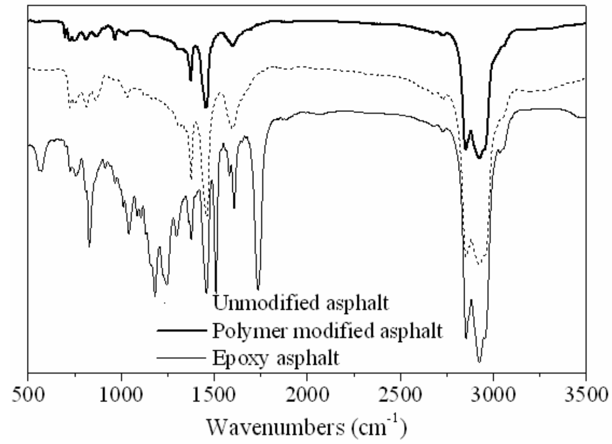

Fig. 2 Results of the IR Test of the Three Types of Asphalt Binders.

Results and Discussions

Infrared Spectroscopy Analysis

Fig. 2 shows the infrared spectroscopy analysis curves of the three types of asphalt binders tested at 25 °C. For all three asphalt binders, the strong peaks within 2850-2960 cm^{-1} region are typical *C-H* stretching vibrations in aliphatic chains. The *C-H* asymmetric deformation in CH_2 and CH_3 and the *C-H* symmetric deformation in CH_3 vibrations are observed at 1400 cm^{-1}-1500 cm^{-1} and 1370 cm^{-1}-1390 cm^{-1}. The characteristic absorption peak around 1545 cm^{-1}-1640 cm^{-1} is attributed to *C=C* stretching vibrations in aromatics.

In addition to these common characteristics represented by the observed absorption peaks, additional peaks are presented for epoxy asphalt binder. The additional peaks are located at 830 cm^{-1} and 1509 cm^{-1}, due to *p*-phenylene groups; at 906 cm^{-1}, due to the epoxy group; and at 1035 cm^{-1}, due to the aliphatic carbon-oxygen stretching (-O-CH_2-). Finally, the peak revealed around 1740 cm^{-1} was caused by ester carbonyl of the cured product of epoxy asphalt. The results indicate that the epoxy resin did not simply mix with the asphalt binder. In the epoxy asphalt binders, the epoxy resin reacted with both the curing agent and the asphalt binder. Observed from the infrared spectroscopy analysis results, the three types of asphalt binders possess different chemical components, especially in regards to epoxy asphalt. The different compositions of the three asphalt binders might lead to different pavement performances.

Complex Modulus and Phase Angle

Fig. 3 shows the temperature dependency of the complex modulus (G^*) and phase angle (δ) for the three types of asphalt binders in the range between -10°C to 45°C. The test results indicate that modified

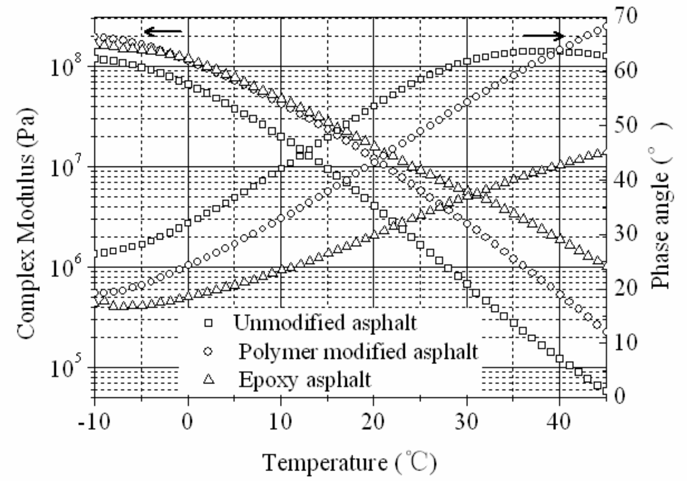

Fig. 3 Complex Modulus and Phase Angle for the Three Types of Asphalt Binders.

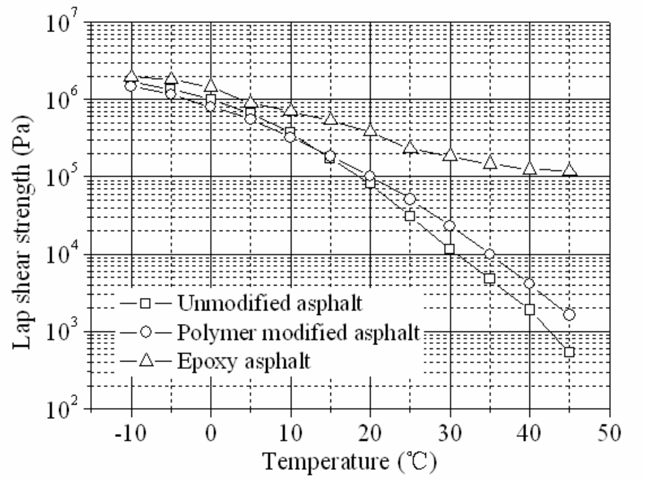

Fig. 5 Log Lap Shear Strength of Asphalt at Different Temperatures.

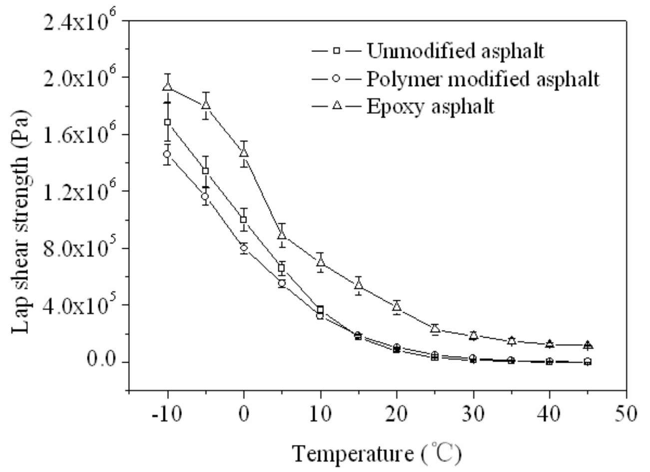

Fig. 4 Results of Lap Shear Strength of Asphalt at Different Temperatures.

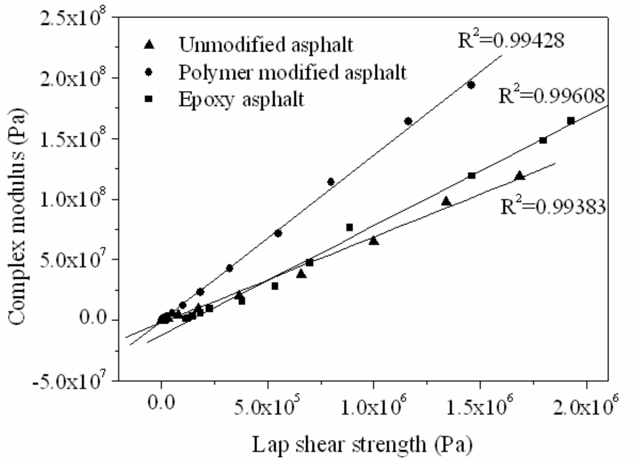

Fig. 6 Complex Modulus as a Function of Lap Shear Strength of Asphalt Binders.

asphalt binders show an increase in the complex modulus and a decrease in the phase angle compared to unmodified asphalt binder, especially the epoxy asphalt. The slopes of the master curves (G^*) decrease from unmodified asphalt and polymer modified asphalt to epoxy asphalt in the temperature range of 0°C to 40°C. The phase angles of the unmodified, polymer modified, and epoxy asphalt all increase with a temperature increase. However, epoxy asphalt had the least increase in phase angle. The polymer modified and epoxy asphalt were less sensitive to temperature changes. In other words, the modified asphalt binders demonstrate a higher ability to maintain elastic/viscous capability than the unmodified asphalt does.

Lap shear strength

Fig. 4 shows the results of the lap shear strength test for the three types of asphalt binders within the temperature range of -10°C to 45°C. Tests were repeated at different temperatures to determine the reproducibility of the lap shear test. Results show that the lap shear strength of all three asphalt binders decreased with the temperature increase. The error bars on the graph indicate that there is little experimental error associated with the lap shear test, and it is a reproducible experimental method aimed to investigate the adhesion of asphalt at working temperatures. The modified asphalt binders show higher lap shear strength than unmodified asphalt binder at the same temperature, with the epoxy asphalt having the highest lap shear strength. The modified asphalt binders seemingly have better low temperature performance.

The lap shear strength test results for the three types of asphalt binders are also presented in logarithm form, as shown in Fig. 5, and tested within the temperature range of -10°C to 45°C. As seen in Fig. 5, dispersions of the three lap shear strength curves increase as the temperature increases. The strength of polymer modified asphalt is lower than that of unmodified asphalt when the temperature is below 10°C. This phenomenon may be attributed to the fact that SBS can improve the flexibility of asphalt at a low temperature. However, the strength of epoxy asphalt binder is higher than that of other asphalt binders for the entire testing temperature range. In addition, the SBS and epoxy resin do not have significant effects on the lap shear strength when the temperature is lower than 20°C. This is observed from the complex modulus tested using the DSR device. The asphalt fails in a cohesive manner when the temperature is higher than 10°C. These results indicate that, at low temperatures, failures might not be caused by a lack of ductility within the asphalt binder but instead by the loss of bond between asphalt and aggregate. Therefore, the lap shear bonds method is effective in testing the low temperature performance of asphalt binder.

In Fig. 6, the complex moduli of asphalt binders are shown as functions of the lap shear strength of asphalt binders tested between -10°C and 45°C. As expected, the complex modulus values increase as the lap shear strength increases. Good linear relationships are observed for all three types of asphalt binders, as indicated by their high coefficients of correlation (R^2 values). Thus, both the complex modulus and lap shear strength tests can be performed to consistently evaluate the low temperature performance of asphalt binders.

Low Temperature Creep Properties

The bending beam rheometer (BBR) was used to accurately evaluate asphalt binder properties at low temperatures. At these temperatures, asphalt binders are too stiff to be reliably evaluated using the parallel plate geometry of the DSR equipment [23-25]. The BBR test measured creep stiffness (S) and the m-value (m) of asphalt binders. Superpave technology specifies that the creep stiffness must not exceed 300 MPa to prevent low temperature cracking. Since low temperature cracking occurs only after the pavement has been in service for some time, this specification addresses these properties using aged binder. The rate of change of asphalt binder stiffness with loading time is represented by the m-value. A high m-value is desired. A minimum m-value of 0.3 after 60 seconds is required by the Superpave PG binder specification.

The effect of diatomite on the stiffness and m-value of asphalt binder at -10°C and -18°C is shown in Table 1. The results reveal that the stiffness of all asphalt binder satisfies the SHRP Superpave specifications at -10°C, and all asphalt binders show higher m-values than the required minimum. The low stiffness value was obtained when polymer was added into the asphalt binder. The results indicate that the asphalt concrete using polymer modified asphalt binders may decrease the susceptibility to low temperature cracking, especially in cold weather areas. However, the epoxy asphalt shows higher stiffness value, which may be attributed to its high mechanical properties. Nonetheless, the stiffness and m-values of these three types of asphalt meet the SHRP Superpave specifications at -10°C. Test results also show that unmodified and polymer modified asphalt binder did not satisfy the SHRP Superpave specification creep stiffness requirement of a maximum of 300 MPa at a loading time of 60 seconds at -18°C. The stiffness value of epoxy asphalt did meet the requirement. The incorporation of polymer or epoxy can improve the low temperature performance of asphalt, but only epoxy asphalt can meet the creep stiffness requirement.

The low temperature properties of these three types of asphalt were also evaluated by the limiting stiffness temperature (i.e. temperature at 300 MPa creep stiffness). Fig. 7 presents the limiting stiffness temperatures for the three types of asphalt binder. The results indicate that the polymer and epoxy resin modification reduces the asphalt limiting stiffness temperature.

Table 1. Effect of Diatomite on Low Temperature Performance Asphalt Binder

Sample	-10℃		-18℃	
	S/MPa	m	S/MPa	m
Unmodified Asphalt	89.5	0.438	312.3	0.302
Polymer modified Asphalt	87.4	0.431	303.3	0.296
Epoxy Asphalt	106.3	0.461	296.2	0.307

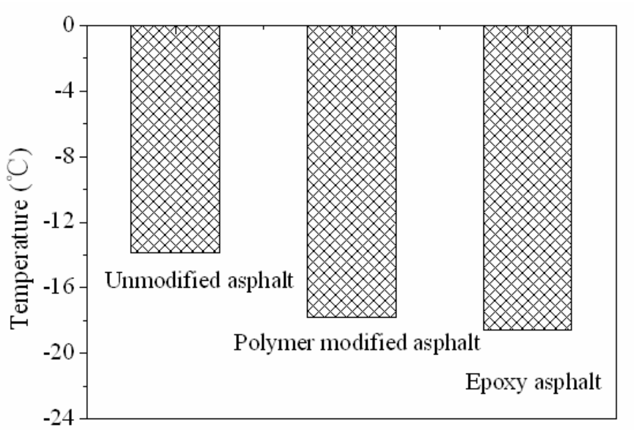

Fig.7 Temperature at 300 MPa Creep Stiffness.

Conclusions

Based on the laboratory test results and analysis of the rheological and mechanistic properties of the three types of asphalt binders, the following conclusions were drawn:

1. At lower temperatures, the complex modulus of modified asphalt binder is higher than that of unmodified asphalt binder, with the epoxy asphalt having the highest complex modulus. The phase angle of modified asphalt binder is low, which increases slightly with increasing temperature.
2. The lap shear strength of modified asphalt binder is higher than that of unmodified asphalt binder. Again, epoxy asphalt shows the highest lap shear strength.
3. The complex modulus increases as the shear strength increases. A lap shear bonds test is reliable and fast for evaluating the performance of asphalt at low temperature.
4. Polymer modified asphalt and epoxy asphalt show better low temperature creep properties than unmodified asphalt. Epoxy asphalt is the best out of the three kinds of asphalt.

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