Technical Paper

ISSN 1997-1400 Int. J. Pavement Res. Technol. 4(4):222-230
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Development of Microindentation Tests for the Specification Grading of Asphalt Cements

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Abstract: This paper documents and discusses the development of instrumented microindentation tests for the specification grading of asphalt cements. Seven recovered asphalts from an Ontario trial were tested with a flat-ended punch indenter in both static creep and dynamic oscillatory tests on thin films. The rheological properties determined include creep stiffness and slope of the creep stiffness master curve (S and m-value); elastic/total work of indentation (W_e/W_t); elastic recovery (ER); viscous creep compliance (J_v); dynamic storage, loss and complex moduli (E', E", and E*); and dynamic phase angle (δ). Dynamic tests were generally found to be more reproducible than creep tests, likely due to the intricacies in contact detection for the latter. Indentation creep stiffness, viscous creep compliance, and phase angle correlated well with performance. Previous investigations revealed that creep properties determined on rolling thin film oven/pressure aging vessel residues for these sections failed to show any correlation with their amount of cracking in service. This is probably due in part to the unrealistic film thicknesses of 1.2 mm and 3.2 mm and temperatures of 163°C and 100°C employed in RTFO and PAV aging methods. Another look at thin film aging protocols is warranted, since thinner films should enable asphalt cements to be aged more realistically at lower temperatures and pressures. More accurate asphalt aging and conditioning protocols combined with the newly developed tests will allow the reliable and precise selection of asphalt cements to ensure their maximum durability.

Key words: *Aging, Asphalt cement, Indentation testing, Specification grading, Thermal cracking.*

Introduction

North American asphalt cement aging protocols involve a rolling thin film oven (RTFO), which simulates accelerated aging during hot-mix production, followed by the pressure aging vessel (PAV), which simulates 8-10 years of slow aging in service [1, 2]. RTFO aging is done on ~1.2 mm thick rolling thin films inside glass bottles at 163°C for 85 minutes. The PAV exposes significantly thicker stationary films of ~3.2 mm of RTFO-aged asphalt cement to an air pressure of 2.1 MPa for a period of 20 hours at a temperature of 100°C. The PAV-aged asphalt cement residue is used to determine the intermediate and low temperature performance grades meant to mitigate cracking [1, 2].

While the 163ºC and 1.2 mm thick rolling films in the RTFO can be considered somewhat representative of those used during construction, the 100ºC and 3.2 mm values in the PAV are likely too high in terms of service conditions. Asphalt has a different phase structure at 100ºC than at typical pavement temperatures and can therefore be expected to age differently. The thick films and high pressures used in the PAV likely suppress the evaporation of even the most volatile asphalt fractions and additives.

Our earlier investigations of nearly 50 pavement trial sections and numerous regular contracts [3-8] have shown that asphalt cements of supposedly identical grades can provide performance varying from best-case to worst-case scenarios. Careful comparisons of laboratory aged and recovered asphalt cements in these studies have revealed that a significant part of the problem lies in the inability of current aging and conditioning protocols to replicate the chemical and physical processes that contribute to hardening in service. Thus, significantly different chemical and physical aging methods are needed if we want to improve our ability to control thermal and fatigue cracking distress.

The main reasons for the compromises made during the adoption of the RTFO/PAV protocol arose from the need for large quantities of material for dynamic shear rheometer (DSR) and bending beam rheometer (BBR) grading and the slow aging of thick films [1, 2]. These situations no longer apply.

The long-term objective of our work is to develop reproducible and accurate specification grading tests for very thin asphalt cement films. Such films should be chemically aged in a manner that better reflects service conditions at lower temperatures and pressures and within reasonable times. Hence, specification properties obtained from the rheological testing of such films after appropriate conditioning at low temperatures should improve our ability to limit pavement cracking.

The objective of the study described herein is to develop and validate creep and dynamic test methods using indentations with a flat-ended cylindrical punch. Flat-ended punches provide a well-defined contact area and therefore allow for the simple and accurate measurement of creep stiffness, slope of the creep stiffness master curve, complex modulus, phase angle, and other properties of interest [9, 10]. The work described encompasses low temperature creep and dynamic tests on recovered asphalt cements from seven test sections of a six year old northern Ontario pavement. Further work will involve the development of improved chemical aging techniques, which, in combination with the new test methods, will allow us to better control pavement cracking distress.

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Note: Submitted October 16, 2010; Revised January 4, 2011; Accepted January 5, 2011.

Table 1. Grading Results for RTFO/PAV-Aged and Recovered Asphalt Cements [3, 6].

Section	Modification	AASHTO M320 Grades, °C RTFO/PAV Residues (2003)	Recovered Residues (2008)
655-1	RET + PPA	-36	-39
655-2	Oxidized + SBS	-36	-34
655-3	SBS	-36	-34
655-4	SBS	-35	-27
655-5	SBS	-35	-36
655-6	Oxidized	-35	-31
655-7	Control	-35	-34
		RTFO/PAV Residues (1992)	Recovered Residues (2004)
631-1	Unmodified	-33.1	-25.5
631-2	Unmodified	-33.2	-29.6
631-3	Oxidized	-36.8	-31.5
		RTFO/PAV Residues (1996)	Recovered Residues (2009)
17-02	SBS	-41	-33
17-03	SBS	-35	-29
17-60	Unmodified	-31	-26
17-61	Oxidized	-35	-34
17-62	SBS	-41	-36

Background

Documented Sensitivities and Shortcomings of Current Aging Methods

The current RTFO/PAV protocol was intended to replicate the aging process as it occurs during hot-mix production and the first 8-10 years of service. The high pressure and temperature employed in the PAV are supposed to ensure that oxygen saturates the film to produce a consistent material with properties similar to that of an 8-10 year old recovered sample within a period of only 20 hours.

However, Bahia and Anderson [2] found that the film thickness in the PAV can have a major impact on rheological properties, as measured by the viscosity at 60ºC. Their data proved that yet undetermined processes can cause significantly increased aging in thinner films, even at the high temperatures and pressures of the PAV. Increases in viscosity ranged from a low of 60 percent to over 500 percent (fivefold increase) for only a 50 percent reduction in film thickness. Earlier studies on variations in PAV aging reached similar conclusions [11].

Our research on pavement trial sections has shown that recovered asphalt cements often grade at warmer temperatures than those predicted by their corresponding RTFO/PAV residues [3, 6, 7]. Table 1 provides a summary of data obtained for several Ontario sections, showing that potential errors with the RTFO/PAV method can seriously confound research on pavement performance. Note that a 6°C deficit in the low temperature grade can reduce confidence from the intended 98% to less than 50% that a pavement is not exposed to damaging temperatures. The above findings from sensitivity and validation studies clearly demonstrate a need to develop improved aging and grading protocols. This project explores the use of modern indentation equipment to solve several of the problems that we and others have identified. A brief review of the history of indentation testing in asphalt follows.

Indentation Testing of Asphalt

Indentation tests on asphalt have a long history dating back to the late 19th century. A penetration test can be considered an extreme indentation reaching depths of up to 800 dmm (decimillimeters). While penetrating the surface of a viscoelastic solid, a needle of fixed dimensions displaces a small volume of material, and as such the test should be able to provide a measure of consistency. It has arguably provided the most successful specification criteria for asphalt in the history of road construction. In large part this is due to its ability to provide simple, rapid, and reproducible results without the need for expensive equipment.

However, several problems with using penetration tests to measure fundamental rheological properties of asphalt were recognized early on and at regular intervals thereafter (see Traxler [12] and references therein):

(1) The needle has end effects.
(2) Its surface can have various degrees of adhesion to the material, and this effect can vary with depth and penetration rate (grade).
(3) Penetration rate is thought to largely depend on the viscous properties of the material, thus creating uncertainty as to its consistency when significant elasticity is present [13].

Traxler's review of these issues concludes that "the penetrometer is an inadequate and unsatisfactory instrument for the rheological investigation of materials (e.g., asphalts) possessing complex flow and elasticity" [12].

Aside from the widely used penetration test, several other attempts have been made to develop indentation tests for asphalt utilizing varying degrees of complexity [1, 14-17]:

(1) Abdel-Moneim and Kirmser [14] described an indenter for the measurement of the asphalt's viscosity at room temperatures. The authors used dimensional analysis to develop an equation for the determination of absolute viscosities in asphalt films confined between relatively large, flat-ended indenters of varying size and the bottom of the sample container. Typical impressions ranged from 1 mm to 4 mm in a 25 mm layer of asphalt.
(2) Anderson *et al*. [1] described stress relaxation experiments in asphalt cements at temperatures ranging from -30°C to 5°C by using sphere indentations to depths from 0.1 mm to 0.3 mm. The relaxation data obtained matched those from more conventional test methods, such as DSR.
(3) Stangl *et al*. [15] and Jäger *et al*. [16] published a series of nanoindentation studies employing a Berkovich tip to probe the morphology and various viscoelastic parameters in creep on asphalt cement films over a range of temperatures (-15°C to 5°C) at relatively short loading times (< 10 s).
(4) Tarefder *et al*. [17] published hardness and elastic modulus data for asphalt cement, mastic, and concrete using spherical and Berkovich nanoindenter tips at room temperature. The asphalt cement results were likely confounded by the very low load and displacement levels. Tests on asphalt mastic and concrete provided more meaningful results, likely because of the higher

load levels employed (10 mN-20 mN) and displacements of up to a few micrometers.

Modern indentation techniques have developed rapidly in recent years [9, 10, 18, 19]. It is now accepted that, with properly designed experiments, indentation tests can provide results that are identical to those obtained with conventional techniques such as DSR [10]. For that reason, we have embarked on this study with the ultimate aim of developing better performance grading tests. We have begun an effort to develop exceedingly simple tests using flat-ended punch indenters. By using flat-ended punches with well defined contact areas and loads in both static and dynamic tests, as well as by restricting the maximum indentation depth to a fraction of that achieved in a regular penetration test, we anticipate that meaningful rheological properties can be obtained.

Materials and Methods

Materials

The materials tested in this study were carefully extracted from field core samples taken from a 2003 pavement trial on Highway 655 just north of Timmins, Ontario. The trial was designed so that all seven asphalt cements aged according to standard RTFO and PAV procedures and were graded in the bending beam rheometer (BBR) at between -35°C and -36°C (see Table 1) [3, 6]. This is just above the -37°C needed to obtain a 98% confidence level to ensure that no damage will occur in any given winter for the Timmins, Ontario, area [3, 6].

The construction of the trial sections was governed by the end result specification system (ERS) of the Ministry of Transportation of Ontario (MTO). The contractor obtained a bonus for the quality obtained, and no notable issues arose regarding any of the measured variables under the ERS system. Voids, voids in the mineral aggregate (VMA), voids filled with asphalt (VFA), lift thickness, fines, and asphalt cement content all fell within the narrow range of acceptable limits. Further details on materials and the design of the trial are given in previous publications [3, 6].

Asphalt extraction was done with tetrahydrofuran (THF). Tetrahydrofuran was selected because it is an excellent solvent for the reactive ethylene terpolymer (RET) and styrene-butadiene (SB) elastomers used in this trial. The solvent was stabilized with 0.025% butylated hydroxytoluene (BHT). The bulk of the solvent was removed at temperatures below 50°C and pressures of 100 mbar - 500 mbar. Final traces of solvent and BHT were removed at low pressures and high temperatures (20 mbar - 40 mbar and 160°C). Infrared spectroscopy was used to make sure that the extraction and recovery did not change the properties of the asphalt cement and that all solvent and stabilizer were removed.

Methods

Asphalt cements were cast in aluminum trays equipped with precisely machined troughs of known depth, permitting the creation of 200 micron, 400 micron and 800 micron thin films. A photograph of a tray is provided in Fig. 1(a). Asphalt samples were dissolved in toluene at roughly a 2:1 ratio by mass and transferred into the troughs by means of a pipette. Once the solvent had evaporated, they were transferred to an ice bath, and a razor blade was run along each trough to ensure the specimens were flush with the aluminum surface. Prior to testing, all samples were placed inside a vacuum oven to eliminate remaining traces of solvent.

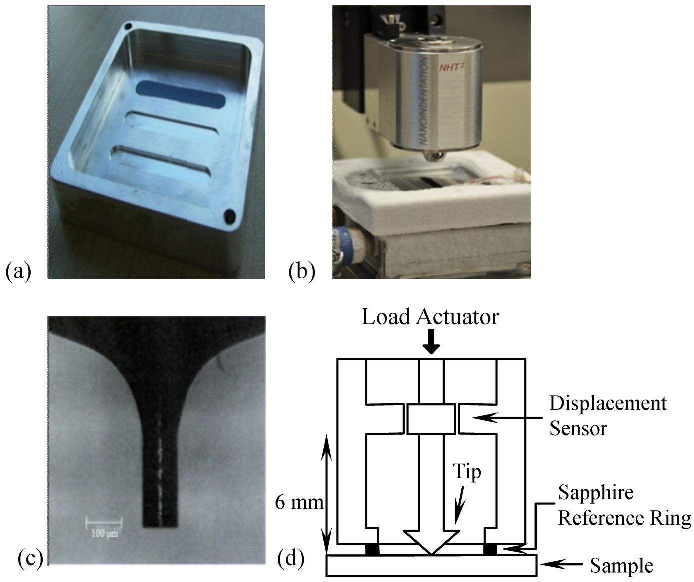

Fig. 1. (a) Sample tray with the 200 micron trough filled with asphalt; (b) Indenter module positioned above the frosted cold stage; (c) Optical image of the 100 micron flat-ended punch; and (d) Schematic of the internal configuration of the CSM NHT^2 indenter head.

All specimens were tested on a model NHT^2 nanohardness testing system developed by CSM Instruments of Peseux, Switzerland. The indenter module and the 100 micron flat-ended punch are shown in Figs. 1(b) and 1(c), respectively. The specimen trays were fit into a cooling stage and brought to the desired temperature by means of a Peltier thermoelectric cooling plate. Ethanol was poured into the trays to prevent the formation of ice, and a thermocouple was kept as close as possible to the test site to regulate the temperature to an accuracy of 0.2°C.

The sample trays were designed to provide a sound surface for the indenter module's reference ring to rest upon, thereby eliminating the possibility of distortion due to the sinking of the module – and thus the "zero" reference for penetration depth – into the asphalt sample during testing. The reference ring is a feature that, aside from providing a reliable and consistent "zero" reference, eliminates the detrimental effects of thermal drift and aids in the stability of measurements [10, 20]. A schematic of the internal configuration of the indenter, including the sapphire reference ring, is shown in Fig. 1(d) [20].

Initial creep tests were conducted with different load levels to establish the range of linear viscoelasticity, and creep compliance was found to be invariant of load for penetration depths of 25 microns and less. A testing protocol was established to ensure indentations would remain within the linear viscoelasticity regime. To more closely emulate the BBR grading test, efforts were made to apply the creep load as quickly as permissible by apparatus

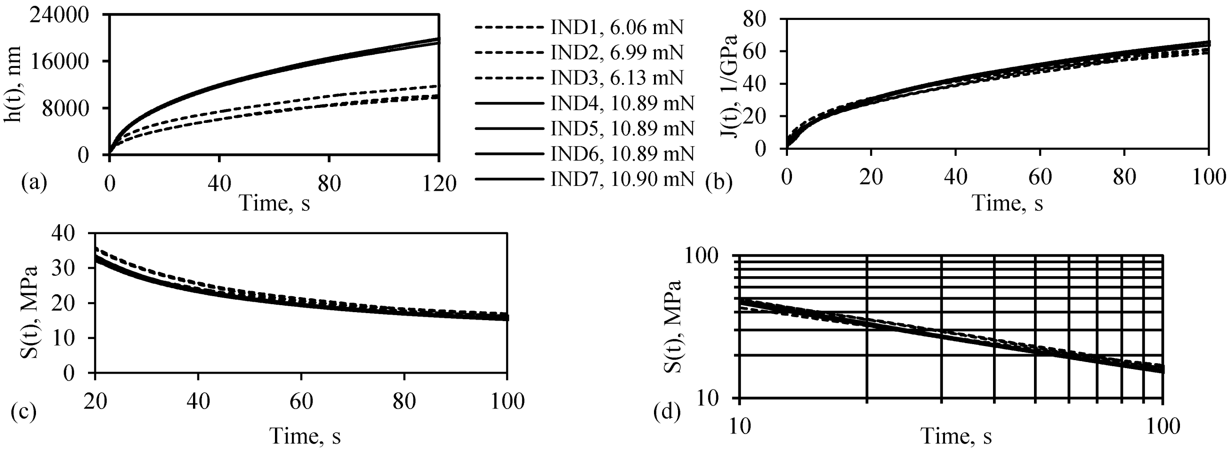

Fig. 2. Typical indentation creep results obtained from a 200 micron thin asphalt cement film at -14°C: (a) Raw data at different creep loads; (b) Compliance versus time at different creep loads; (c) Stiffness versus time at different creep loads; and (d) Creep stiffness master curve (i.e., log-log plot of Fig.2(c)).

limitations. Maximum levels were established within two seconds of loading and were held constant for a period of two minutes. This was followed by a 30 second unload to a level suitable for relaxation, which was then maintained for a duration of four to six minutes with every other indent. Dynamic tests were conducted at a frequency of 0.5 Hz with a target sinus amplitude of 4 mN. The actual amplitude increases to the target level during the first few microns of indentation.

All specimens were conditioned for 30 minutes prior to testing. Typically, two grids of four indentations were made at two different areas along the specimen length, followed by a single grid of dynamic indentations at a third site. A minimum spacing of 1 mm was maintained between each indent within a grid, and indents were centered in the test trough to eliminate the possible influence of the trough walls. All indentations were made within two hours of the end of the 30 minute conditioning period.

The 2008 distress surveys for all seven test sections were conducted over 50 m stretches at the beginning, middle, and end of each 500 m section of Highway 655, north of Timmins, Ontario. Distress in 2008 is expressed as linear crack length per 500 m of road. For the 2010 survey, transverse cracks were classified as quarter lane, half lane, full lane, or full width. The width of the pavement was measured in order to calculate approximate cracking lengths for each test section. The crack counts and lengths provided in the Results and Discussion section are approximate indicators of distress for 150 m of each 500 m section. Further details are provided in previous publications [3, 6].

Results and Discussion

Linear Viscoelasticity and Reproducibility in Creep Testing

Linear viscoelastic conditions were ascertained by testing at different load levels (indentation rates) and depths. Typical experimental creep results are provided in Fig. 2. The raw penetration data were first corrected for load-frame compliance by the instrument software, and the corrected penetration (denoted as h(t)) was used in subsequent transformations. Creep stiffness (denoted as S(t)) was taken to be the inverse of creep compliance, and the data beyond 20 seconds were fit to an equation of the form $S(t) = A + B\log(t) + C[\log(t)]^2$, again with the intent of simulating the BBR method. Prony series were also considered as a fitting option for creep compliance data. However, this introduced unnecessary difficulties and was much less amenable to reliable fitting algorithms. The values of creep compliance [J(t)] and creep stiffness [S(t)] were calculated using a reference length of 400 microns (the trough depth) at 60 seconds of loading. Since the creep rate is nearly constant by this time, the slope of the stiffness curve can be calculated to give a measure of how the creep stiffness changes with loading time. The m-value, a grading parameter used in the BBR test, was also calculated based on its definition as the slope of the stiffness master curve:

$$m(t) = \frac{d(\log[S(t)])}{d(\log(t))} \tag{1}$$

The extent to which film thickness influences creep data was investigated through tests on 655-1 PAV residue cast in 200 micron and 400 micron troughs. The results, as presented in Table 2, show that creep stiffness, m-value, and other properties of interest were largely unaffected in this range, suggesting that there is little or no interaction between the flow zone underneath the 100 micron flat-ended punch and the trough bottom. Additional experiments on thinner films are planned, since this issue could potentially become relevant under more critical circumstances.

Variability in the creep experiments could be largely attributed to minor misalignments, surface contamination, and hitherto unknown instrumental instability. Misalignments and surface contaminations can spoil the instantaneous full contact that is desired with the flat-ended punch, hence confusing the contact detection from which the software calculates the creep curve. Experiments at very low temperatures were also found to be less reproducible due to electronic instabilities possibly related to the condensation of moisture within the indenter module. These instabilities occurred sporadically and have now been addressed by the instrument

Table 2. Reproducibility of Creep Properties of 655-1 PAV Residue Determined at -24°C.

Date	Thickness, m	Force, mN	Depth, m	S(60 s), MPa	m (60 s)	$W_{elastic}/W_{total}$ (120 s), %
Jun-17	400	27.7	4.59	411	0.383	7.3
Jun-29	200	26.0	3.73	468	0.365	7.8
Jun-30	400	26.0	3.74	470	0.374	8.8

Note: The depth, as measured at the end of the loading segment, is influenced by the applied load. $W_{elastic}/W_{total}$ is the work ratio calculated from the areas under the loading and unloading segments of the force-displacement curves.

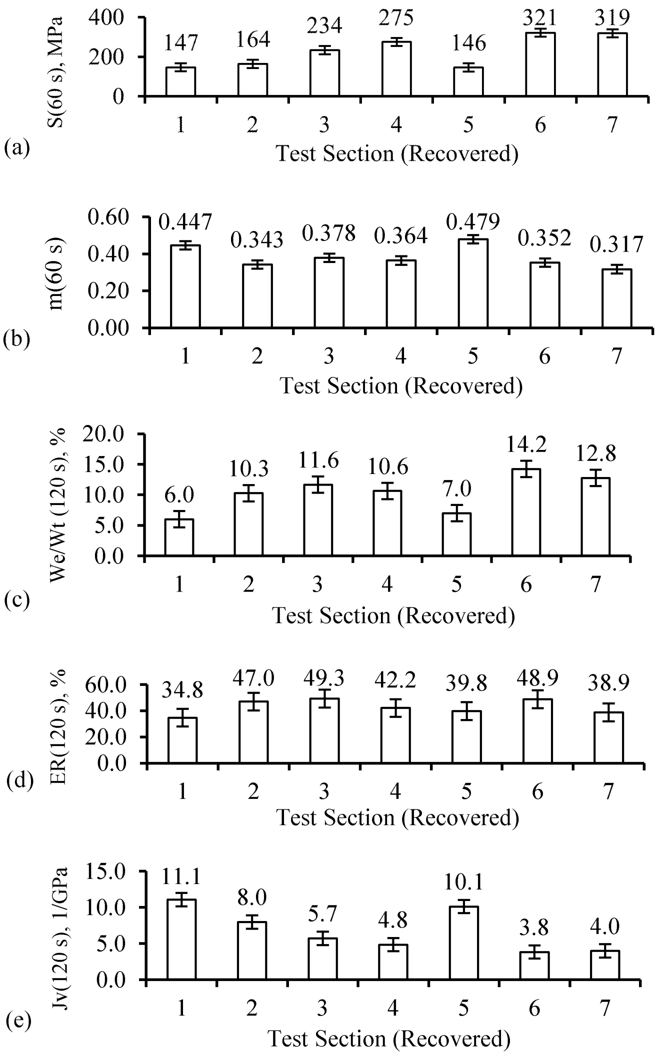

Fig. 3. Indentation creep and recovery data: (a) Creep stiffness; (b) Slope of the creep stiffness master curve (m-value); (c) Elastic versus total work of indentation; (d) Elastic recovery; and (e) Viscous compliance. Note: All tests were done at -14°C and error bars represent ±1 pooled standard deviation.

manufacturer. Nevertheless, the data, as shown in Fig. 2 and Table 2, establishes that indentation is potentially a powerful technique for the reproducible collection of creep properties in thin films at low temperatures. These findings are due, in large part, to the sapphire reference ring on the NHT[2], which is designed to address instrument and thermal drift. These issues are especially important at long loading times in creep and in the low frequency domain of dynamic tests [10, 20].

Creep Specification Grading of Recovered Asphalt Cements

The seven recovered asphalt cements from the Highway 655 pavement trial were tested according to the following protocol at -14°C:

(1) conditioning for 30 minutes at the test temperature;
(2) creep testing for 2 minutes at loads of about 15 mN; and
(3) unloading for 4-6 minutes until full recovery at a load of about 1 mN-2 mN.

We have used the recovered asphalt cements in this study to conduct a direct comparison with field performance. PAV residues were not considered since they were known to show poor correlation with performance (see Table 1). As part of our future efforts, we will be testing thin films aged under various conditions (temperature, pressure, time) to develop improved specification grading techniques. The indentation tests, as described here, will allow us to determine rheological properties on very thin films, which we believe to be more suitable for realistic chemical aging.

The displacement data recorded during the loading segment were processed according to the scheme depicted in Fig. 2 to calculate S(60 s) and m(60 s) at -14°C. The unloading segment of each curve was used to calculate the elastic to total work ratio, the percentage elastic recovery, and the absolute viscous compliance after 120 seconds of loading at low temperatures, such as $W_{elastic}/W_{total}$ (120 s), ER(120 s), and J_v(120 s), respectively. The findings for this series of experiments are provided in Fig. 3. These graphs show that there are significant differences among the seven asphalt cements and that most of the properties evaluated provide similar rankings.

Dynamic Testing of Recovered Asphalt Cements

The seven recovered asphalt cements from the Highway 655 pavement trial were further tested in a dynamic mode. Fig. 4 provides typical raw and processed data for a dynamic indentation. Under dynamic testing, the displacement response of the material can be expressed as:

$$h(t) = h_0 \sin(\omega t + \delta) \tag{2}$$

where h_0 is the amplitude of displacement, ω is the driving frequency, and δ is the phase angle (time lag) between the applied force and the material response. The viscoelastic behavior of the specimen may be expressed in terms of the loss and storage moduli, which together constitute the complex modulus. These values are computed by the indentation software according to the following equations:

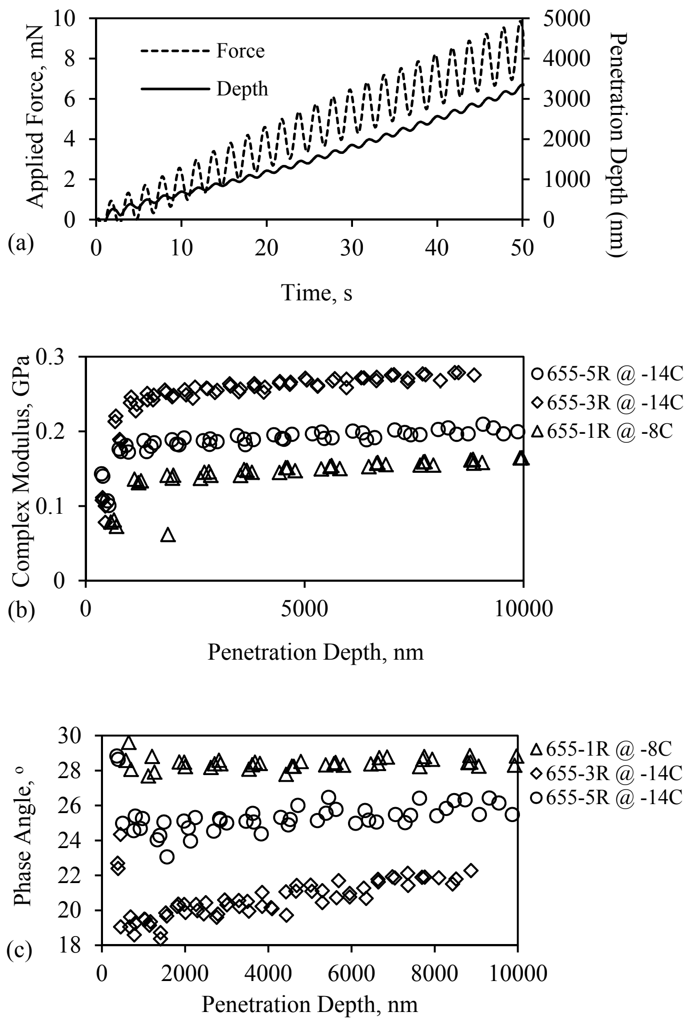

Fig. 4. Typical dynamic indentation results: (a) Raw force and displacement traces; (b) Complex moduli as a function of indentation depth; and (c) Phase angles as a function of indentation depth. Note: (a) shows a single test result obtained on a 400 micron film while (b) and (c) show four replicates each for three different recovered asphalt cements.

$$E^* = E' + iE'' = \frac{f_0}{h_0}\left(\frac{\sqrt{\pi}}{2\sqrt{A}}\right)\cos\delta + i\frac{f_0}{h_0}\left(\frac{\sqrt{\pi}}{2\sqrt{A}}\right)\sin\delta \tag{3}$$

where E^*, E', and E'' are complex, storage, and loss modulus, respectively; A is the contact area; and f_0 is the amplitude of the applied force. A more in-depth overview of the analysis of dynamic indentations may be found in the literature [10, 18].

As a general trend, the results show slight increases in both phase angle and complex modulus with increased penetration depth, as seen in Fig. 4. This may have been due to the same issues that confound the historical penetration test (i.e. adhesion of the material to the sides of the indenter, thus changing the effective contact area). However, the data in Fig. 4 show that the effect is relatively modest for slight indentations, and differences between asphalt cements are more significant than those for any given asphalt within the first 2 microns to 10 microns. Fig. 4 also shows that a minimum indentation depth of roughly one to two microns was required to achieve full contact and stabilized results. Compared to the static creep data, the dynamic test results were generally found to be more reproducible.

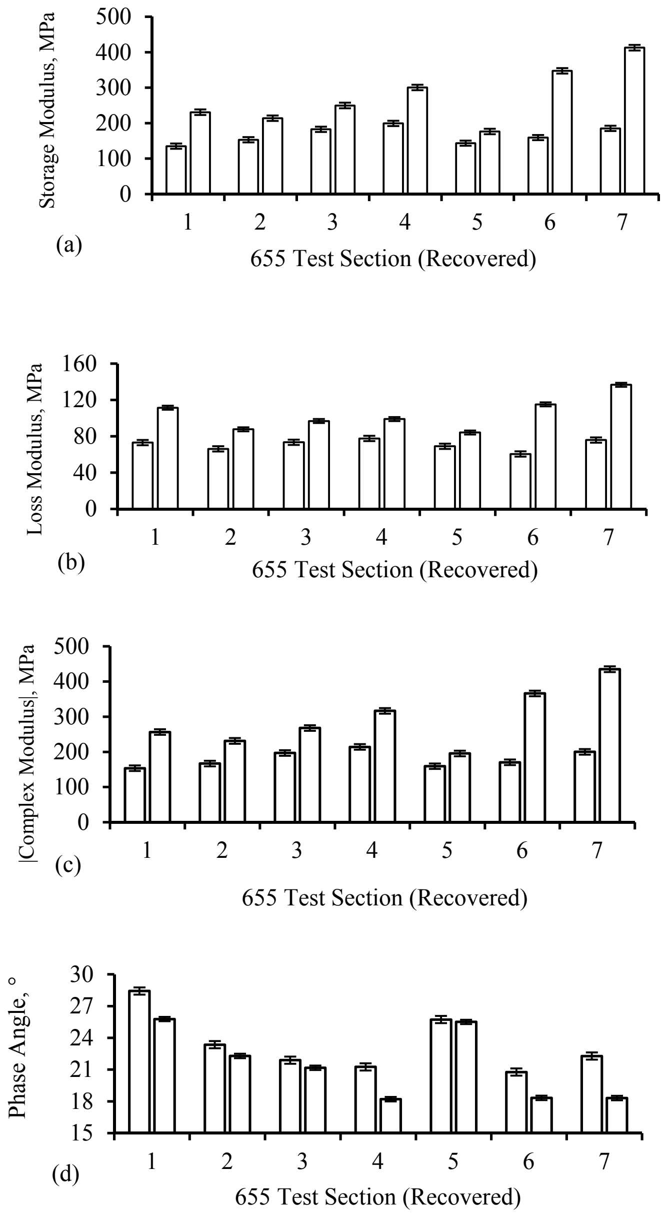

Fig. 5. Dynamic rheological data for recovered binders at 0.5 Hz and -8°C (first column) and -14°C (second column): (a) Storage moduli; (b) Loss moduli; (c) Complex moduli; and (d) Phase angles. Note: Error bars reflect ± 1 pooled standard deviation.

This is likely due to the uncertainty in the contact detection for creep tests. The dynamic rheological data were averaged between 4 microns to 8 microns of indentation depth, and the findings for all asphalt cements at -8°C and -14°C are presented in Fig. 5. The data, as summarized, demonstrate that indentation is also a very powerful technique for the reproducible collection of dynamic properties in thin films at low temperatures.

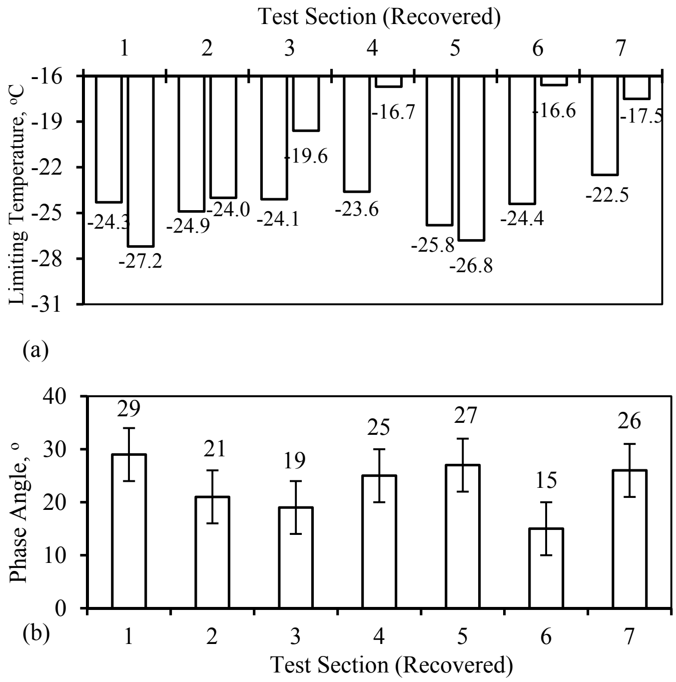

Fig. 6. Conventional grading of asphalt cements recovered from 2009 core samples: (a) BBR and (b) DSR at -14°C. Note: Limiting stiffness temperatures are provided in the first column and limiting m-value temperatures in the second column. Error bars on phase angles provide ± 1 pooled standard deviation. Significant errors in DSR data show that at this temperature the machine is at the limit of its capabilities with 8 mm parallel plates.

Comparison between Indentation, Conventional Grading, and Field Distress Data

The BBR and DSR results for the asphalt cements recovered from 2009 core samples are provided in Fig. 6. Field performance of the trial sections is reflected by the thermal distress data in Fig. 7.

These graphs show that the rankings, as produced by the various indentation properties (Figs. 3 and 5), largely match those for the BBR limiting temperature (Fig. 6(a)) and DSR phase angle (Fig. 6(b)). This comparison shows that the indentation test is able to determine meaningful rheological properties on very thin asphalt films over a range of temperatures.

Depending on how the cracking survey results are presented (e.g. transverse crack lengths, longitudinal crack lengths, transverse crack counts, etc), the sections' performance is ranked in slightly different orders. The 2008 and 2010 surveys rank the sections in a slightly different order due to differences in pavement age (note: the rather significant changes in 2008 [Table 1] and 2009 limiting BBR temperatures), survey lengths, and the criteria that were used to count cracks. Results from 2008 are different from those in 2010 in part due to a significant number of shorter cracks connecting to form fewer larger ones. Hence, in our opinion, it is best to separate the performance into the following three categories:

(1) Sections 1 and 5 are performing as expected/desired, with Section 1 doing slightly better;

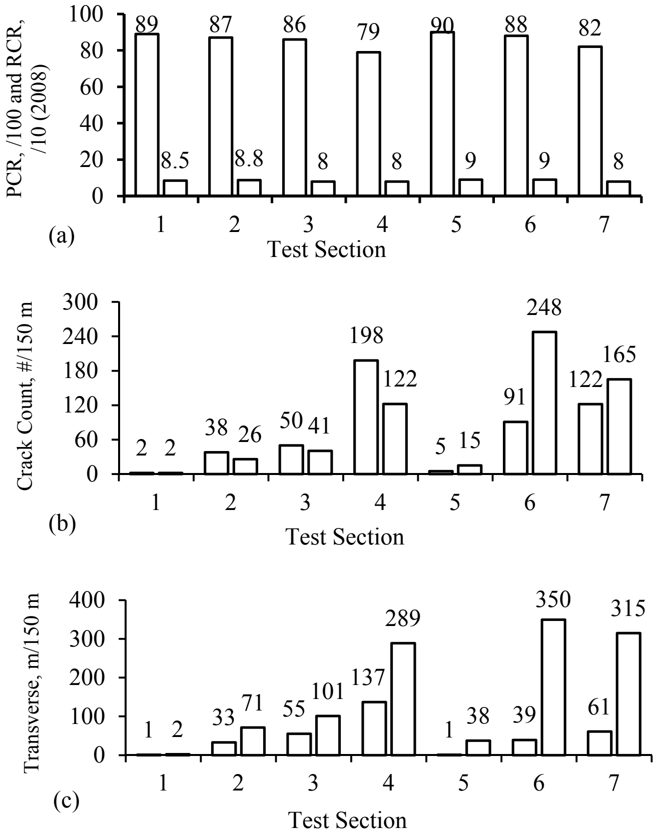

Fig. 7. Pavement distress indicators: (a) Pavement condition rating (PCR) and Ride comfort rating (RCR) indices for August 2008 [6]; (b) Approximate transverse crack counts for 2008 (first column) [6] and 2010 (second column); and (c) Approximate transverse crack lengths for 2008 (first column) [6] and 2010 (second column).

(2) Sections 2 and 3 are cracked significantly more than desired; and

(3) Sections 4, 6, and 7 are cracked excessively for a seven year old trial.

A more detailed analysis of the cracking data is likely not justified due to the inherent difficulties associated with cracking distress surveys. In years to come, the picture will become clearer with Section 1 likely surviving largely free of distress and the others deteriorating to states requiring early reconstruction before they reach their design life. Section 5 may also remain in fairly good shape, although the base asphalt was found to be prone to physical hardening, which will eventually show up as increased thermal distress [6].

The findings from DSR, BBR, and indentation test methods agree well with the performance rankings presented in Fig. 7. Asphalt cements used in Sections 1 and 5 appear to perform best in service, which is reflected in the grading test results (low stiffness, high m-value, low elasticity, and high phase angle). Materials used for Sections 4, 6, and 7 appear to perform worst, both in service and in the grading tests (high stiffness, low m-value, high elasticity, and low phase angle). The creep stiffness [S(60 s)], viscous creep compliance [J_v(120 s)], and dynamic phase angle [δ(0.5 Hz)] in indentation are slightly better than the m-value [m(60 s)] at ranking the performance of this set of seven asphalt cements. However, this result may be due to the samples having been conditioned for only 30 minutes at -14°C prior to testing. Physical hardening effects during longer conditioning periods will likely improve the match between the ranking in m(60 s) (Fig. 3(b)) and the ranking in field performance (Fig. 7) [3-7]. It is somewhat remarkable that the indentation stiffness [S(60 s)] does a significantly better job than the BBR [S(60 s)] at ranking the performance of these seven materials. Limiting BBR stiffness temperatures for these recovered materials were determined and fell within a narrow range of -22.5°C to -25.8°C. Further work on the effects of conditioning time on the indentation properties will be needed to shed light on these uncertainties.

In contrast to the results of the recovered materials, the current specification tests on RTFO/PAV-aged materials rank all of these asphalt cements at either -35°C or -36°C. Thus, they should have survived the cold spell in January 2004, during which the surface temperature fell to -34°C on two occasions. It is therefore reasonable to conclude that it would be possible to more reliably and precisely select asphalt cements to ensure their maximum durability, provided that the current RTFO/PAV aging and low temperature conditioning protocols can be improved. This issue is the focus of ongoing investigations by the authors, and we hope that the newly developed indentation tests will provide much needed tools to bring this effort to a successful conclusion.

Conclusions

Given the results presented in this paper, the following summary and conclusions are provided:

- Modern instrumented microindentation tests are able to provide routine rheological testing data on much smaller samples yet maintain the accuracy and precision analogous of those produced with BBR and DSR equipment.
- Indentation creep properties appear to correlate well with the observed variability in pavement performance for the seven recovered asphalt cements, as investigated in this study. This contrasts with the BBR grading on RTFO/PAV-aged material, which ranked all sections in a narrow range of -35°C to -36°C.
- Dynamic indentation tests appear to provide accurate and reproducible rheological properties. The low temperature indentation phase angle, which is a measure of the asphalt cement's ability to relax thermal stress, was found to correlate well with pavement cracking distress.

Given the encouraging findings of this study, the time seems to have come to explore new thin film aging protocols to produce asphalt cements with properties that better reflect those of materials recovered from 8-10 year old pavements.

Acknowledgments

The authors wish to thank E.I. du Pont Canada, Imperial Oil of Canada, the Ministry of Transportation of Ontario, and the Natural Sciences and Engineering Research Council of Canada for their continuing financial support. Appreciation is expressed to Eric Moult and Syed Rubab for their assistance with the BBR and DSR testing of the recovered asphalt cements and to the staff of the Northeastern Region Office of the Ministry of Transportation of Ontario for their help with the collection of cracking distress information from this trial. Further appreciation is expressed to Maureen Garvie, Sogol Borjian and Ryan Eveleigh for their help with the proofreading of this manuscript.

Disclaimer

None of the sponsors necessarily concurs with, endorses, or has adopted the findings, conclusions or recommendations, either inferred or expressly stated in the subject data developed.

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