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**Innovations Deserving  
Exploratory Analysis Programs**

***Highway IDEA Program***

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*Development of an Improved Asphalt Binder Specification  
Testing Approach*

Final Report for Highway IDEA Project 104

Prepared by:  
Simon A. M. Hesp, Queen's University, Kingston, Ontario

***August 2006***

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**TRANSPORTATION RESEARCH BOARD  
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*FINAL REPORT*

# **Development of an Improved Asphalt Binder Specification Testing Approach**

*Part I. Testing for Reversible Ageing*

*Part II. Asphalt Binder Failure Testing*

*Part III. Asphalt Mixture Testing  
and Field Validation*

**August 2006**

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# Authors, Acknowledgements, and Disclaimer

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This report was prepared by the principal investigator, Dr. Simon A. M. Hesp, at Queen's University in Kingston, Ontario.

Financial support for this work from DuPont Canada, Imperial Oil of Canada, McAsphalt Industries, the Ministry of Transportation of Ontario through the Highway Infrastructure Innovations Funding Program (HIIFP 2003-2005), the Innovations Deserving Exploratory Analysis Program (IDEA 104) as administered by the National Cooperative Highway Research Program, and the Natural Sciences and Engineering Research Council of Canada is hereby gratefully acknowledged.

The experimental data documented in this report were obtained under the supervision of Professor Hesp by graduate students Adrian Andriescu, Serban Iliuta and Michael Ou Zhao and undergraduate students Thomas Bodley, Michelle Edwards, and Daniel Bodley. Special thanks go out to Wayne Kelly and Jim Pretty of the Ministry of Transportation of Ontario for collecting some of the data for binders from Highway 655 test sections.

Ludo Zanzotto of the University of Calgary, Chuck McMillan of Alberta Transportation, and Sirous Alavi of Sierra Transportation Engineering are gratefully acknowledged for making the original C-SHRP binders from the Lamont, Alberta test road available.

Liaisons for the Ontario Ministry of Transportation were Pamela Marks and Kai Tam, while for the NCHRP-IDEA program these were Edward Harrigan and Inam Jawed. Further guidance and support was kindly provided by Lyle Moran of Imperial Oil of Canada and Gregg Babcock of the E.I. du Pont Company.

None of the sponsoring agencies necessarily concur with, endorse, or adopt the findings, conclusions, or recommendations either inferred or expressly stated in subject data developed in this study.

# Foreword

by Simon Hesp,  
Principal  
Investigator

August 1, 2006

This report presents the progress obtained towards the development of an improved asphalt binder specification for low temperature and fatigue failure in asphalt pavements.

The research effort has delivered three new laboratory test methods for the improved performance grading of asphalt cements. The first, *LS-308 Determination of Performance Grade of Physically Aged Asphalt Cement Using Extended Bending Beam Rheometer (BBR) Method*, has been published in the Laboratory Testing Manual of the Ministry of Transportation of Ontario and has gone through a successful round-robin study in industry. The extended BBR method as described in LS-308 is expected to significantly reduce transverse cracking distress in years to come. Most of the research that contributed to the development of LS-308 is described in *Part I. Testing for Reversible Ageing* of this final report.

The second method is focussed on fatigue failure and is published as *LS-299 Asphalt Cement's Resistance to Fatigue Fracture Using Double-Edge-Notched Tension Test (DENT)* in the Laboratory Testing Manual of the Ministry of Transportation of Ontario. The method measures the resistance to ductile failure and has shown a reasonable correlation with early distress in a new Ontario pavement trial. The Ministry of Transportation of Ontario has implemented LS-299 in its own laboratories and is soon expected to conduct a round-robin study with industry. It is expected that the method will help to better control fatigue cracking distress in future pavements once the method has been further validated. The research that contributed to the development of LS-299 is described in *Part II. Asphalt Binder Failure Testing* of this report.

A third method based on compact tension (CT) testing of asphalt binders is yet to be published as LS-296. The main principles behind the method have been presented at the 2006 Transportation Research Board Meeting and will be published in the Transportation Research Records (Edwards and Hesp, TRR 2006). Once the method is implemented it is expected that it will also help to better control fatigue and low temperature cracking distress in future pavements. The research that contributed to the development of LS-296 is described in *Part II. Asphalt Binder Failure Testing* of this report.

The project work was divided into distinct tasks that were documented and discussed as they were completed. The intent was to provide the sponsoring agencies with a "work-in-progress" document, which by the summer of 2006 was combined into this report constituting three parts.

The development of the three new test methods has been supported by the Ministry of Transportation of Ontario with the construction of three major pavement trials.

## Foreword

by *Simon Hesp,*  
*Principal*  
*Investigator*

*August 1, 2006*

The first trial was constructed in 2003 on a portion of Highway 655 located 60 km north of Timmins, Ontario. Most of the data in this report relate to the materials used in the seven test sections of this first trial. The second trial was constructed in the summer of 2006 on a portion of Highway 417 just east of Ottawa, Ontario. It has seven test sections and will help to better understand how to control reflection cracking. A third trial with eight test sections has been approved for Highway 655 and will be constructed in 2007. It will focus on the use of heavily modified asphalt binders to reduce low temperature and fatigue distress. Once the third trial is completed, this construction effort will provide a total of 22 test sections approximately 11 km in length made with a wide variety of modified and straight binders. It is likely that transverse and wheel path cracking will be better controlled once the new test methods are implemented and the trials are used to set appropriate limits associated with the specification criteria developed through this project.

The effort described in this report was jointly funded by DuPont Canada, Imperial Oil of Canada, McAsphalt Industries, the Ministry of Transportation of Ontario, the National Cooperative Highway Research Program through their Innovations Deserving Exploratory Analysis Program (IDEA 104), and the Natural Sciences and Engineering Research Council of Canada.

Part of this work has already been published, or has been accepted for publication, in the peer-reviewed scientific literature (Iliuta et al., *Proceedings, Canadian Technical Asphalt Association*, 2004, 123-158; Zhao and Hesp, *International Journal of Pavement Engineering*, Vol. 7(3), 2006, 199-211; Edwards and Hesp, *Journal of the Transportation Research Board*, Preprint Paper 06-1186, In Press, 2006), while other parts have been submitted for publication (Bodley et al., *Journal of the Association of Asphalt Paving Technologists*, Submitted, August 2006).



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**Development of an Improved Asphalt  
Binder Specification Testing Approach  
*Part I. Testing for Reversible Ageing***

**November 15, 2004**

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Publication Title	<b>Development of an Improved Asphalt Binder Specification Testing Approach Part I. Testing for Reversible Ageing</b>
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Sponsors:	Imperial Oil Limited, McAsphalt Industries, Ministry of Transportation of Ontario, National Cooperative Highway Research Program – Innovations Deserving Exploratory Analysis, Natural Sciences and Engineering Research Council of Canada
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Abstract	<p>Regular grading according to the AASHTO M320 specification, and grading after different conditioning times, was performed in order to get a better understanding of the reversible changes that occur in asphalt upon storage at cold temperatures. It was found that most binders aged rapidly by showing an increase in creep stiffness and a decrease in creep rate with conditioning time. After only three days of storage at cold temperatures, several binders lost anywhere from 9°C to 14°C from their performance grade (PG grade) while a select group of superior binders lost less than 2°C.</p> <p>The binders used for the C-SHRP trial in Lamont, Alberta were assessed for their tendency to age at low temperatures with encouraging results. While the correlation between cracking severity and regular AASHTO M320 and MP1a grades (with one hour conditioning before testing) was poor (<math>r^2 = 0.73</math> and <math>r^2 = 0.49</math>, respectively), the predictive ability of the BBR method increased significantly when binders were tested after one day of conditioning (<math>r^2 = 0.97</math>). A test section constructed in late 2003 on Highway 655 in northern Ontario, with the PG 64-34 binder that aged worst in this trial, has already started to crack significantly after a cold spell in the area during early January 2004 when the pavement temperature reached <math>-34^\circ\text{C}</math> on two occasions. The 500-m-long section showed over 70 m of cracks only months after construction. In contrast, another section constructed with a PG 64-34 binder that aged by the least amount showed no cracks. A third section for which the binder aged a moderate amount also showed no cracks, but this is believed to have been due to the fact that this binder possessed high toughness.</p> <p>For oxidized binders the ageing was found to be particularly severe. Our finding in this respect is not new in that this was first reported as early as 1936 in a paper by Traxler and Schweyer (Proc., ASTM, 1936, p. 547), and on several occasions since then by others. However, with the increasing use of oxidized binders in the paving industry in Ontario and elsewhere, it is now most important that the detrimental effects of reversible ageing at low temperatures are better recognized and taken into consideration for performance grading.</p>
Key Words	Reversible Ageing, Physical Ageing, Physical Hardening, Low-Temperature Performance Grading, Bending Beam Rheometer, Transverse Cracking



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# Executive Summary

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The purpose of this research was to assess the reversible changes that occur in straight, oxidized, and polymer-modified asphalt binders when these are stored at low temperatures and to relate these changes to field performance. Rapid structuring or ageing that occurs on a molecular level in many asphalt binders during the first few days of cold weather is believed to be an important factor that needs to be considered in the development of improved low-temperature and fatigue binder specification test methods. Since the phenomenon is most easily investigated with the bending beam rheometer (BBR samples are reusable) it was decided to use this instrument to evaluate the effects of storage temperature and time for a wide range of different asphalt binders. The ultimate goal of this research is to replace the AASHTO M320 method for low-temperature and fatigue grading of binders with a more fundamental approach which would not only account for the deleterious effects of low-temperature reversible ageing but also for the large differences in fracture properties that exist for binders of the same BBR grade.

For a number of materials a correlation between the tendency to age at low temperatures and fracture performance in service is given. Earlier research on this phenomenon dealt solely with laboratory tests and therefore provided only limited insight into how important the effect can be in terms of field performance prediction (Traxler and Schweyer (1936), Traxler and Coombs (1937), Brown et al. (1957), Brown and Sparks (1958), Struik (1978), Posadov and Rozental (1985), Bahia (1991), Bahia and Anderson (1991 and 1993), Santagata et al. (1996), Planche et al. (1998), Phillips (1999), Anderson and Marasteanu (1999), Lu and Isacsson (2000), Basu et al. (2003), Soenen et al. (2004), and others). Typical asphalt binders that are produced and sold in Canada and the United States were evaluated in order to find out what storage temperatures and times are sufficient to capture the changes so that these can be specified in an improved asphalt binder grading method.

Ten of the binders evaluated were those used for the Canadian Strategic Highway Research Program (C-SHRP) trials in Lamont, Alberta and Hearst, Ontario. These test roads were constructed in 1992 and therefore a significant amount of performance data is already available. Another seven binders investigated were used in late summer 2003 for the construction of test sections on Highway 655 just north of Timmins, Ontario. The binders that were selected for this trial were nearly identical in terms of their current AASHTO M320 low-temperature grading temperatures (continuous grades ranged from  $-34^{\circ}\text{C}$  to  $-36^{\circ}\text{C}$ ) but are expected to show a wide range in long-term performance. The remaining binders investigated were obtained from local asphalt suppliers as well as from the Materials Reference Library (MRL) of the U.S. Strategic Highway Research Program (SHRP) and represented a broad range of qualities that are available in Canada and the United States. Special care was taken to include a wide range of straight, oxidized, and polymer-modified materials.

The materials investigated behaved rather similarly in terms of their change in limiting temperatures as defined by the AASHTO M320 specification; most binders lost anywhere from 4°C to 7°C after three days of storage at cold temperatures. Superior were those straight or modified binders made with California Valley (MRL code AAG), Cold Lake (MRL code AAL and Lamont binders RR-3L and RR-7L), and Lloydminster (MRL code AAA, Lamont binder RR-6L, and 655-1) crude sources, all of which lost around 2°C. These results are in general agreement with those obtained by SHRP researchers (Bahia and Anderson (1991 and 1993)) and by others following them (Santagata et al. (1996), Planche et al. (1998), and Phillips (1999)).

In contrast, oxidized materials were generally more susceptible to reversible ageing at low temperatures with several of them losing more than 9°C (118-1, C-1 Ox, 655-2 Ox, and Lamont binder RR-1L). The finding that oxidation appears to make binders particularly prone to low-temperature ageing is not new, in that this was first reported in the 1930s (Traxler and Schweyer (1936), Coombs and Traxler (1937), Traxler and Coombs (1937)), and on several occasions since then by others (for instance, Brown et al. (1957), Posadov and Rozental (1985), and Masson et al. (2002)).

The C-SHRP binders used for the Lamont, Alberta trial were assessed for their tendency to age at low-temperatures with encouraging results. While the correlation between cracking severity and regular AASHTO M320 and MP1a grades (with one hour conditioning before testing) was poor ( $r^2 = 0.73$  and  $r^2 = 0.49$ , respectively), the predictive ability of the BBR method increased significantly when binders were tested after one and three days of conditioning at -10°C and -20°C ( $r^2 = 0.97$  and  $0.95$ , respectively).

A Highway 655 pavement test section constructed with a PG 64-34 binder that aged worst in this trial has already started to crack significantly after a cold spell in the area during early January 2004 when the pavement temperature reached -34°C on two occasions. The 500-m-long trial section showed over 70 m of low-temperature cracks only months after the end of construction in late 2003. In contrast, another section constructed with a PG 64-34 binder that performed best showed no cracks. A third section for which the binder aged significantly with a three day loss of 5°C at low temperatures also showed no cracks, but this is believed to have been because this binder possessed high toughness. Binder toughness may have limited the cracking that occurred and thus providing a way of stress relaxation without the occurrence of catastrophic failure.

The differences found in low-temperature ageing can partly explain the vast in-service performance differences that are regularly observed for binders of the same AASHTO M320 grade. A complete explanation of field performance can be provided if large differences in fracture properties for binders and/or mixtures of the same grade are also taken into consideration. The fracture properties of a number of the binders and mixtures investigated will be discussed in parts 2 and 3 of this report.

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# Introduction

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The asphalt binder specification method developed under the SHRP program, now known as AASHTO M320, employs a limit on binder creep stiffness and creep rate (AASHTO (2002)). A thin asphalt beam is cooled for one hour at the grading temperature, after which it is loaded in three-point bending to measure the stiffness at 60 seconds,  $S(60)$ , and the slope of the creep curve, commonly known as the  $m$ -value, also at 60 seconds,  $m(60)$ . The binder specification sets an upper limit of 300 MPa on the stiffness and a lower limit of 0.3 on the  $m$ -value. If a binder passes these two criteria then it passes the grading test and can be used in a particular climatic zone where the pavement surface temperature reaches  $10^{\circ}\text{C}$  below the grading temperature only once every 50 years. The philosophy behind this specification dates back to work from the 1950s and 1960s, when researchers at Shell Laboratories in Amsterdam found a reasonable correlation between the binder stiffness at a fixed loading time and various failure properties in the binder and mixture.

Van der Poel (1954, 1955) used penetration and ring and ball softening points to determine the stiffness of binders as a function of loading time and temperature. In one of his early papers, he showed that the failure conditions in the Fraass test could be related to the binder stiffness reaching a critical value after 11 seconds of loading. Based on Van der Poel (1954, 1955) and other unpublished work at Shell Laboratories, Krom and Dormon (1963) were the first to present a binder specification scheme that limits the binder stiffness at specific loading times and temperatures to control cracking due to traffic ( $t = 10^{-2}$  s and low temperatures) and thermal stresses ( $t = 10^4$  s and low temperatures).

Heukelom (1966) went further and tested a wide range of binders for which he found there to be a high correlation between binder stiffness and actual failure properties. Hence, it was suggested that the stiffness, which had become relatively easily accessible through Van der Poel's nomograph, was a good surrogate for the failure properties. Heukelom (1966) concluded that "Van der Poel's stiffness concept has provided a valuable means of simplifying the description of, not only rheological, but also fracture properties of asphalt cements and asphalt mixtures." Following this early work at Shell Laboratories, a large number of other researchers have thus focussed their attention on stiffness as a binder specification parameter at low temperatures (e.g., Hills and Brien (1966), McLeod (1968), Fromm and Phang (1970), Readshaw (1972), Hills (1974), Deme and Young (1987), Anderson et al. (1994) and others).

It is not widely recognized, however, that the correlation made by Heukelom (1966) was only valid for unmodified binders and that there was a fair degree of scatter. While modifiers were used only sparingly in the 1960s, today the situation is different, in that in some areas nearly half of all binders are modified (air blown, polymer modified, gelled, "engineered," etc.) (Bardesi et al., 1999). Different modification techniques result in asphalt binders with vast differences in fracture properties in both the brittle state (e.g., Lee and Hesp (1994), Lee et al. (1995), Hoare and Hesp (2000), Anderson et al. (2001) and others) as well as the ductile state (Andriescu et al.



(2004a and 2004b)). Such differences in fracture properties are now believed to explain in part the vast in-service performance differences that are found for binders of the same AASHTO M320 grade (Iliuta et al. (2004a and 2004b)). A detailed discussion of this issue will be provided in a subsequent report.

A second and perhaps equally important factor on which the early low-temperature studies were largely silent is the fact that almost all binders show some degree of reversible structuring or ageing when stored at cold temperatures. Although this phenomenon had regularly been discussed in publications from the 1930s (Traxler and Schweyer (1936), Traxler and Coombs (1937)), 1950s (Brown et al. (1957), Brown and Sparks (1958)) and 1970s (Struik (1978)), apparently the publications by Van der Poel (1954, 1955) and Heukelom (1966 and 1969) and those following them make only indirect mention of it. Heukelom (1973) divided asphalt into three subclasses: Type S for bitumens that rendered a straight line on his bitumen test data chart; Type B for blown bitumens; and Type W for waxy bitumens. It is unclear if this was done to address the fact that both the waxy and blown bitumens are particularly prone to the reversible structuring processes that were described by others and therefore failed to give straight lines in Heukelom's bitumen test data chart, or if there were other reasons.

Furthermore, the approach proposed by the Shell researchers was totally dependent on surrogate properties measured at ambient and high temperatures (penetration, penetration index, ring and ball softening point) to obtain the stiffness (also a surrogate property) to predict failure properties (failure strain) at much lower temperatures and higher stiffness. Most of these studies were done in the laboratory, and therefore it is difficult to determine the veracity of many of the proposed specification schemes.

Today there are some companies that sell wax modifiers to reach a certain AASHTO M320 performance grade and there are many that oxidize binders to increase their grade. Both modification approaches raise serious concerns with regard to the reversible ageing process and how it negatively affects performance. This development makes asphalt binder grading more challenging but, as will be discussed later, there are ways to deal with the reversible structuring processes that occur during cold storage.

A detailed review of the reversible ageing studies since 1936 follows, as well as a discussion of our own experimental work on the development of an improved binder specification that accounts for the reversible ageing effect in a practical manner.

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# Background on Reversible Ageing

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## EARLY STUDIES

The *reversible ageing* term is used here to describe processes that have variously been described with the terms *age hardening* (Traxler and Schweyer (1936)), *steric hardening* (Brown et al. (1957)), *physical ageing* (Struik (1978)), and *physical hardening* (Bahia (1991)) and that have a similar effect on performance properties as those that occur at high temperature due to volatilization and oxidation. Asphalt binders, when stored at cold temperatures, go through slow molecular changes making the material stiffer and less able to relax stresses. An important difference is that structural changes are reversible by simply heating the material, whereas chemical changes are permanent.

The first in-depth study of the reversible ageing process in asphalt was published by Traxler and Schweyer (1936) although others had much earlier recognized the phenomena. The authors measured the viscosity of 10 asphalt materials of different origin and manufacturing method after leaving these undisturbed for various periods at room temperature (25°C). It was reported that the asphalt materials age hardened at different rates, that reheating would bring the samples to their original state, and that the addition of filler had little effect. From these experimental observations it was concluded that the process they called *age hardening* was primarily due to a thermally unstable structure within the asphalt.

It is interesting to note that they observed the fact that air-blown asphalt exhibits a strong tendency to age:

“Data for asphalt J, which is an air blown bitumen are given in Table II but are not plotted. This asphalt exhibited an abnormally fast rate of time-hardening which necessitated in the later experiments the use of a shearing stress which was higher than that used with the other asphalts.” (Traxler and Schweyer (1936, p. 547))

In a subsequent paper, a series of five different air-blown bitumen were discussed and the authors come to the following conclusions:

“The amount of permanent hardening due to volatilization or chemical reaction is small in comparison with the increase in viscosity due to the structure which develops with time and which may readily be destroyed by heat. In general, air blown asphalts show greater rates of age-hardening than steam or vacuum distilled asphalts.” (Coombs and Traxler (1937, p. 294))

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In yet another paper by the same authors on the same subject, the following comments are made:

“The more highly blown an asphalt, the more rapid is the increase in consistency with time. The rate of age hardening may be considered as a sensitive measure of differences in degree of internal structure.” and “The explanation offered for the increase in consistency with time is that a gradual isothermal sol-gel transition takes place, the magnitude of which depends on the source and method of processing.” (Traxler and Coombs (1937, p. 552))

Interestingly, much of this knowledge appears to have been lost for many in the asphalt industry, as judged by the growing use of air-blown asphalt.

After a somewhat extended period during which the topic received little attention, Brown and coworkers at the Standard Oil Company in Indiana investigated the reversible ageing phenomenon for four different binders ranging from a hard air-blown roofing asphalt to a soft cutback grade (Brown et al. (1957)). The study proposed the term *steric hardening* to distinguish the process from the reversible hardening that occurs due to the effects of deformation as in strain hardening. The research assessed the effect by measuring the creep strain under a constant tensile load at a fixed time at either 22°C (for roofing, industrial, and paving asphalt) or 2°C (for cutback asphalt). The authors noted that the conditions selected were those that were most convenient, but that the general process occurs under a much wider range of temperatures, times, and test conditions.

Brown et al. (1957) describes the hardening process as follows:

“To produce these hardening effects, some structure must develop or, at least, some molecular reorientation must occur in the asphalts with time. This may result either by the molecular species assuming a new equilibrium condition of statistical configuration characteristic of the lower temperature or else by increased association or solvation of these molecules to bulkier complexes.” (Brown et al. (1957, p. 491))

This description suggests that the collapse of free volume due to a lowering of the temperature as well as the isothermal sol-gel transition proposed by Traxler and coworkers and perhaps wax crystallization play important roles. The authors conclude with the following advice:

“Other than the helpful annealing technique, the authors offer no solution to the problem of speeding the attainment of steric hardening equilibrium.”

and

“Use of dispersion additives to prevent asphaltene association may prove effective in shifting the equilibrium orientation to one more quickly attainable. Meanwhile, steric hardening must be taken into account in any rheological studies of complex hard asphalts.” (Brown et al. (1957, p. 494))

The importance of this last sentence to the development of an improved low-temperature asphalt binder specification will be discussed later with relevant field performance data.

Struik (1978) published a book *Physical Aging in Amorphous Polymers and Other Materials* which provides a wealth of information on the reversible ageing process in a vast range of amorphous solids including polymers, metals, sugar, cheese, and asphalt. A total of 40 different substances were investigated and it was shown that the process of physical ageing is a basic feature for a wide range of materials below their glass transition temperature. The term *physical ageing* was adopted since it distinguished the process from chemical ageing processes such as thermal and photo-oxidation.

Struik (1978) primarily focussed his fundamental and detailed studies on polymers for which the physical ageing phenomenon generally occurs between the glass transition temperature,  $T_g$ , and the highest secondary transition temperature,  $T_\beta$ . Struik's free volume approach was mainly qualitative and based on the following argument:

“When the polymer is cooled to some temperature  $T_1$  below  $T_g$ , the segmental mobility  $M$  will be small, but not zero. Since at this stage the free volume  $v_f$  is greater than it would be at equilibrium, the volume will continue to decrease slowly. This contraction will be accompanied by a decrease in the mobility,  $M$ , with concomitant changes in all those properties of the glassy polymer which depend on it.” (Struik (1978, p. 9))

Although Struik presented only few experimental results on asphalt, he came to a similar conclusion as Coombs and Traxler (1937) and Brown and coworkers (1957) on the relevance of the reversible ageing process to performance testing:

“In addition from being theoretically interesting, the phenomenon of aging is very important from a practical point of view. Several properties of glassy polymers, e.g. their small-strain mechanical properties, undergo marked changes and strongly depend on aging time. In the testing of such plastics, the aging time is just as important as other parameters such as temperature, stress-level, humidity, etc. Furthermore, a knowledge of the aging behaviour of a material is indispensable to the prediction of its long-term behaviour from short-term tests.” (Struik (1978, p. 2))

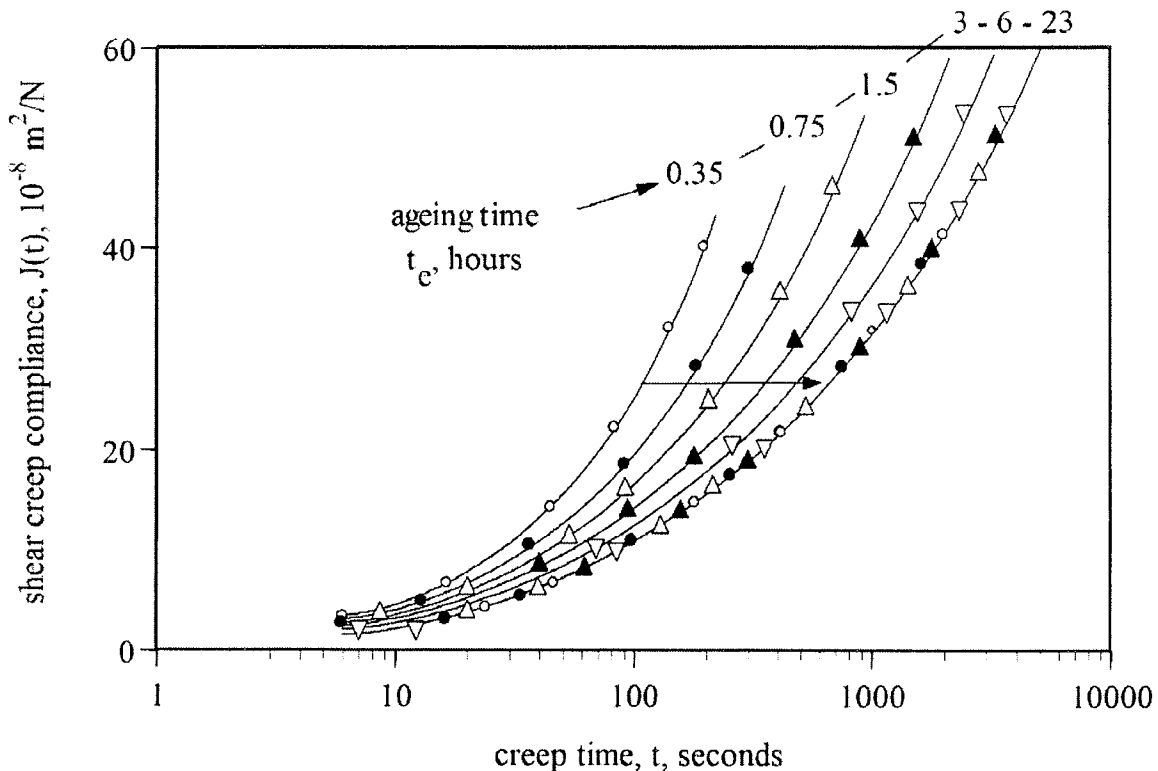
and

“It is of little use to measure creep, stress-relaxation, etc., with accurate instruments and thermostats if one ignores the aging effect.” (Struik (1978, p. 29))

The conclusions provided in the last two sentences are particularly relevant to performance grading of asphalt binders given the fact that the AASHTO M320 standard, as well as other

proposed methods, rely so heavily on time–temperature superposition.

Struik (1978) discussed his results for heavy roofing bitumen with the aid of two graphs that show the characteristic features of all materials that age reversibly. A plot of the creep compliance,  $J(t)$ , as a function of creep time for various ageing times produces a set of curves that all have the same general shape. The author recognizes that this is a common feature among all materials that age allowing individual curves to be shifted horizontally to produce a single “master curve.”



**Figure 1. Shear Creep Compliance of a Heavy Roofing Bitumen Quenched from 20°C to 0°C as a Function of Ageing Time (Reproduced from Struik (1978, p. 17))**

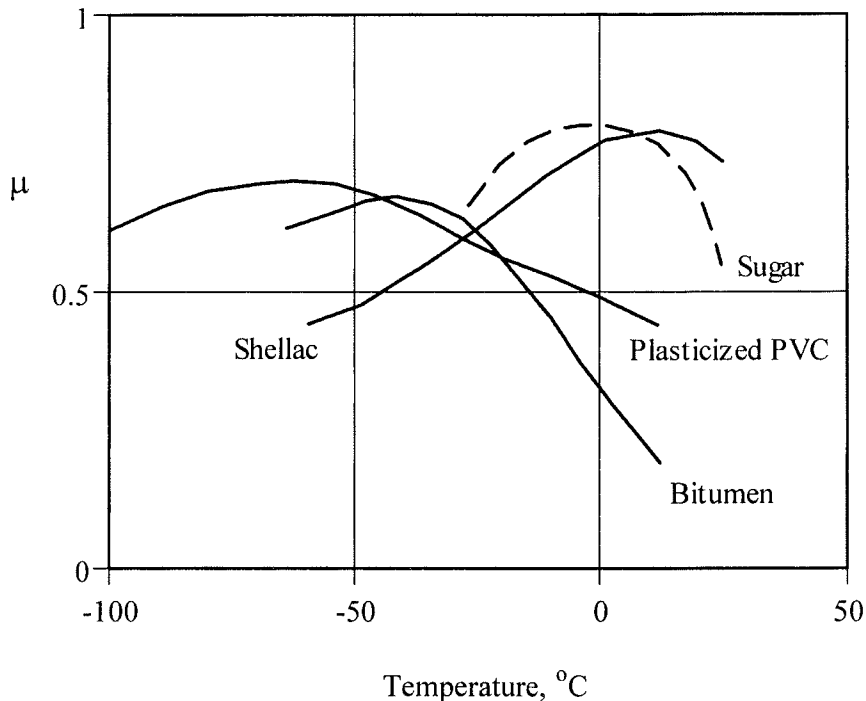
The double-logarithmic shift rate,  $\mu$ , was defined as follows:

$$\mu = -\frac{d \log a}{d \log t_e} \quad (1)$$

It was found that  $\mu$  was largely independent of ageing time except for very short loading times and high stiffness. Above the glass transition,  $\mu$  was found to be zero for all polymers investigated. Below the glass transition, it rapidly increased to unity remaining there over a wide range of colder temperatures, to eventually decrease again due to the loss in segmental mobility.



For the heavy roofing bitumen, as well as for a range of other materials,  $\mu$  was quite variable and peaked between 0.65 and 0.80, which was attributed to the unusually broad glass transition for this group of materials. Figure 2 shows the results that were obtained for bitumen, plasticized PVC, sugar, and shellac. It is interesting to note that for this blown bitumen the ageing rate was found to be quite significant from temperatures of  $-50^{\circ}\text{C}$  to  $+10^{\circ}\text{C}$ , which covers a large part of the service range in a typical pavement.



**Figure 2. Physical Ageing Shift Factor as a Function of Temperature for Various Materials (Reproduced from Struik (1978, p. 21))**

Struik (1978) took a largely qualitative approach since no free-volume model available from the literature was able to describe ageing quantitatively. As will become apparent from the discussion that follows, for asphalt materials produced today the situation is no different. Although more is known than in the early days, for the most part the reversible ageing processes are affected by too many variables, making a theoretical description difficult if not impossible. Hence, in this report the ageing process will be considered from a purely qualitative perspective. The loss in AASHTO M320 grade temperature will be used as a measure of ageing and the importance of the degree of ageing will be assessed with respect to performance prediction in service.

## STRATEGIC HIGHWAY RESEARCH PROGRAM

Researchers supported through the Strategic Highway Research Program (SHRP) spent a considerable amount of time on the study of reversible ageing, with a particular emphasis on the

ageing that occurs below the glass transition temperature (Bahia (1991), Bahia and Anderson (1991 and 1993)). The experimental approach followed largely the directions taken by Struik (1978) in his investigation of the process for a variety of materials.

Bahia and Anderson (1991) adopted the term *physical hardening* to describe the reversible ageing process in order to distinguish it from oxidative ageing and perhaps from the *physical ageing* term which had been used by Struik (1978). Bahia (1991) states that the hardening phenomenon was “not reported before for asphalt cements” (p. iii), that “no direct measurements at low temperatures have ever been published” (p. 4), “this type of physical hardening of asphalt cements has not been reported before” (p. 4), “The hardening, being systematically reported for the first time, was herein referred to as low-temperature isothermal physical hardening” (p. 228).

From their experimental results on a set of eight SHRP core asphalts, the authors concluded that reversible ageing below the glass transition region is mainly due to the collapse of free volume. It was argued that slow crystallization and structure formation were unlikely contributing factors to the hardening process. This was a conclusion that was later disputed by Anderson and Marasteanu (1999), who tested the physical hardening rate as a function of wax content and concluded that there was a strong link.

By comparing the physical hardening shift factors of different asphalt binders with their molecular characteristics, the SHRP researchers concluded that the higher molecular weight fractions caused a high degree of hardening. It was also found that intermolecular bonds are of importance with regard to the rate and final degree of hardening.

Interim versions of the BBR low-temperature specification test protocol that was developed through SHRP allowed for a determination of the grade temperature after both 1-hour and 24-hour conditioning, but the provision never appears to have found wide acceptance, for reasons that are not well documented (Anderson and Kennedy (1993)).

## **CURRENT RESEARCH**

Santagata et al. (1996), Lu and Isacson (2000), and Lu et al. (2003) studied the effect of polymer modifiers on the low-temperature reversible ageing process. Santagata et al. (1996) studied 16 different modified binders and concluded that no simple effect of the polymer modifier can be found and that different polymers have “widely different effects on the ageing rates.” Ethylene vinylacetate (EVA) copolymers were found to make the hardening worse, while ethylene methylacrylate (EMA) and radial styrene-butadiene-styrene (SBS) copolymers were found to lessen it. Lu and Isacson (2000) studied 5 unmodified and 35 polymer-modified binders and came to a somewhat different conclusion in that, in most cases, the effect of polymer modification was found to be “insignificant.” However, a close inspection of their ageing shift factors leads one to believe that certain polymers can in fact have significant effects on the hardening process.

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More recent papers by Lu et al. (2003) and Soenen et al. (2004) discuss results on 20 different straight and modified mixtures in the thermal-stress-restrained specimen test (TSRST). The authors found that the physical hardening phenomenon as found in the binders was only slightly influenced by the presence of modifiers. They did not observe any effect of physical hardening in the TSRST.

Prior to the work on modified binders, Romero et al. (1999) studied the physical hardening phenomenon for two SHRP core binders, AAM-1 and AAM-2, which were known to suffer a great deal from the hardening process. The authors investigated the binders with TSRST in two different aggregates. The main conclusion stated by the authors was that although some TSRST properties were affected by storage time, "other factors, such as mineral fillers and air voids of the mixtures, had a greater influence on the results than did physical hardening."

These studies show that either the reversible ageing phenomenon is of no importance to performance in service, or that TSRST has only limited value for performance prediction. Results documented and discussed in this report suggest that the reversible hardening phenomenon is of importance, and hence the use of TSRST is of only limited value since it is inherently poor at reproducing the exact conditions that exist over the life of the pavement.

Anderson and Marasteanu (1999) presented double-logarithmic shift rates for four SHRP MRL binders and, in contrast to the results from Struik (1978), found that  $\mu$  did approach unity at low enough temperatures. Further, they also found that the shift rate was substantial above the glass transition especially for binders that contained a large amount of wax. The authors came to conclude that "physical hardening is caused in addition to free volume collapse by the formation of crystalline fractions (wax)" and "asphalt binders with higher wax contents show stronger physical hardening both below and above their  $T_g$ ." This conclusion stands in contrast with those obtained in the earlier SHRP research (Bahia (1991)) and the one more recently obtained by Lu et al. (2004, p. 1) who stated that "there was no simple relationship between asphalt cracking and bitumen wax content" and Soenen et al. (2004, p. 8) who stated that "there is no direct relation with the wax content."

Phillips (1999) presented a proposal to use so-called pseudo Black diagrams plotting the logarithm of the creep modulus,  $\log S$ , versus the slope of the creep compliance curve,  $m$ , to determine the effects of reversible hardening. The author concludes that for many binders the use of only a horizontal shift factor is insufficient due to thermorheologically complex behaviour. Hence, a pseudo Black diagram is used to differentiate between thermorheologically simple and complex behaviour. The author found for a set of 16 different binders that the effect of isothermal storage for one day corresponded to a loss in low-temperature grade ranging from 1.5°C to 6°C. Phillips (1999, p. 3) comments that "the difference between the least hardening and the most hardening binder is thus equivalent to 4.5°C, which can be significant in Superpave grading."

Marasteanu and Anderson (2001) corrected Phillips's (1999) analysis in that the polynomial he used to fit the log S versus log t data is an approximation with substantial errors at both short and long loading times. The authors stated that pseudo Black diagrams should not be used for rheological evaluations (Marasteanu and Anderson (2001, p. 38)), a conclusion which may not be totally warranted.

Dongré (2000) conducted stress relaxation tests on four different asphalt binders after 1 and 24 hours of conditioning and hypothesized that the physical hardening is offset by the effects of relaxation, which typically require less time.

It is evident from the above review of the literature that there is still much uncertainty in the literature about whether the reversible ageing process in asphalt binders is important for performance or whether it is just a curious phenomenon of the binder with little relevance to the asphalt mixture and pavement.

Basu et al. (2003) tested a group of nine binders that had all been used in test sections in Ontario. He found that these binders lost anywhere from 3°C to 10°C after three days of conditioning at their grading temperatures. The binders investigated included straight run and oxidized as well as polymer-modified materials. These and other binders were later investigated by Iliuta et al. (2004a and 2004b) who found the correlation between the grade temperatures after three days of conditioning and the probable cracking onset temperatures in the test sections to be much stronger than the correlation with the regular AASHTO M320 grade temperatures. To the best of our knowledge, these results are the first and only to indicate with field distress data that the reversible ageing phenomenon is a probable and serious cause of early distress in many northern pavements.

The results documented and discussed in this report will add to the evidence presented by Iliuta et al. (2004a and 2004b). A large number of binders are investigated for their tendency to reversibly age at low temperatures, and their loss in grade temperature is correlated with field performance where possible.

# Experimental

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## MATERIALS

The materials investigated in this project were obtained from various sources.

The seven original binders used in the C-SHRP test road in Lamont, Alberta were obtained from Sierra Transportation Engineers in Reno, Nevada, who operates the SHRP Materials Reference Library (RR-2L, RR-3L, RR-4L, RR-6L, and RR-7L), and from Dr. Ludo Zanzotto at the University of Calgary (RR-1L and RR-5L).

Two of the three original binders used in the C-SHRP test road in Hearst, Ontario were no longer available, and hence field-aged binders were extracted from core samples that were taken after 12 years of service. Binder samples were extracted with a minimum of four tetrahydrofuran (THF) solvent washings. The bulk of the solvent was removed by rotary evaporation at moderate temperatures until no more THF was visibly distilled. In order to assure that no further solvent remained in the samples, rotary evaporation was continued for an additional two hours at a temperature of 150°C.

Seven binders were obtained from the most recently constructed low-temperature pavement trial north of Timmins, Ontario. The binders were sampled during the paving of each test section from the line used to transfer the material from their storage tank into the asphalt plant.

Seven SHRP MRL binders were investigated for their tendency to age reversibly at low temperatures. These binders had been thoroughly investigated during the SHRP program and hence provided a ready source of widely available asphalts of varying quality.

The last group contains oxidized and wax-modified binders that were obtained from commercial sources and were investigated according to standard procedures for their tendency to age during storage.

A listing of all binders and their pertinent information is given in Table 1. The binder codes as given were those used by the respective sources.

## EXPERIMENTAL METHODS

### Laboratory Chemical Ageing

All binders were aged according to standard protocol. Aging was performed in both the rolling thin film oven (RTFO) and in the pressure aging vessel (PAV) according to AASHTO T240-97 and PP1-98 methods prior to testing for low-temperature properties.



**Table 1. Pertinent Properties of Binders Investigated for Reversible Ageing**

Binders	Crude Source(s)	Modification	Grades†
<u>Lamont, AB</u>			
RR-1L	Boundary Lake	Oxidized	80/100 A, PG 58-22
RR-2L	Montana/Bow River	Unmodified	150/200 B, PG 52-28
RR-3L	Cold Lake	Unmodified	300/400 A, PG 46-34
RR-4L	Redwater	Unmodified	80/100 C, PG 58-22
RR-5L	Lloydminster	Oxidized	80/100 A, PG 64-28
RR-6L	Lloydminster	Unmodified	150/200 A, PG 52-28
RR-7L	Cold Lake	Unmodified	200/300 A, PG 52-34
<u>Hearst, ON</u>			
RR-1H	Venezuelan	Unmodified	150/200 A, PG 52-33
RR-2H	Lloydminster	Unmodified	150/200 A, PG 52-33
RR-3H	Bow River	Oxidized	200/300 B or A*, PG 46-37
<u>Timmins, ON</u>			
655-1	Lloydminster	RET	PG 64-37
655-2	Unknown	Oxidized/SBS	PG 64-35
655-3	Unknown	SBS	PG 64-36
655-4	Unknown	SBS	PG 64-35
655-5	Unknown	SBS	PG 64-35
655-6	Unknown	Oxidized	PG 58-35
655-7	Unknown	Unmodified	PG 52-35
<u>SHRP MRL</u>			
AAA-2	Lloydminster	Unmodified	200/300, PG 46-37
AAC-2	Redwater	Unmodified	AC-5, PG 52-27
AAE	Lloydminster	Oxidized	60/70, PG 70-22
AAG-2	California Valley	Unmodified	AR-2000, PG 58-22
AAK-1	Boscan	Unmodified	AC-30, PG 64-28
AAL	Cold Lake	Unmodified	150/200, PG 58-33
AAN	Bow River	Unmodified	85/100, PG 58-28
<u>Various Companies</u>			
A-1	Lloydminster	Wax	PG 58-34
B-1	Unknown	Oxidized	PG 58-34
B-2	Unknown	Oxidized	PG 64-28
B-3	Unknown	Oxidized	PG 70-28
C-1	Unknown	Oxidized	PG 58-34

Note: RET = reactive ethylene terpolymer of ethylene, butyl acrylate, and glycidyl methacrylate; SBS = styrene-butadiene-styrene linear block copolymer; and A, B, and C refer to three different commercial sources of the listed binders. † The grade information is as given by EBA (1994), Robertson (1995), and Gavin (2003). \* This binder graded as either a Group A or B depending on which viscosity was used in the Bitumen Test Data Chart (EBA (1994)).

### **Bending Beam Rheometer Conditioning and Testing Protocols**

Samples tested in the bending beam rheometer (BBR) were conditioned for one hour at a given temperature and then tested at either two or three different temperatures to determine a pass and a fail temperature. In some instances, the materials were conditioned for longer periods and tested after various conditioning times to determine their loss in grade temperatures.

Whether the cooling liquid had any effect on the reversible ageing process was investigated by storing duplicate sets of samples in an air-cooled freezer. Two different binders were compared in this manner and the differences in the results between air and liquid cooled samples were found to be within the experimental error of BBR. Hence, it was decided to cool all samples in a water-alcohol mixture.

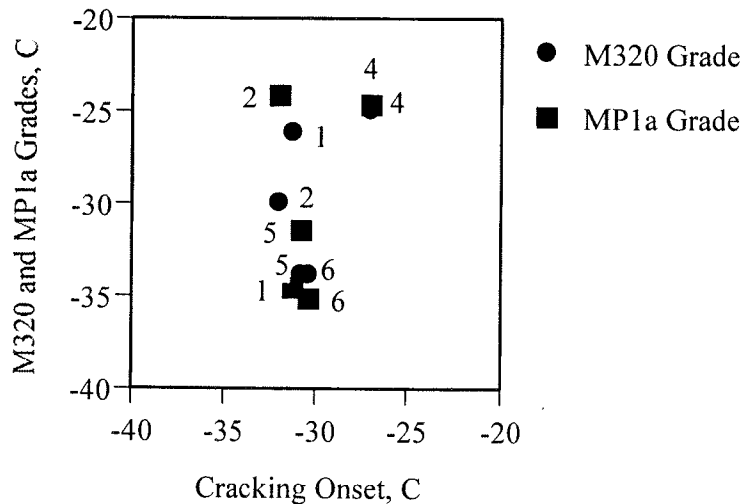
A typical protocol for the reversible ageing test consisted of storing six to eight BBR specimens at three different temperatures for 1 hour, 24 hours, and 72 hours and test them after each conditioning time at three different temperatures to obtain a pass and a fail according to the AASHTO M320 criteria (AASHTO (2002)). The samples were conditioned for between 10 and 15 minutes at the testing temperature before the actual creep experiment was conducted.

# Results and Discussions

## LAMONT, ALBERTA C-SHRP TEST ROAD

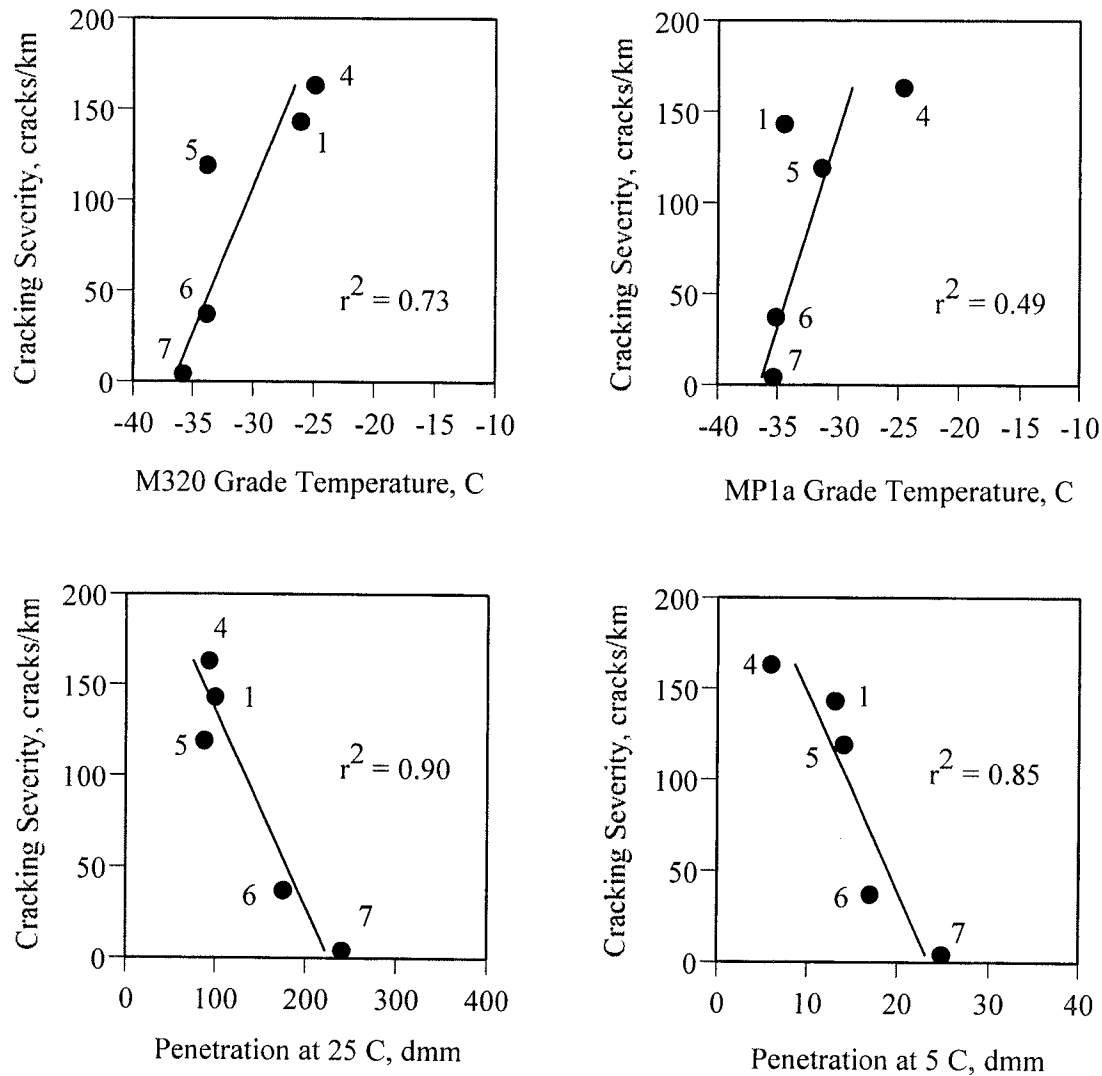
Field performance data for the Lamont, Alberta C-SHRP test road has been published in the Proceedings of the Canadian Technical Asphalt Association on two occasions: early data were presented by Robertson (1995), while more recent results were given by Gavin et al. (2003). Iliuta et al. (2004b) uses the field performance data from these sources to argue that the AASHTO M320 specification method needs to be improved. The arguments as presented in Iliuta et al. (2004b) will be presented here with slight modification. This is followed by the reversible ageing test results on the original binders and a discussion of those results.

The Lamont test road contained seven sections ranging from 417 to 500 m in length (EBA (1994)). The different binders that were used in each section and their respective grades are listed in Table 1. The cracking onset temperatures as provided by Anderson et al. (1998 and 1999) are provided in Figure 3, while the 12-year cracking severity as correlated with various binder properties is provided in Figure 4. Since Section 3 has no cracks it is left out of the comparison. Section 2 with 180 cracks/km had a low thickness and significantly higher air voids content and hence was also excluded from the analysis (EBA (1994, p. 75)).



**Figure 3. Comparison between AASHTO M320 and MP1a Grade Temperatures and Cracking Onset Temperatures as Measured at 12-mm Depth**

Note: Labels at each symbol relate to test sections for which cracking onset was determined. AASHTO M320 and MP1a temperatures as reported by Robertson (1995) and Bouldin et al. (2000) and cracking onset temperatures as reported by Anderson et al. (1999) and Gavin et al. (2003).



**Figure 4. Comparison between Cracking Severity and AASHTO M320, MP1a and Penetration Values at 25°C and 5°C for Lamont, Alberta Test Sections**

Note: Label at each symbol relates to section number. Binder properties are as reported by EBA (1994) and Gavin et al. (2003); severity is as reported by Gavin et al. (2003). Section 3 was excluded because it has no cracks. Section 2 was excluded because it has significantly lower thickness and high voids.

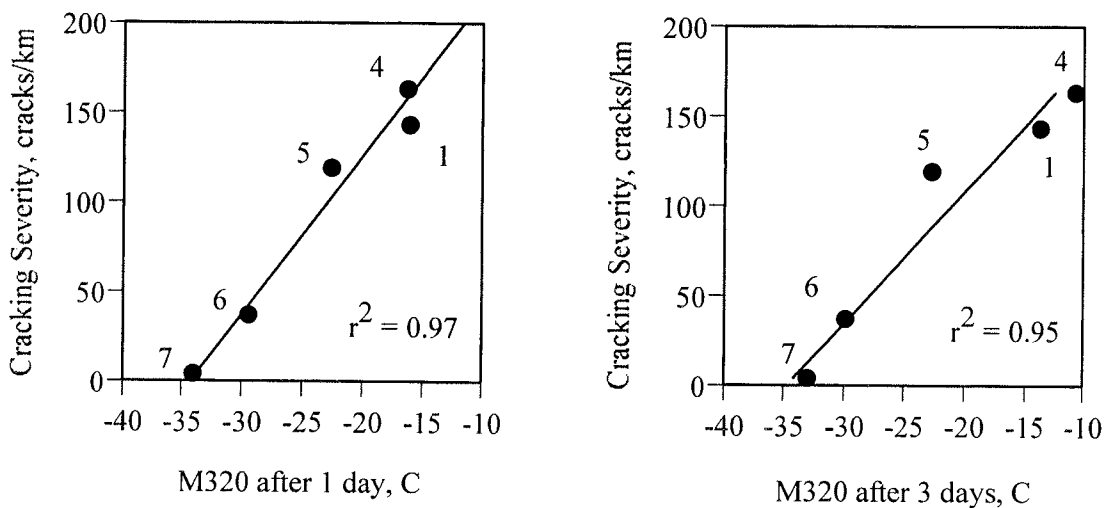
The data provide several interesting points of discussion. First, the cracking appears to start in all sections in a narrow range between  $-30^{\circ}\text{C}$  and  $-32^{\circ}\text{C}$  at 12 mm depth. (The exception is Section 4 for which it was suspected that the thermocouple was incorrectly calibrated.) This appears counter-intuitive given the fact that the binders that cracked varied by more than  $10^{\circ}\text{C}$  in their SHRP performance grades. Possible reasons for this could be related to flaws in the design of the crack detection system, with perhaps the aluminum foil breaking in all sections at about the same temperature. An alternative explanation could relate this to a failure at the binder-aggregate

interface, which could have occurred at about the same temperature in all sections causing transverse cracking (at about the same temperature). However, this is only speculation, and the exact reason for this observation is probably impossible to determine at this moment.

Second, the correlation between the limiting temperatures as measured according to the AASHTO M320 method and the 12-year cracking severity is weak and it appears that the approach misses the poor performance of the oxidized binder of Section 5. The MP1a method still misses the poor performance of the two oxidized binders in Sections 1 and 5, which are the very binders that it was designed to detect and exclude (Bouldin et al. (2000) and Bouldin and Dongré (2000)).

A third observation relates to the predictive ability of the penetration grades at 25°C and 5°C. These appear to predict the cracking severity with much better accuracy than the AASHTO M320 and MP1a methods with correlation coefficients of 0.90 (Pen at 25°C) and 0.85 (Pen at 5°C) compared to 0.73 (M320) and 0.49 (MP1a). This could be a coincidence or perhaps due to fundamental reasons that are unknown at present. Experience with other pavement trials can only shed light on this issue.

The seven original binders from Lamont were recently tested for their tendency to age while stored at cold temperatures with encouraging results. The binders were stored at -10°C and -20°C for periods of up to three days. The limiting temperatures were determined and the warmest values were used to correlate with the cracking severity. The comparison between cracking severity and AASHTO M320 grades after conditioning for 24 and 72 hours is given in Figure 5.



**Figure 5. Comparison between Cracking Severity and AASHTO M320 Grades after One and Three Days of Isothermal Storage for Lamont, Alberta Test Sections**

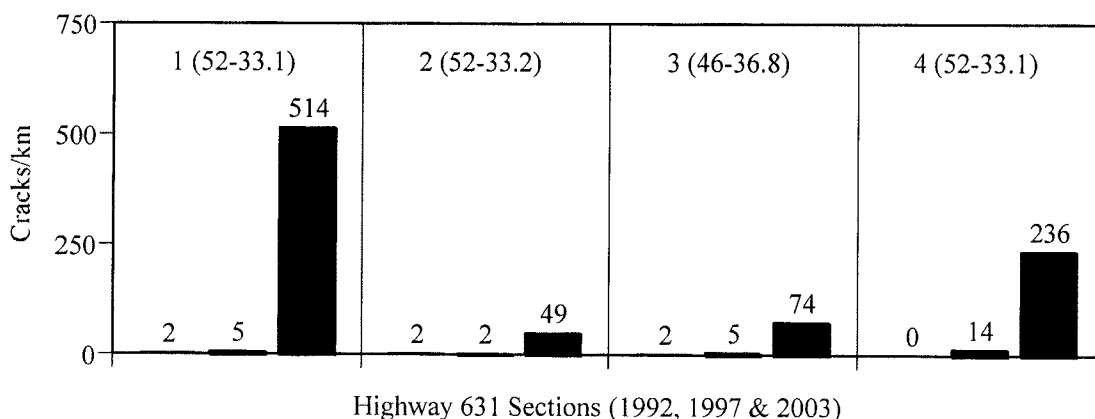
Note: Labels at each symbol relate to test section number. Warmest grade temperatures after one and three days conditioning at either -10°C or -20°C were used for the correlation. Section 3 was excluded because it shows no cracks. Section 2 was excluded because it has significantly lower thickness and high voids content. Cracking severity is as reported by Gavin et al. (2003).

It appears that BBR results on the conditioned samples provide a much-improved prediction of performance compared to the results after just one hour of conditioning under standard AAHTO M320 protocol. It should be noted that the binders from Sections 1, 4, and 5 lost as much as 10.5°C, 14°C, and 7.6°C, respectively, after only three days of conditioning whereas the binders from Sections 3, 7 and 6 lost only between 0.3°C and 1.5°C. As with the penetration test results this better correlation with cracking performance in service could again be a coincidence. However, it is more likely that the limiting properties as measured with BBR have some relationship to performance and that the reversible ageing process does indeed increase the amount of low-temperature cracking. This would contradict the findings of the growing list of studies that concluded from laboratory experiments that reversible ageing is unimportant (Dongré (2000), Lu et al. (2003), Soenen et al. (2004) and others). However, it would agree with our own observation that premature cracking in test roads near Petawawa and Bracebridge, Ontario could be explained, at least in part, by the effects of reversible ageing (Iliuta et al. (2004a)).

## HEARST, ONTARIO C-SHRP TEST ROAD

The performance of the C-SHRP test road near Hearst, Ontario is discussed in Hesp (2004a) and Iliuta et al. (2004a and 2004b) of which the aspects that relate to reversible ageing at low temperatures will be reviewed next.

The C-SHRP test road in Hearst was constructed in 1991 and hence it provides us with reliable transverse and fatigue cracking data. As shown in Figure 6, early rankings in terms of low-temperature cracking performance often change but this should no longer be a concern today after 12 years of service. Over the period prior to our visit in the summer of 2003, it has likely been exposed to temperatures close to its design temperature on many occasions, while it also has gone through 12 of what were likely extended periods of freeze-thaw distress.



**Figure 6. Transverse Cracking History for the C-SHRP Test Road Near Hearst, Ontario**

Note: PGAC grades are given in brackets after the section number and calculated from the data presented by Robertson (1995).

The main issue is what factor or factors are responsible for the large performance differences as reported in Figure 6 for binders of nearly the same SHRP and CGSB performance grades. The difference in cracking severity between Sections 1 and 2, which have identical performance grades, is a factor of 20 as measured by total crack length. Furthermore, Section 3 has 50 percent more cracks compared to Section 2, despite the fact that its performance grade is as much as 3.6°C lower than that of Section 2. Finally, comparing the performance of Sections 1 and 4, which were constructed with the same binder, shows that doubling the asphalt pavement thickness (as was done in Section 4) reduces the cracking severity for this particular binder by as much as 54 percent.

### Structural and Design Issues

The first obvious place to look for possible causes for the observed performance differences in the Highway 631 test road is in the structural and design aspects of the test sections. Given the fact that this was one of the original C-SHRP test sites, it is unlikely that structural and/or design factors confound the findings. However, it was considered prudent to determine the variations in pavement thickness, void content, and asphalt cement content for the four test sections. Table 2 shows the results for this analysis on a total of 30 core samples taken at 50-m intervals all through the site. The average core thickness and void content for the sections were determined from measurements on eight cores for each of Sections 1, 2, and 3 and six cores for Section 4. Asphalt cement contents were determined by ignition. The average is for two cores and the error is the difference between the two determinations divided by two.

**Table 2. Structural and Design Parameters for C-SHRP Test Sections on Highway 631**

Source	Thickness, mm	Voids, %	Asphalt Cement, wt %
631-1	45.0 ± 5.1 (40-52)	6.9 ± 0.09 (5.0-8.2)	5.36 ± 0.22
631-2	46.3 ± 3.6 (42-51)	6.7 ± 0.06 (5.5-7.9)	5.82 ± 0.01
631-3	46.3 ± 6.4 (37-54)	7.3 ± 0.06 (5.8-8.2)	5.73 ± 0.14
631-4	88.8 ± 5.0 (83-95)	-	-

Note: Averages are given with standard deviations while numbers in brackets give the range as found for the core thickness and void contents. Void and binder contents for Section 4 were not determined but were considered to be close to those of Section 1.

The data in Table 2 clearly show that this was a carefully constructed test site and that structural and design factors (other than the double thickness in Section 4) are unlikely factors to explain the large differences in performance between sections. The slightly low asphalt cement content in Section 1 may be representative or it may just be because only two cores were taken for ignition from each section. However, if representative then it would probably be able to explain a small part of the 20-fold difference in cracking severity between Sections 1 and 2.

### Chemical Ageing Effects

If large differences in the tendency of each binder to chemically harden were present then this would explain to some extent the observed performance differences. The binders used in Hearst were from three different sources and hence are likely to show variations in their loss of grade temperature after 12 years. Table 3 shows the comparison between the original binder grading temperatures with those determined on materials extracted from the 12 year old pavement.

**Table 3. PAV-aged versus Field-aged AASHTO M320 Properties for Highway 631 Binders**

Source	Original PAV Material		12 Years Aged In Service		Loss in Grade Temperature, °C
	T(S=300 MPa)	T(m=0.3)	T(S=300 MPa)	T(m=0.3)	
RR-1H	<b>-23.1</b>	-25.0	-21.1	<b>-15.5</b>	7.6
RR-2H	<b>-23.2</b>	-26.4	<b>-19.6</b>	-19.7	3.6
RR-3H	<b>-26.8</b>	-28.2	-23.6	<b>-21.5</b>	5.3

Note: Numbers in bold are those that determine the performance grade which is set at a temperature 10°C below the measured limiting temperature. Original binder properties as reported by Robertson (1995).

The results show that there are significant differences in chemical ageing behavior among the three binders (as measured by BBR on extracted samples). The binder used in Section 2, with the least amount of cracking, shows a significantly lower tendency for chemical ageing, gaining an edge of 4.1°C after 12 years of service compared to the binder used in Section 1. Given the fact that the above properties were determined on binder extracted from whole asphalt core samples, the question is raised of how much the ageing varied with depth in the pavement. Perhaps the binder used in Section 1 with the highest number of cracks has aged significantly on the top surface thus making it more prone to crack initiation and therefore large-scale transverse cracking.

The loss in grade temperatures of between 3.6°C and 7.6°C over and above the PAV-aged materials is a serious cause for concern. The RTFO/PAV ageing protocol was supposed to age binders to the equivalent of about 7 to 8 years of field service. Recent research by our group suggests that this is equivalent to only 2 to 3 years at best, suggesting that an improved laboratory ageing method would go a long way to reduce the widespread occurrence of premature low-temperature distress (Hesp (2004a)).

Although the differences among the three sections are significant, it was considered that this may not be the only factor to explain the differences in cracking severity of a factor of 20. Hence, other factors including differences in reversible ageing, and in brittle and ductile fracture performance were also investigated.

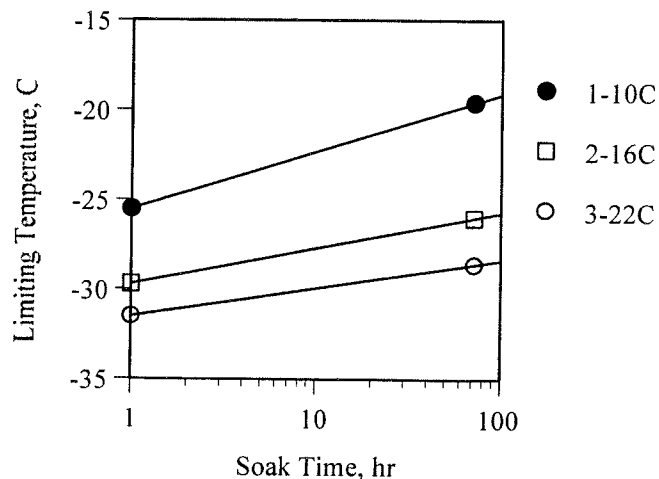
### Reversible Ageing Effects

A second likely contributing factor for the large differences in performance as observed on Highway 631 comes from the tendency of different binders to suffer to different degrees from reversible ageing. The binder used in Sections 1 and 4 would only have to age by a few degrees



more than the one from Section 2 to explain a large part of the difference in cracking severity. Temperature statistics are such that if the “real” performance grade would have been reached for Section 2 on only a couple of occasions, then Section 1 would only have to physically age by a few degrees more to have been exposed to numerous more days of damaging temperatures.

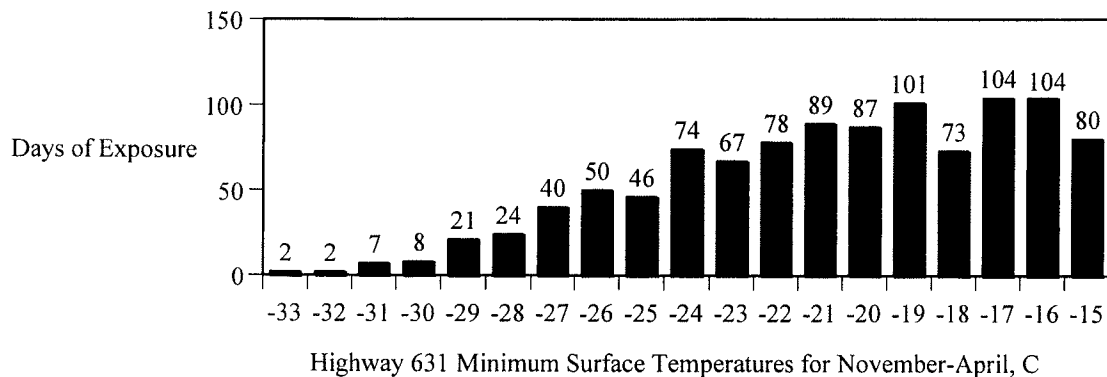
Figure 7 provides the rates of reversible ageing for the three binders as extracted from the asphalt mixture after 12 years of service. The data show that all three binders reversibly age to a significant degree. For Sections 1 and 2 the maximum (worst) rate was found to occur for conditioning temperatures of  $-10^{\circ}\text{C}$  and  $-16^{\circ}\text{C}$ , respectively, while for Section 3 it happened at  $-22^{\circ}\text{C}$ . The results show that the physical ageing mechanism is a contributing factor that can explain a large portion of the severe cracking in Section 1 and a small portion of the factor of 20 difference in cracking severity between Sections 1 and 2. While after 1 hour of conditioning the difference in grade temperature between Sections 1 and 2 is already a significant  $4.1^{\circ}\text{C}$  (see Table 3), this worsens to  $6.4^{\circ}\text{C}$  after 72 hours of conditioning.



**Figure 7. Physical Ageing Rates for Binders Used on C-SHRP Sections on Highway 631**

Note: Section numbers in legend are followed by soak temperature where maximum ageing occurred.

Figure 8 illustrates the possible consequences of this difference in grade temperature in light of the past 12 years of temperature records available for Kapuskasing Airport. If the true grades were those as measured after 72 hours of conditioning, then Section 1 would have sustained damage for approximately 595 days over the life of the road versus approximately 154 days for Section 2. This difference of nearly a factor of 4 is likely responsible for a large part of the 20-fold difference in performance between Sections 1 and 2.



**Figure 8. Minimum Pavement Surface Temperatures for Highway 631 as Calculated from Air Temperatures as Recorded at Nearest Weather Station in Kapuskasing**

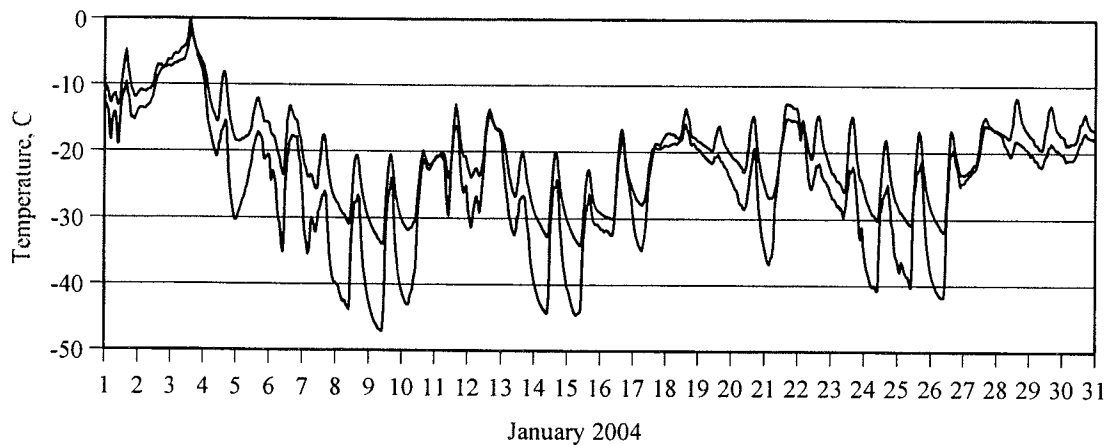
That there is more to this issue becomes clear from a comparison between Sections 1 and 4 which were made with the same binder from Venezuela but which had different thicknesses. The fact that Section 4 has only 46 percent of the cracking severity when compared with Section 1 suggests that more than half of the cracks in Section 1 are related to some form of fatigue distress (thermal, spring-thaw, or traffic induced). If some of the cracks in Section 4 are also related to fatigue then the proportion of fatigue related cracks in Section 1 would even be higher than 46 percent.

Further, the effects of chemical and reversible ageing also fail to explain the difference in cracking severity between Sections 2 and 3 with 49 and 74 cracks, at performance grades of  $-26.0^{\circ}\text{C}$  and  $-28.6^{\circ}\text{C}$ , respectively, after three days of conditioning. Both binders suffer equally from reversible ageing so there must be other factors to explain the significant difference in performance. This analysis suggests that the fatigue properties of the binders must have some impact on the level of transverse cracking. Hence, brittle fracture energies at  $-30^{\circ}\text{C}$  and  $-24^{\circ}\text{C}$  as well as ductile fracture energies at  $0^{\circ}\text{C}$  and  $25^{\circ}\text{C}$  were determined in order to further understand the observed distress levels. The results of the analysis are discussed in Iliuta et al. (2004b) and will be further analyzed in a subsequent report on fracture properties.

## COCHRANE, ONTARIO TEST ROAD

The newest of the test roads near Cochrane, Ontario has seven test sections with different modified and straight binders (Iliuta et al. (2004b)). It experienced severely cold weather just three months after the end of construction. Whereas the road was designed to withstand a minimum surface temperature of  $-34^{\circ}\text{C}$  after some 7 to 8 years of service, the pavement temperature hit an all-time low for the area in early January 2004 when the air temperature reached to approximately  $-47^{\circ}\text{C}$ . (The two thermocouple loggers recorded air temperatures of  $-46.1^{\circ}\text{C}$  and  $-48.2^{\circ}\text{C}$ .) The measured pavement temperature at approximately 5 mm depth was –

34°C and a surface temperature calculated according to the latest LTTPBIND® algorithm would have been around -37°C. Figure 9 provides a plot of the average air and 5-mm deep pavement temperatures recorded. The graph shows that for eight days in January 2004 the air temperature dropped below -40°C and the pavement temperature as measured at 5 mm depth dropped below -30°C. The record low pavement temperatures were reached on January 9 and 15 both at 9:30 a.m. when the average read -34.0 and -34.1°C, respectively. Given the fact that this road was designed to withstand a pavement surface temperature of -34°C after some 7 to 8 years of service, this was a near-perfect accelerated pavement test!

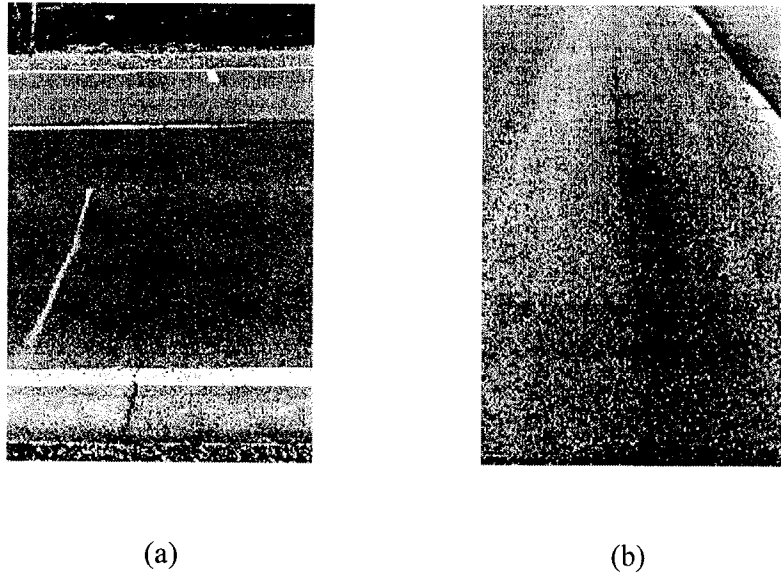


**Figure 9. Average Air and Pavement Temperatures for January 2004 on Highway 655**

Note: Air temperature is average of two thermocouple loggers whereas the pavement temperature is an average of four readings. Air readings varied by no more than 2°C between thermocouples whereas pavement readings varied by no more than 1.5°C between thermocouples.

On January 31, 2004, the site was visited to look for possible distress and to switch one of the two air thermocouple loggers. During this initial assessment only two transverse cracks were found in Section 2 (one had started at the joint with Section 1 while the other was located near the joint with Section 3) and one in Section 3 (located close to the joint with Section 4).

On April 24, 2004, the site was again visited for a second look for distress and to download the pavement temperature data. Surprisingly, a large amount of wheel path cracking was observed in Sections 2, 3, and 4 and a lesser amount in Section 6. Section 2 with 60 m of wheel path cracking also showed six transverse cracks, although these were all located either at the beginning or end of the section. Section 3 with 40 m of wheel path cracking showed only a single transverse crack that was less than half a lane wide towards the end of the section. Section 4 also had about 40 m of wheel path cracking and only a single transverse crack about half-a-lane wide approximately 180 m into the section. Section 6 showed approximately 5 m of wheel path cracking about 325 m into the section. Section 7 had only a single transverse crack about half-a-lane wide approximately 230 m into the section. Finally, only Sections 1 and 5 showed no detectable damage.



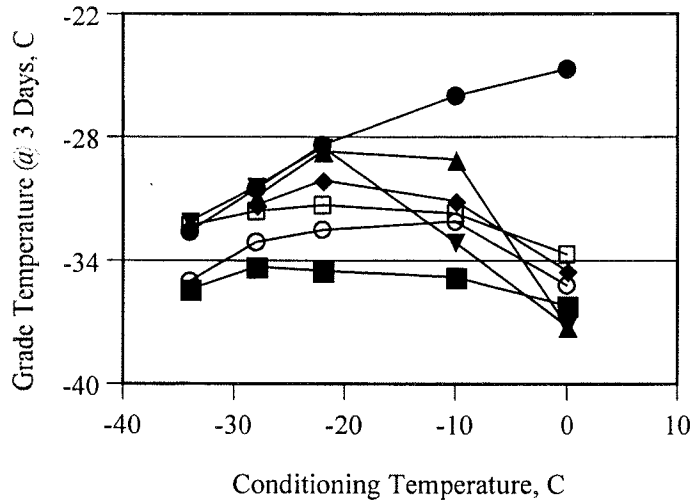
**Figure 10. (a) Representative Transverse Crack in Section 2 and (b) Representative Wheel Path Crack in Section 3**

Figure 10 shows a picture of (a) a transverse crack that has occurred in Section 2 and (b) the typical wheel path cracking that occurred in Section 3.

A noteworthy aspect of the cracking within the site is that all of the wheel path distress occurred on the left side of the southbound lane. This can be partially explained by the fact that loaded logging trucks go south to Timmins and return empty. However, it may be more difficult to explain why the distress was all confined to the left wheel path. The exact reason for this is difficult to determine but might have to do with the way in which the pavement thaws during spring. Water may collect primarily under the center of the road until it can escape later during spring thaw. This would expose the left wheel path to a longer period of an unstable base compared to the right wheel path. A second contributing factor may have been the higher *transverse* restraining stress in the left wheel path compared to the right one (which is closer to the unrestrained edge of the pavement). Such higher level of thermal stress would cause more damage initiation in the left wheel path during the early morning hours of January 8 to 10, 14 to 15 and 24 to 26, which only became visible during the spring thaw. However, there may be other reasons for the fact that only the left wheel path was affected. We expect that significant damage will show in all wheel paths for most sections. This study will have to show in years to come if some of the tougher modified binders can reduce or completely mitigate this distress and thereby produce a benefit to the user agency.

The reversible ageing phenomenon has been investigated for the seven binders in this trial. Section 2, with the most severe wheel path and transverse cracking, was made with the binder that was the most susceptible to reversible ageing. The binder used in Section 1 with no cracks showed the lowest tendency for reversible ageing. Section 5 with a moderate degree of reversible

ageing showed no cracks which is believed to be due to the high toughness of this material. Figure 11 shows the losses in grade temperatures for all binders after three days of conditioning at various temperatures.



**Figure 11. Losses in Grade Temperatures for Binders of Highway 655 Trial**

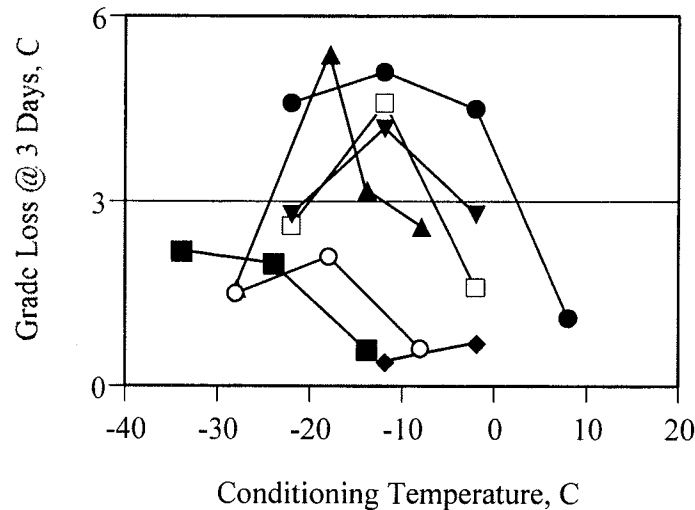
Note: ■ = 655-1; ○ = 655-3; □ = 655-5; ◆ = 655-4; ▲ = 655-6; ▼ = 655-7; and ● = 655-2.

The data in Figure 11 show that the oxidized binders in Sections 2 and 6 show the most severe reversible ageing during cold storage. The fact that the binder from Section 6 ages less than the one from Section 2 could be explained by the fact that the supplier for Section 6 was not able (or did not wish) to supply the requested PG 64-34 but rather ended up providing a PG 58-34 instead.

Another observation from the data in Figure 11 is that the general shape of each curve is similar to the one for bitumen in Figure 2. The shift factors as measured by Struik (1978) peak at some intermediate temperature due to the factors that relate to the free volume collapse and the molecular mobility. The loss in limiting BBR temperature also appears to peak at intermediate temperatures confirming Struik's (1978) analysis of the reversible ageing process.

## SHRP MRL BINDERS

A set of seven SHRP MRL binders were investigated for their tendency to reversibly age when stored at low temperatures. The results of this investigation are provided in Figure 12, which shows the losses in BBR limiting temperatures as a function of storage temperature. The set contains five binders from western Canadian sources and includes materials that are known to perform poorly (e.g., AAC-2 Redwater with a high wax content) and those that are generally considered to provide superior performance (e.g., AAA-2 Lloydminster and AAL Cold Lake). The figure also contains data for a Boscan asphalt (AAK-1) and an air-blown Lloydminster (AAE).

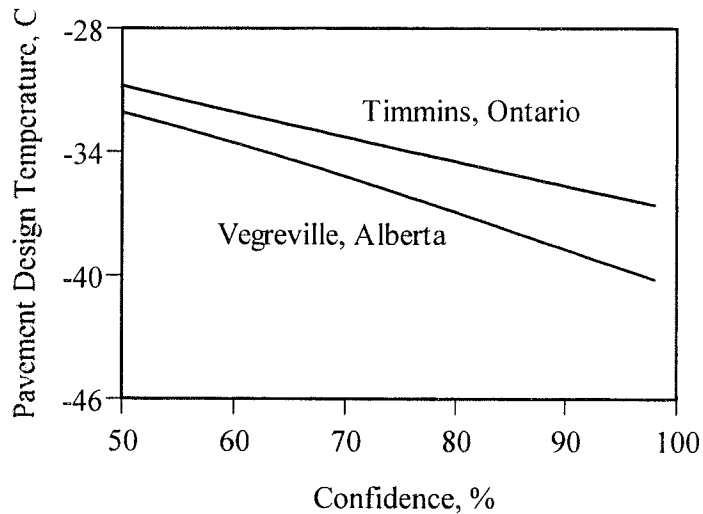


**Figure 12. Losses in Limiting BBR Temperatures for SHRP MRL Binders after Three Days of Isothermal Conditioning at Various Temperatures**

Note: ■ = AAA-2 Lloydminster, ● = AAC-2 Redwater, ▲ = AAE oxidized Lloydminster, ◆ = AAG-2 California Valley, □ = AAK-1 Boscan, ○ = AAL Cold Lake, and ▼ = AAN Bow River.

The results in Figure 12 are interesting from several perspectives. First, the differences in the loss in grade temperature are substantial yet not as high as those found for Lamont, Hearst, and Cochrane materials. Comparing binders AAG-2 and AAC-2 or AAE after three days of conditioning shows a difference in reversible hardening of approximately 4.7°C. After several weeks of ageing in the field, this could become a difference of perhaps 10°C, which is very substantial in terms of the weather statistics that hold for many parts of Canada. Figure 13 shows the typical LTPPBind® weather statistics for stations in Vegreville, Alberta (the nearest weather station to the C-SHRP site in Lamont) and Timmins, Ontario (the nearest weather station to the Highway 655 test road). This data shows that a loss in grade temperature of six degrees results in a loss of confidence from 98% to around 50% that a particular road is not going to be exposed to damaging temperatures in a given winter. Hence, losses of 3°C and higher are in fact already significant and can result in premature and severe cracking.

The worst performing binders in this set are again the oxidized (AAE) and Redwater (AAC-2) materials. However, they lose less than the same source materials in Lamont (RR-5L and RR-4L) perhaps because the grades are not the same. The best performing binders in this set are California Valley (AAG-2), Lloydminster (AAA-2) and Cold Lake (AAL), which is in general agreement with the findings of the SHRP program (Bahia (1991)) and those from Lamont discussed earlier (RR-3L, RR-6L and RR-7L). Once more, the loss in grade temperature peaks at some intermediate conditioning temperature, suggesting that the free volume collapse — together with the reduction in mobility — is responsible for a large part of the ageing.



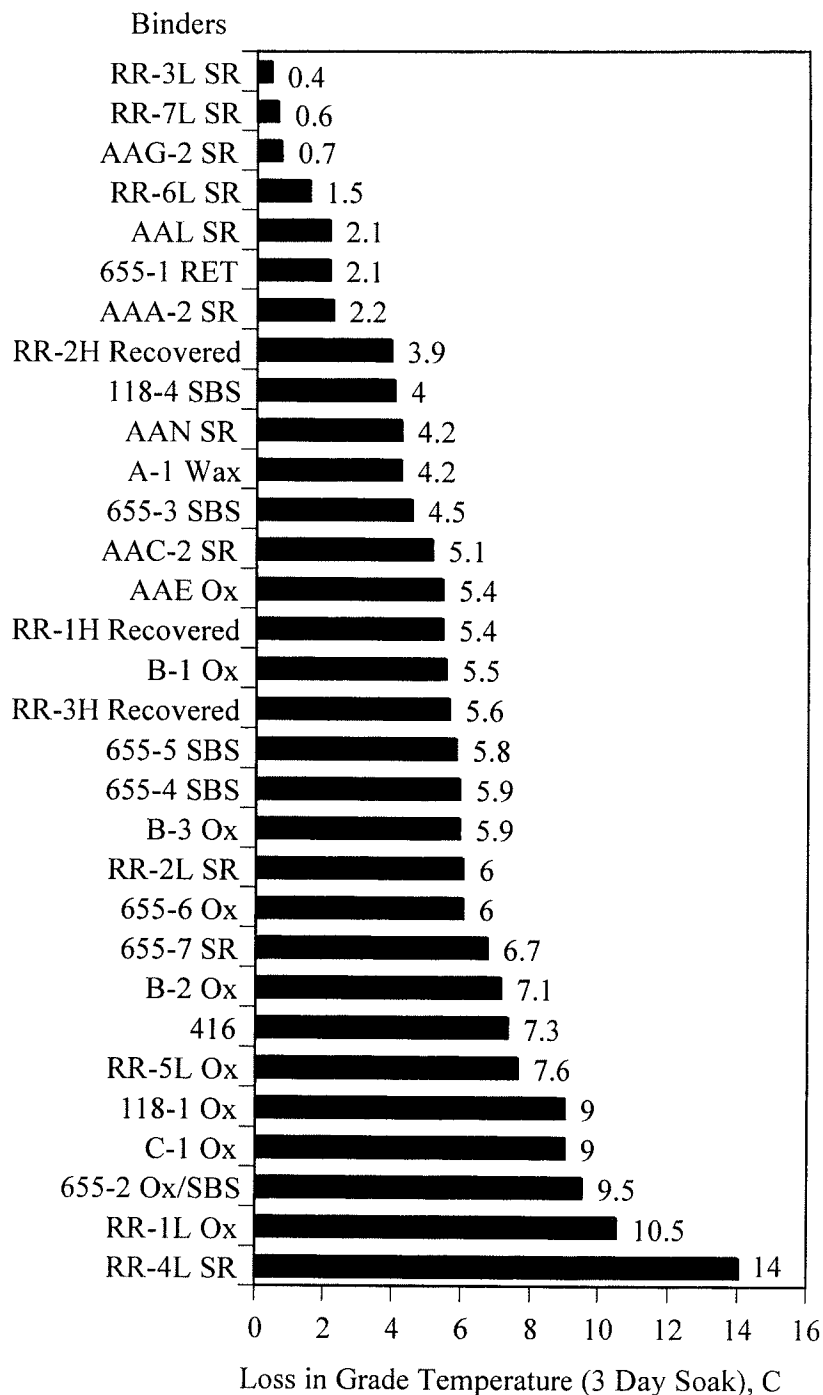
**Figure 13. Typical Statistics for Weather Stations Closest to Lamont, Alberta C-SHRP and Cochrane, Ontario Trials (LTPPBind® (1999))**

## WAX-MODIFIED AND OXIDIZED BINDERS

A final area of study considered the reversible ageing behavior of a limited number of wax-modified and oxidized binders. As the review of the literature indicated, the presence of wax and oxidized materials raises concerns regarding their increased tendency for reversible ageing. The results for these materials, contrasted with a number of other binders that have been tested at Queen's University in recent months, are given in Figure 14. Binders were tested at various temperatures and the worst three-day loss is given in the graph. However, the worst three-day loss as determined does not necessarily correspond to the absolute worst loss possible since time limitations made it possible to test only one or two conditioning temperatures for some binders.

Some European countries limit the amount of wax in their asphalt binders because of a conceived negative effect on performance. In North America there are no such regulations and it is left to the Superpave® performance grading method to decide whether or not a certain material can be used. This has led certain companies to develop wax modifiers that can increase the high-temperature performance grade of a binder without affecting the low-temperature BBR grade by much. Such products are suspect and hence we are investigating one of these to consider its relative performance to other modified materials. Unfortunately, the supplier of the product was only willing to provide us with slightly modified Lloydminster asphalt of a PG 58-34 grade.

The use of oxidized binders is becoming widespread in Ontario and elsewhere, since it is generally considered to be the cheapest way to obtain a certain performance grade. Hence, a number of oxidized binders from test sections in Lamont, Hearst, and Cochrane as well as some additional commercial products were obtained and evaluated for their tendency to reversibly age at low temperatures. The results are included in Figure 14.



**Figure 14. Losses in Limiting BBR Temperatures for Oxidized and Wax-Modified Asphalt Binders in Comparison to Various Straight and Polymer-Modified Binders**

Notes: Highway 416 cracked in its first winter and is suspected to have been an oxidized/polymer-modified hybrid asphalt (Hesp (2004b)). RET = reactive ethylene terpolymer, Ox = oxidized binder, SR = straight run, SBS = styrene-butadiene-styrene polymer. Binder details are given in table 1.



The data as presented in Figure 14 suggest that there are serious reversible ageing problems for many of the binders sold on the Canadian marketplace and in particular for oxidized binders. Five of the six worst performing binders in this investigation were oxidized while none of the six best performing binders were oxidized. As shown in Figure 13, the loss of only three degrees in the low-temperature grade could already result in serious distress over the life of a pavement. Hence, it is likely that the reversible ageing effect is a major cause for the large scale low-temperature cracking that is still so prevalent in much of the northern United States and Canada.

The modifier used in Binder A-1 Wax caused the reversible ageing effect to worsen by a factor of two compared to a straight run Lloydminster asphalt such as AAA-2. The high wax content in Binder RR-4L SR caused a severe loss of 14°C in the low-temperature grade after three days of storage at cold temperatures.

Binder 655-5 SBS aged a considerable amount, losing 5.8°C after three days, yet it has so far shown no detectable damage after a particularly severe winter (Iliuta et al. (2004b)). This may be due to the fact that this binder was much tougher than all others tested. However, it is somewhat premature to draw any firm conclusions and long-term performance monitoring will have to determine whether a higher toughness can in fact lessen or completely mitigate the distress.

## Conclusions

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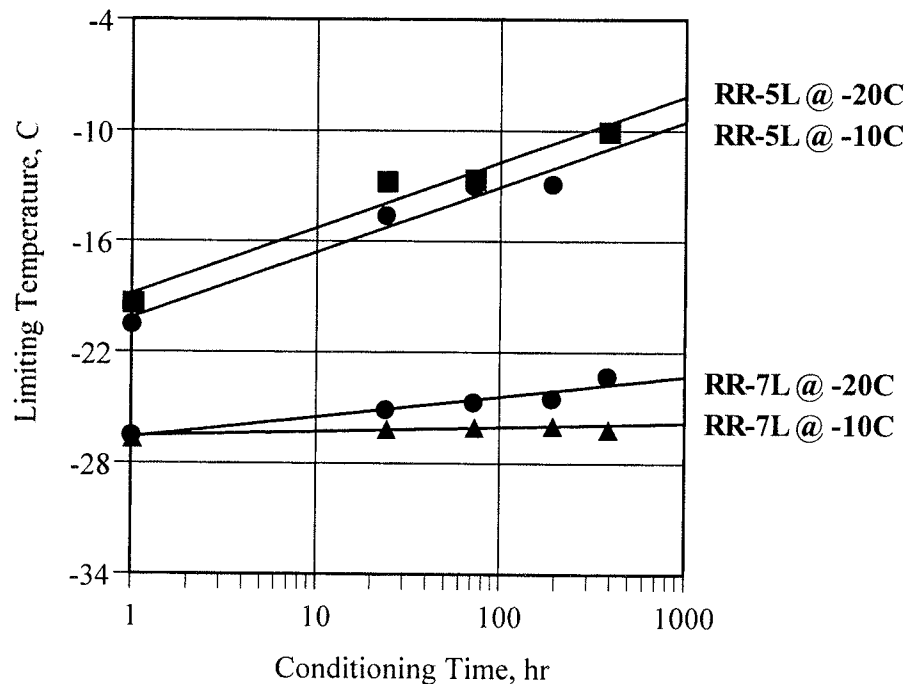
Given the review of the literature and the results presented in this report, the following conclusions are provided:

- The AASHTO M320 (BBR) and MP1a (BBR + DTT) are unable to provide an accurate indication of low-temperature performance in service.
- The error in performance prediction is large for wax-modified and oxidized binders, which could crack many times more severely than a good quality straight-run asphalt of the same grade. For the C-SHRP test sections in Lamont, Alberta, the difference in cracking severity for binders of nearly the same AASHTO M320 grade was found to be a factor of 30. For the C-SHRP sections in Hearst, Ontario the difference was found to be a factor of 20. A large part of these performance differences can be explained through the effects of reversible ageing at low temperatures.
- Superior binders were those obtained from California Valley, Cold Lake, and Lloydminster crude sources which typically lost 2°C or less from their low-temperature BBR grade after three days of cold storage. Inferior binders were those obtained through oxidation and from a Redwater crude source which lost anywhere from 9°C to 14°C from their low-temperature BBR grades.
- Wax content and a host of other factors affecting the free volume and molecular mobility influence the rate and degree of reversible ageing at low temperatures. Hence, it is unlikely that an all-inclusive theory will ever be able to describe this phenomenon.
- High toughness binders may be able to lessen the severity of low-temperature stress cracking and partially offset the effects of reversible ageing.

## Recommendations

Given the review of the literature and the results presented in this report, the following recommendations are provided:

- The reversible ageing process needs to be accounted for in an improved specification method. The most elegant way in which this could be accomplished is by using the shift factor,  $\mu$ , as first discussed by Struik (1978). Figure 15 shows a plot of the loss in grade temperature versus the isothermal storage time for two samples. A practical way to implement this would be to measure the shift factor for storage times of 20 minutes, 1 hour, 3 hours and 12 hours and to use these to predict the loss in grade temperature by extrapolation after perhaps 72 hours.



**Figure 15. Losses in Grade Temperatures for Oxidized Lloydminster (RR-5L) and Straight Cold Lake (RR-7L) Asphalts as a Function of Isothermal Conditioning Time**

Alternatively, a grading test can be conducted after one or several days of isothermal conditioning at some agreed-upon temperature above the pavement design temperature.

- Carefully designed pavement trials should be constructed to determine if the toughening effect of certain polymer modifiers can partially or completely offset the detrimental effects of reversible ageing at low temperatures.

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**Development of an Improved Asphalt  
Binder Specification Testing Approach  
*Part II. Asphalt Binder Failure Testing***

**October 7, 2005**

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Publication  
Title

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<b>Sponsors:</b>	E.I. du Pont Canada, Imperial Oil of Canada, Ministry of Transportation of Ontario, National Cooperative Highway Research Program – Innovations Deserving Exploratory Analysis, Natural Sciences and Engineering Research Council of Canada
<b>Publication Date</b>	October 7, 2005
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**Abstract**

Part II of this final report documents and discusses an investigation of the fracture performance properties of selected asphalt binders that were used in various pavement trial sections in Alberta and Ontario and additional materials that are sold commercially in Canada and the United States. Ductile fracture properties were determined through the essential work of fracture method using double-edge-notched tension specimens. Brittle fracture properties were determined using single-edge-notched three-point bend specimens. The effects of temperature and rate of loading on fracture properties were investigated. Finally, the compact tension method for the determination of brittle fracture properties was developed.

The ductile fracture properties for a set of Highway 655 binders varied by a significant amount. Essential works of fracture were found to vary by a factor of around 4 while plastic works of fracture varied by a factor of around 10, at low rates and/or high temperatures. These binders had Superpave® grades that ranged from a low of PG 52-34 to a high of PG 64-34. Equally significant differences were found for binders from other pavement trials and commercial sources. Brittle fracture energies for the Highway 655 binders were found to be more consistent which may have been due to the relatively low levels of modification for this set of binders. Other binders tested were found to have a very large range of brittle fracture properties depending on the binder source as well as the modification type and level. Differences in fracture properties were able to explain variations in field cracking severity where rheological results were inconclusive.

Ongoing efforts are focused on determining the ductile failure properties in the respective asphalt mixtures and to develop exceptionally tough binders for further evaluation and use in new pavement trial sections.

**Key Words**

Low-Temperature Binder Grading, Fracture Energy, Essential Work of Fracture, Plastic Work of Fracture, Master Curve, Transverse Cracking, Compact Tension

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## Executive Summary

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This report documents and discusses an investigation of the ductile and brittle state fracture performance properties of selected asphalt binders that were used in various pavement trial sections in Alberta and Ontario and additional materials that are sold commercially in Canada and the United States.

Ductile fracture properties were determined through the essential work of fracture method using the double-edge-notched tension (DENT) geometry. By performing the DENT test on a range of specimens with varying notch depths (i.e., ligament lengths) it is possible to partition the essential work,  $w_e$ , needed to form two new fracture surfaces, from the non-essential plastic work,  $w_p$ , dissipated in areas away from the fracture process zone. This separation of energies is important since in the pavement the fracture process is often much more localized than what it is in the asphalt binder. Hence, a regular direct tension test on the binder would overestimate the performance of some binders more than others and is therefore expected to provide a poor prediction of in-service performance. It is hypothesised that both the essential and plastic works of fracture need to be high to assure good performance. In addition, for binders with equal or similar fracture energies, the ones with low yield stresses are preferred over those with higher yield stresses. The critical crack opening displacement,  $\delta_t$  or CTOD, can be obtained from the ratio of essential work over the net section yield stress,  $w_e/\sigma_y$ , and provides a measure of strain tolerance in the ductile and brittle-to-ductile states in the presence of sharp flaws (cracks). It is believed that this property will be able to provide an improved grading for asphalt binders in terms of fatigue resistance.

The ductile fracture properties for a set of Highway 655 binders varied by a significant amount. Essential works of fracture were found to vary by a factor of around 4 while plastic works of fracture varied by a factor of around 10, at low rates and/or high temperatures. These binders had Superpave® grades that ranged from a low of PG 52-34 to a high of PG 64-34. Equally significant differences were found for binders from other pavement trials and commercial sources. The binders from Highway 655 that showed the highest essential and plastic works of fracture survived the particularly severe winter of 2004 unscathed while those with inferior properties incurred a considerable amount of wheelpath distress.

Brittle fracture properties were determined in single-edge-notched three-point bending (SENB) and in compact tension (CT). The effects of temperature and rate of loading on fracture properties were investigated.

Brittle fracture energies for the Highway 655 binders were found to be more consistent compared to the ductile fracture properties, which may have been due to the low levels of modification for this set of binders. Other binders tested were found to have a very large range of brittle fracture properties depending on the binder source as well as the modification type and level. High fracture energies in the 150-450 J.m<sup>-2</sup> range were obtained for a number

of Cold Lake asphalts modified with linear styrene-butadiene (SBS) diblock and triblock copolymers. The polymers were compatibilized in the binder by reacting the asphaltene and resin phases with a dianhydride to lower their activity and hence promote the swelling of the polymer with the oily phase, allowing for the formation of a continuous three-dimensional network. The network was formed at five percent of SBS by weight of the binder, which is somewhat higher than what was present in the other binders tested.

The CT tests were conducted on specimens with different widths and notch depths. The fracture toughness,  $K_{Ic}$ , was found to be constant, irrespective of the notch depth or specimen width for both straight and modified binder. Consequently, plane-strain conditions were satisfied for the specimen geometries and sizes tested. Fracture energy,  $G_f$ , was found to decrease with notch depth, which was thought to be due to energy absorbing mechanisms away from the crack tip. Deeper notches or an energy correction were able to account for this issue. A slightly different fracture energy,  $J_f$ , was obtained in a more direct fashion from the slope of a plot of the normalized failure energy,  $U_f/B$ , versus notch depth,  $a$ .  $J_f$  provided results close to those obtained with an energy correction from a single notch depth. Reproducibility of the fracture test was found to be good with pooled standard deviations of 5-10 percent for  $K_{Ic}$  and 15-20 percent for  $G_f$ , which is typical for such tests. Given the fact that brittle fracture properties can vary by orders of magnitude for binders of the same Superpave® grade, it is concluded that the test method has a high ability to reveal statistically significant differences in toughness.

# Introduction

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The asphalt binder specification method developed under the SHRP program, now known as AASHTO M320, employs a limit on binder creep stiffness and rate (AASHTO (2002)). An asphalt beam is cooled for one hour at the grading temperature, after which it is loaded in three-point bending to measure the stiffness at 60 seconds,  $S(60)$ , and the slope of the creep stiffness master curve, commonly known as the  $m$ -value, also at 60 seconds,  $m(60)$ . The specification sets an upper limit of 300 MPa on the stiffness and a lower limit of 0.3 on the  $m$ -value. If a binder passes these two criteria then it passes the grading test and can be used in a particular climate where the pavement temperature reaches  $10^{\circ}\text{C}$  below the grading temperature only once in 50 years. The philosophy behind this specification dates back to work from the 1950s and 1960s, when researchers at Shell found a reasonable correlation between the stiffness at a fixed loading time and various failure properties (Van der Poel (1954, 1955), Heukelom (1966, 1969, 1973)).

It is not widely recognized, however, that the correlation made by the Shell researchers were only valid for unmodified binders and that there was a fair degree of scatter. While modifiers were used only sparingly in the 1960s, today the situation is different, in that in some areas nearly half of all binders are modified (Bardesi et al. (1999)). Different modification techniques result in asphalt binders with vast differences in fracture properties in both the brittle state (e.g., Lee and Hesp (1994), Lee et al. (1995), Hoare and Hesp (2000), Anderson et al. (2001a) and others) as well as the ductile state (Andriescu et al. (2004a and 2004b)). Such differences in fracture properties are now believed to explain in part the vast in-service performance differences that are found for binders of the same AASHTO M320 grade (Iliuta et al. (2004a and 2004b)).

A second and perhaps equally important factor on which most low-temperature studies have been largely silent is the fact that almost all binders show some degree of reversible structuring or ageing when stored at cold temperatures. Although this phenomenon had occasionally been discussed in publications from the 1930s (Traxler and Schwyer (1936), Traxler and Coombs (1937)), 1950s (Brown et al. (1957), Brown and Sparks (1958)) and 1970s (Struik (1978)), apparently the publications by Van der Poel (1954, 1955) and Heukelom (1966, 1969, 1973) and those following them make only indirect mention of it. Heukelom (1973) divided asphalt into three subclasses: Type S for bitumens that rendered a straight line on his bitumen test data chart; Type B for blown bitumens; and Type W for waxy bitumens. It is unclear if this was done to address the fact that both the waxy and blown bitumens are particularly prone to the reversible structuring processes that were described by others and therefore failed to give straight lines in Heukelom's bitumen test data chart, or if there were other reasons.

Finally, the approach proposed by the Shell researchers was totally dependent on surrogate properties (penetration, penetration index, ring and ball softening point) to obtain the stiffness (a surrogate property) to predict failure properties (failure strain) at much lower temperatures in the mixture. Most of these studies were done in the laboratory, and therefore it is difficult to determine the veracity of many of the proposed specification schemes.



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# Background on Fracture Grading

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## LOW-TEMPERATURE FAILURE IN ASPHALT PAVEMENTS

Pavements try to shrink when the temperature drops but are prevented from doing so through the friction with the underlying frozen base. Hence, a thermal stress builds up, primarily in the longitudinal direction where the restraint is highest, which increases with a decrease in temperature. At relatively warm temperatures the stress is able to relax but this becomes progressively more difficult when the temperature gets colder and molecular motions become restricted.

At some point the thermal stresses in the pavement reach high enough that damage is sustained. The actual damage process is ill understood but the amount of it is generally thought to depend on a number of factors. The thermal coefficients of the material, the level of friction with the base, the rate of temperature change, the ability of the material to relieve thermal stress, the existing damage in the pavement, and the stiffness of the respective components of the pavement all play an important role in low-temperature performance of asphalt pavements. In a typical road the thermal stresses can easily reach 4-5 MPa during periods of extreme cold weather before the material starts to sustain damage. Whether this damage becomes visible in the form of catastrophic transverse cracks or whether it can be restricted to the accumulation of microcracks is not well understood (Hesp et al. (2000)).

It is generally found that damage starts at the binder-aggregate interface where the triaxial state of stress is highest due to the thermal coefficient mismatch between binder and aggregate. The formation of microcracks has been followed by direct methods such as visual observation and microscopy (Hills (1974), Jacobs (1995), Kim and El Hussein (1995), Shin and co-workers (1996), Radovski (2000)) and by indirect methods such as acoustic emission (Hesp et al. (2000)). The latter method has shown that microcracking can sometimes start by as much as 4-6°C warmer than the temperature at which the highest thermal stress is reached.

The fact that the damage initiates at the coarse aggregate interface indicates that the stiffness mismatch and hence the creep properties should be important parameters that govern to some degree the low-temperature cracking process. However, it is also important to understand what happens prior to and after the microcracking initiates. The fracture properties in the brittle and ductile states have a large influence on how visible cracks and stress concentrations start and progress and hence should be considered in any specification scheme that effectively addresses the distress caused by cold weather. Rheological properties are not solely important but rather both low-strain rheological and high-strain fracture properties need to be better assessed to allow us to improve our ability to predict low-temperature fracture performance.

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## EARLY STUDIES ON GRADING OF ASPHALT BINDERS

Van der Poel (1954, 1955) used penetration and ring and ball softening points to determine the stiffness of binders as a function of loading time and temperature. In one of his early papers, he showed that the failure conditions in the Fraass test could be related to the binder stiffness reaching a critical value after 11 seconds of loading. Based on Van der Poel (1954, 1955) and other unpublished work at Shell Laboratories, Krom and Dormon (1963) were the first to present a binder specification scheme that limits the binder stiffness at specific loading times and temperatures to control cracking due to traffic ( $t = 10^{-2}$  s and low temperatures) and thermal stresses ( $t = 10^4$  s and low temperatures).

Heukelom (1966) went further and tested a wide range of binders for which he found there to be a high correlation between binder stiffness and actual failure properties. Hence, it was suggested that the stiffness, which had become relatively easily accessible through Van der Poel's nomograph, was a good surrogate for the failure properties. Heukelom (1966) concluded that "Van der Poel's stiffness concept has provided a valuable means of simplifying the description of, not only rheological, but also fracture properties of asphalt cements and asphalt mixtures." Following this early work at Shell Laboratories, a large number of other researchers have thus focussed their attention on stiffness as a binder specification parameter at low temperatures (e.g., Hills and Brien (1966), McLeod (1968), Fromm and Phang (1970), Readshaw (1972), Hills (1974), Deme and Young (1987), Anderson and Kennedy (1993), Anderson et al. (1994) and others).

The currently used bending beam rheometer specification test method is based in large part on the pioneering work of the Shell researchers. However, as mentioned previously, the use of modified binders has changed the fracture behaviour to an extent that it is likely no longer correct to simply substitute rheological properties for fracture properties. A more accurate way to grade binders is to measure true failure properties in the presence of sharp cracks to measure resistance to crack initiation and propagation in both brittle and ductile states.

## CURRENT RESEARCH ON FRACTURE GRADING

A little over ten years ago, research at Queen's University started on the fracture performance grading of asphalt binders (Lee and Hesp (1994), Morrison et al. (1995), Lee et al. (1995)). Although bending tests on unnotched binder samples at low-temperatures (e.g., see Heukelom (1966) and others), and on notched mixture samples at all temperatures (e.g., see Ioannides (1997)), had a long history in asphalt research, the use of a sharply-notched binder sample in three-point bending to obtain the plane-strain fracture toughness,  $K_{Ic}$ , and fracture energy<sup>†</sup>,  $G_{Ic}$  or  $G_f$ , had never been adopted for asphalt binders until our effort started in the early 1990s.

These initial efforts were largely focussed on determining the effects of modification on the low-

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<sup>†</sup> Note that  $G_{Ic}$  is used here to indicate the mode I, plane-strain fracture energy or critical strain energy release rate, which is a property that is independent of specimen geometry and size, whereas  $G_f$  is used to indicate a more generic fracture energy, which may or may not be independent of geometry and size but which still reflects the resistance to failure in the presence of a sharp crack.

temperature fracture toughness ( $K_{Ic}$ ) and fracture energy ( $G_{Ic}$  or  $G_f$ ). More recently the scope has expanded to also include an assessment of the ductile essential ( $w_e$ ) and plastic ( $w_p$ ) works of fracture (Andriescu et al. (2004a and 2004b)). This research has revealed that, for binders with nearly identical Superpave® grades, the differences in fracture energies in both brittle and ductile states could be substantial. Brittle state fracture energies that range from 10 to 300 J.m<sup>-2</sup>, and ductile state essential fracture energies that range from 2 to 20 kJ.m<sup>-2</sup>, are all possible depending on the modification technology used (Hoare and Hesp (2000), Roy and Hesp (2001), Andriescu et al. (2004a and 2004b)). The effect of polymer modification comes out clearly in the fracture properties whereas the Superpave® grading method appears to be blind to it. Hence, it has been suggested that a fracture mechanics-based binder grading method would be better at differentiating the good from the not so good binders (Ponniah and Hesp (1996), Hesp (2004a, 2004b), Iliuta (2004a and 2004b)).

An additional significant finding of this work was that the ductile-to-brittle transition temperature depends a great deal on the presence of the notch. Unnotched samples can become brittle nearly 10°C colder than notched samples for some modified binders while for others the difference can be as small as 2°C or 3°C. Hence, the notched test is more discriminating in that it tests the binder under the worst possible scenario.

Following the observation that fatigue fracture can significantly aggravate transverse cracking distress during periods of cold weather, our efforts have recently focused on investigating the ductile fracture properties of asphalt binders at 0°C and also at higher temperatures (Andriescu et al. (2004a and 2004b)). To measure the ductile fracture properties of asphalt binders, the essential work of fracture approach as originally developed by Cotterell and Reddel (1977) based on Broberg's (1968) unified theory of fracture was used with significant success.

The essential work of fracture method is based on the principles of *mechanics* and *conservation of energy* and as such it will likely provide a more accurate approach to grading of asphalt binders for fatigue than for instance force-ductility measurements (e.g., Anderson and Wiley (1976)), tensile tests (Heukelom (1966)), or fatigue tests on binders in necked torsion beams (Pell (1962)), in between cone and plate (Phillips (1999)), or parallel plates (Soenen and Eckman (1999, 2000), Shenoy (2002), Anderson et al. (2001b)), all of which involve a significant number of assumptions or experimental artefacts (e.g., see Anderson et al. (2001)).

An energy analysis during the yielding, necking and tearing processes that occur in ductile failure is used to provide a simple relationship for separating the work of fracture for crack formation from the work associated with plastic deformation away from the fracture zone. Tests on plastics are often done in double-edge-notched tension but other geometries such as three-point bending and single-edge-notched tension have also been used in a limited number of studies (Mai et al. (2000), Hashemi (2003a)). The approach has been successful in the investigation of many ductile materials under various loading rates, when significant flow occurs during failure. The total energy,  $W_t$  (J), determined from the area under the force displacement record, is divided into two. The energy required for the fracture process (i.e., essential fracture energy),  $W_e$  (J), and the energy required for the plastic deformation outside the fracture process zone (i.e., plastic or non-essential fracture energy),  $W_p$  (J):

$$W_t = W_e + W_p \quad [1]$$

The essential work of fracture approach assumes that the essential work term is only surface related and generates new surfaces whereas the plastic component is only volume (i.e., plastic zone) related and may involve a single or perhaps various energy dissipation mechanisms (Cotterell and Redell (1977), Karger-Kocsis (1999)).

As recently discussed by Hashemi (2003b), the method implies a number of assumptions and conditions, of which the most important ones for our work on asphalt are as follows:

- load-displacement diagrams need to be similar in appearance for all ligament lengths (i.e., the data need to show self-similar behavior);
- ligaments need to be fully yielded before cracking initiates (this can be observed from the shape of the load-displacement curve); and
- the volume of the plastic zone is proportional to the square of the ligament length multiplied by sample thickness.

When written in specific terms (i.e., energy per unit surface area), equation [1] becomes:

$$W_t = w_e BL + \beta w_p BL^2 \quad [2]$$

or, after rearrangement,

$$w_t = w_e + \beta w_p L \quad [3]$$

where

$w_t$  = specific total work of fracture ( $J/m^2$ );

$w_e$  = specific essential work of fracture ( $J/m^2$ );

$w_p$  = specific plastic work of fracture ( $J/m^3$ );

$B$  = sample thickness (m);

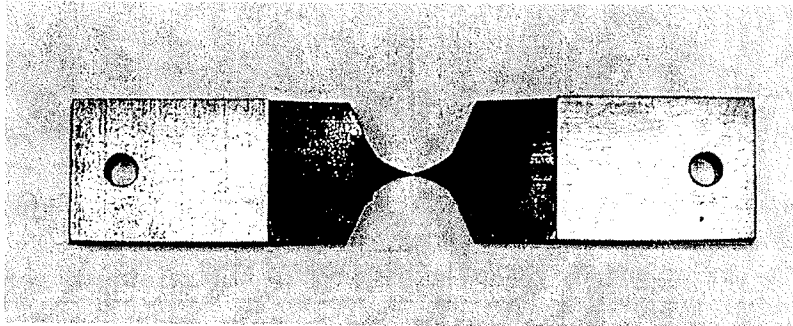
$L$  = ligament length (m); and

$\beta$  = scaling factor describing the shape of the plastic zone (Mai et al. (2000)).

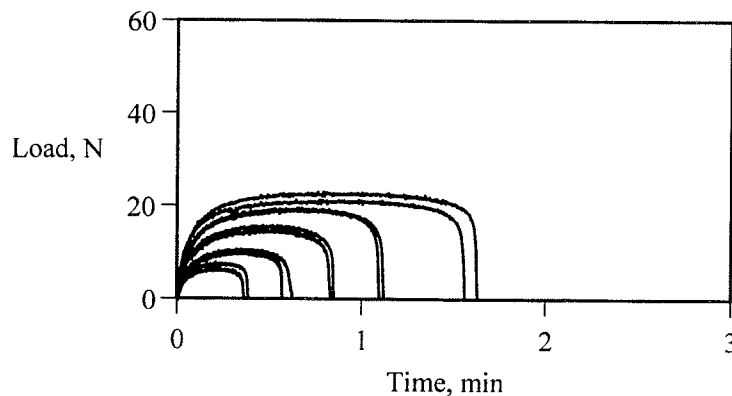
A plot of the total specific fracture work,  $w_t$ , versus ligament length,  $L$ , will thus result in a straight line of which the intercept equals  $w_e$  and the slope equals  $\beta w_p$ . It is acknowledged that for thin sheets (i.e., plane stress conditions apply), the specific essential work of fracture changes with the thickness of the material.

Figure 1 shows a failed sample in which a characteristic elliptical shape for the yielded zone around the crack surface is visible. The curves in Figure 2 provide the force-displacement data for a set of five duplicate samples of a modified binder (ten tests in total). Figure 3 shows the analysis with the specific total works of fracture plotted as a function of ligament length for a number of modified binders (Equation [3]). The slopes and intercepts for this set of binders are given in Figure 4. The results show that the above stated assumptions are met for the testing of asphalt binders at 25°C. The

curves are self-similar and the data fit the equation from the essential work of fracture method. Hence, for this reason we have now started to investigate this approach further in order to include it into our improved low-temperature asphalt binder specification method.



**Figure 1. Asphalt Binder Sample after Failure in the Double-Edge-Notched Tension Test (Andriescu et al. (2004a))**

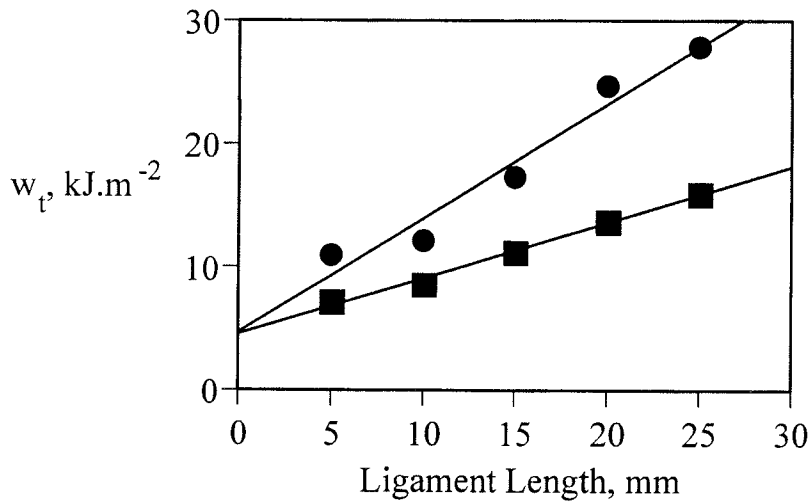


**Figure 2. Typical Reproducibility for Double-Edge-Notched Tension Test.**

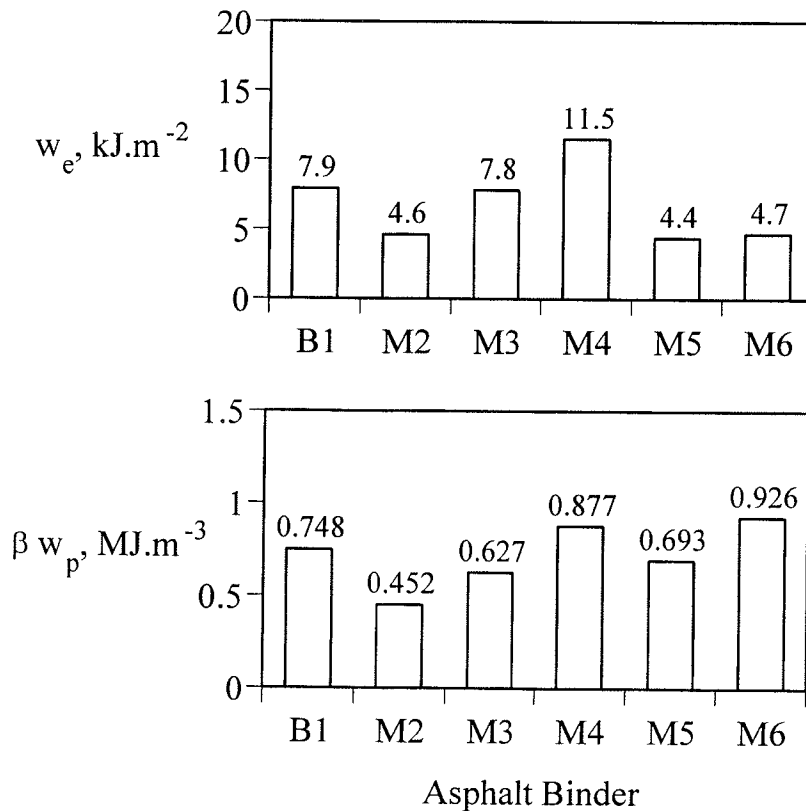
Data for an SB diblock modified asphalt binder of PG 70-34 grade.

Reproduced from Andriescu et al. (2004a).

If asphalt binders are tested in a force-ductility test on an unnotched sample or in between parallel plates in a rheometer then some may show very good performance due to the absence of a sharp notch but the essential work of fracture may still be low leading to problems in the road. It should be realized, however, that only future well-designed pavement trials can properly guide us to set appropriate specification limits for these new test methods. These limits will depend on the traffic level, pavement design, climate, and maybe as yet poorly understood factors.



**Figure 3. Specific Total Work of Fracture versus Ligament Length for Two Binders with Equal Essential but Different Plastic Works of Fracture (Andriescu et al. (2004a))**



**Figure 4. Essential and Plastic Works of Fracture for Set of Straight and Modified Asphalt Binders (Andriescu et al. (2004a))**

This report documents and discusses an investigation of the fracture performance properties of selected asphalt binders that were used in various pavement trial sections in Ontario and Alberta and additional materials that are sold commercially in Canada and the United States.

# Experimental

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## MATERIALS

The materials investigated in this project were obtained from various sources.

The seven original binders used in the C-SHRP test road in Lamont, Alberta were obtained from Sierra Transportation Engineers in Reno, Nevada, who operates the SHRP Materials Reference Library (RR-2L, RR-3L, RR-4L, RR-6L, and RR-7L), and from Dr. Ludo Zanzotto at the University of Calgary (RR-1L and RR-5L).

Two of the three original binders used in the C-SHRP test road in Hearst, Ontario were no longer available, and hence field-aged binders were extracted from core samples that were taken after 12 years of service. Binder samples were extracted with a minimum of four tetrahydrofuran (THF) solvent washings. The bulk of the solvent was removed by rotary evaporation at moderate temperatures until no more THF was visibly distilled. In order to assure that no further solvent remained in the samples, rotary evaporation was continued for an additional two hours at a temperature of 150°C.

Seven binders were obtained from the most recently constructed low-temperature pavement trial north of Timmins, Ontario. The binders were sampled during the paving of each test section from the line used to transfer the material from their storage tank into the asphalt plant.

Seven SHRP MRL binders were investigated for their resistance to fracture at low and ambient temperatures. These binders had been thoroughly investigated during the SHRP program and hence provided a ready source of widely available asphalts of varying quality.

The last group contains oxidized and wax-modified binders that were obtained from commercial sources and were investigated according to our procedures for their ability to resist fracture.

A listing of all binders and their pertinent information is given in Table 1. The binder codes as given were those used by the respective sources.

## EXPERIMENTAL METHODS

### Laboratory Chemical Ageing

All binders were aged according to standard protocol. Aging was performed in both the rolling thin film oven (RTFO) and in the pressure aging vessel (PAV) according to AASHTO T240-97 and PP1-98 methods prior to testing for low-temperature properties.



**Table 1. Pertinent Properties of Binders Investigated for Fracture Performance**

Binders	Crude Source(s)	Modification	Grades†
<u>Lamont, AB</u>			
RR-1L	Boundary Lake	Oxidized	80/100 A, PG 58-22
RR-2L	Montana/Bow River	Unmodified	150/200 B, PG 52-28
RR-3L	Cold Lake	Unmodified	300/400 A, PG 46-34
RR-4L	Redwater	Unmodified	80/100 C, PG 58-22
RR-5L	Lloydminster	Oxidized	80/100 A, PG 64-28
RR-6L	Lloydminster	Unmodified	150/200 A, PG 52-28
RR-7L	Cold Lake	Unmodified	200/300 A, PG 52-34
<u>Hearst, ON</u>			
RR-1H	Venezuelan	Unmodified	150/200 A, PG 52-33
RR-2H	Lloydminster	Unmodified	150/200 A, PG 52-33
RR-3H	Bow River	Oxidized	200/300 B or A*, PG 46-37
<u>Timmins, ON</u>			
655-1	Lloydminster	RET	PG 64-37
655-2	Unknown	Oxidized/SBS	PG 64-35
655-3	Unknown	SBS	PG 64-36
655-4	Unknown	SBS	PG 64-35
655-5	Unknown	SBS	PG 64-35
655-6	Unknown	Oxidized	PG 58-35
655-7	Unknown	Unmodified	PG 52-35
<u>SHRP MRL</u>			
AAA-2	Lloydminster	Unmodified	200/300, PG 46-37
AAC-2	Redwater	Unmodified	AC-5, PG 52-27
AAE	Lloydminster	Oxidized	60/70, PG 70-22
AAG-2	California Valley	Unmodified	AR-2000, PG 58-22
AAK-1	Boscan	Unmodified	AC-30, PG 64-28
AAL	Cold Lake	Unmodified	150/200, PG 58-33
AAN	Bow River	Unmodified	85/100, PG 58-28
<u>Various Companies</u>			
A-1	Lloydminster	Wax	PG 58-34
B-1	Unknown	Oxidized	PG 58-34
B-2	Unknown	Oxidized	PG 64-28
B-3	Unknown	Oxidized	PG 70-28
C-1	Unknown	Oxidized	PG 58-34

Note: RET = reactive ethylene terpolymer of ethylene, butyl acrylate, and glycidyl methacrylate; SBS = styrene-butadiene-styrene linear block copolymer; and A, B, and C refer to three different commercial sources of the listed binders. † The grade information is as given by EBA (1994), Robertson (1995), and Gavin (2003). \* This binder graded as either a Group A or B depending on which viscosity was used in the Bitumen Test Data Chart (EBA (1994)).

### Brittle State Fracture Testing

The plane-strain low-temperature fracture toughness,  $K_{Ic}$ , and fracture energy,  $G_f$ , were determined in single-edge-notched three-point bending (SENB) and in compact tension (CT).

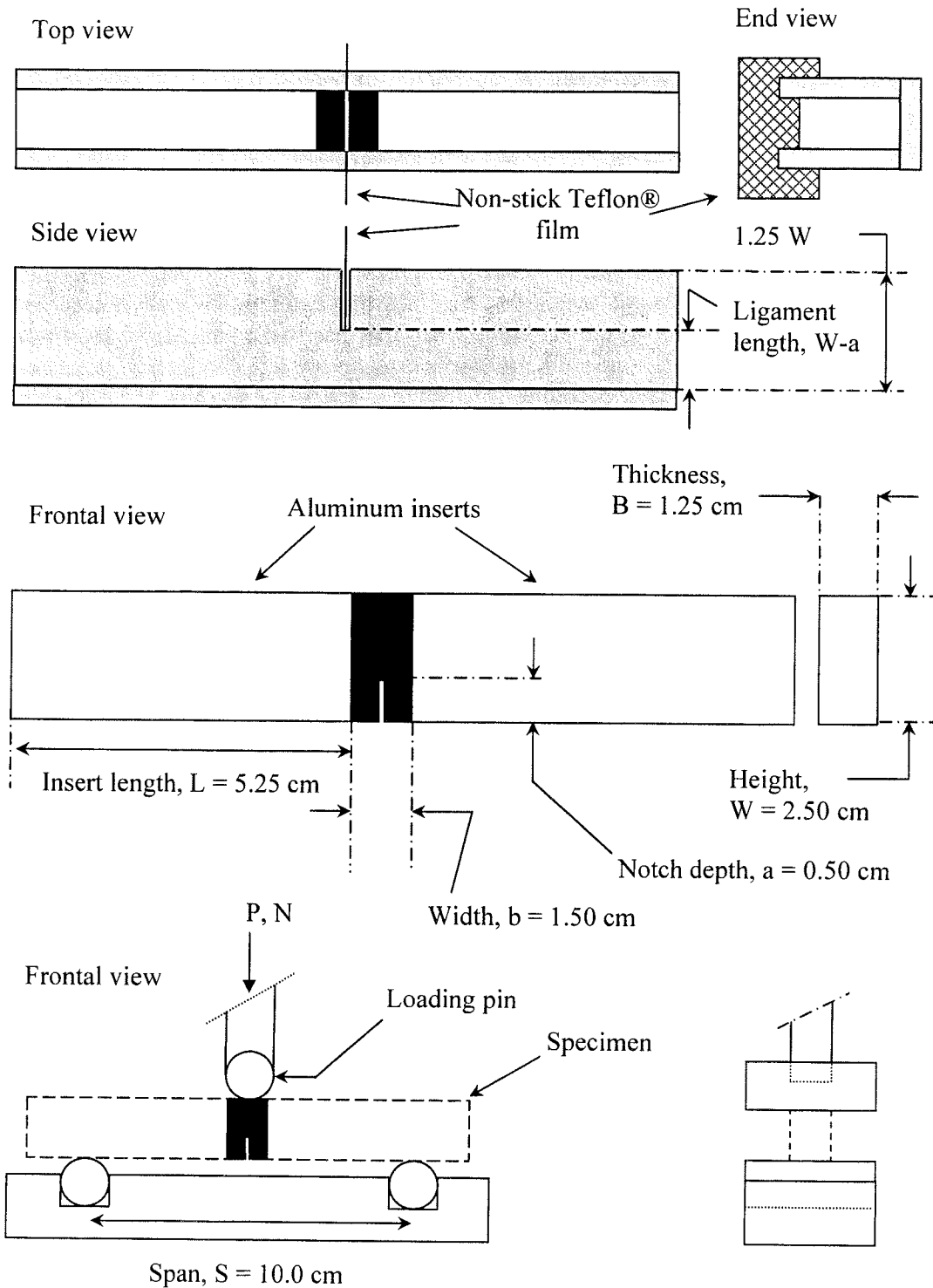
The SENB fracture properties of the binders were determined according to procedures outlined in *LS-296 (draft) – Asphalt Cement Grading for Fracture Performance using Single-Edge-Notched Bend Procedure* (MTO (2005a)). In brief, samples were cast with the aid of Teflon® release film in between aluminum inserts that were held in place by support spacers. A 5-mm deep and 25- $\mu$ m wide notch was introduced in the asphalt part of the beam by inserting a straight Teflon® sheet across the mold prior to pouring the binder in the cavity between the inserts. Schematics of the mold and sample are provided in Figures 5 (top) and 5 (middle), respectively. The binder was slowly cooled over a one-hour period after which the mold was placed in a freezer for one hour. Molds were then disassembled and the specimens stored for one day prior to testing.

Conditioned samples were tested at a range of temperatures that included their brittle-to-ductile transition. A schematic of the setup is provided in Figure 5 (bottom). The test software accurately integrated the load versus displacement curve to provide a measure of fracture energy according to standard procedure. Samples were tested at varying rates of loading in order to assess the effect of loading rate.

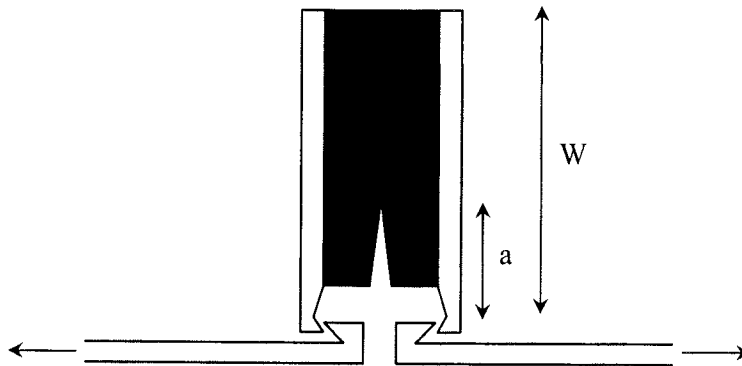
The CT fracture test is now described in *LS-298 (draft) – Asphalt Cement Grading for Fracture Performance using Compact Tension Procedure* (MTO (2005b)), which assesses a binder's fracture toughness, energy, and crack tip opening displacement, on a notched specimen in compact tension (CT). The compact tension test was performed using Instron's AsphaltPro 5525 machine. The instrument was equipped with a 500 N load cell, which was high for this application since failure loads were mostly below 100 N. The temperature bath was able to hold the test temperature constant to within approximately 0.1°C. For a detailed account of the development of this method the reader is referred to Edwards and Hesp (2006).

A schematic of the set up and the sample geometry is provided in Figure 6. The tensile tester was fitted with two special grips with integral knife edges that fit into the aluminum end pieces of the asphalt specimens. This way of testing is similar to that described in ASTM method *E 1304-97 Standard Test Method for Plane-Strain (Chevron-Notch) Fracture Toughness of Metallic Materials* (ASTM (2005)).

Samples were prepared with the aid of silicone molds in which the inserts were placed before the samples were poured to the geometry of Figure 6. A set of samples was sharpened prior to testing by sliding a razor blade through the notch. However, most samples were tested as obtained directly from the molds. The loading rate in each test was kept constant at 0.01 mm/s while the temperature was varied. Samples were conditioned for 20 hours before testing.



**Figure 5. Single-Edge-Notched Three-Point Bend Fracture Test for Asphalt Binders: Mold Design (top); Sample Dimensions (middle); and Three-Point Bend Test Setup (bottom)**



**Figure 6. Compact Tension Test Setup, Specimen Geometry and Critical Variables**  
( $a$  = notch depth, angle of notch =  $30^\circ$ ,  $W$  = specimen height) (Edwards and Hesp (2006))

### Ductile State Fracture Testing

The essential work of fracture,  $w_e$ , and plastic work of fracture,  $w_p$ , were determined in double-edge-notched tension (DENT). The fracture properties of the binders were determined according to procedures outlined in *LS-299 (draft) - Asphalt Cement Grading for Fracture Performance using Double-Edge-Notched Tension Procedure* (MTO (2005c)), which assesses a binder's essential and plastic works of fracture,  $w_e$  and  $w_p$ , as well as the critical crack tip opening displacement,  $\delta_t$  or CTOD, in double-edge-notched tension.

In brief, samples were poured in silicone molds with aluminum inserts for ready attachment to the test frame. The notch depth in the double-edge-notched tension sample was varied to provide ligament lengths of 5, 10, 15, 20, and 25 mm. Figure 1 provides a photograph of a sample after failure which illustrates the elliptical shape of the ductile zone.

Samples were tested in duplicate and the total works of fracture,  $W_t$ , were used to determine the specific total works of fracture,  $w_t$ , by dividing through the cross sectional area for each ligament,  $LB$ . The specific total work of fracture was plotted against the ligament length to provide a measure of the essential and plastic works of fracture according to equation (3). The essential and plastic works of fracture were determined at temperatures of 0, 12.5, 25, and 37.5°C, and loading rates ranging from 0.1 to 3,000 mm/min. The resulting data were shifted to form ductile fracture energy master curves for each binder. The shift factors obtained by forming a smooth fracture energy curve at 0°C were compared with the shift factors obtained to produce the rheological master curves. For a detailed discussion on the development of this test method, and the rheological characterization of these binders, the reader is referred to the doctoral dissertation of Andriescu (2006).

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# Results and Discussions

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## LAMONT, ALBERTA TEST ROAD MATERIALS

A detailed analysis of the performance of the Lamont, Alberta C-SHRP test road is provided in a previous report on reversible ageing in these and other binders (Hesp (2004c)). The findings as discussed in the report indicated that the reversible ageing phenomenon can explain almost entirely the anomalies found with regards to the expected versus predicted transverse cracking performance according to the current AASHTO M320 specification. However, this was a trial with only straight and air blown binders and not a single polymer-modified test section. Hence, fracture properties are expected to vary less than for materials in which tough polymer modifiers are incorporated. Nevertheless, this part of our study considers the fracture properties for the Lamont binders in both their brittle and ductile states in order to verify the accuracy of the entire binder grading approach.

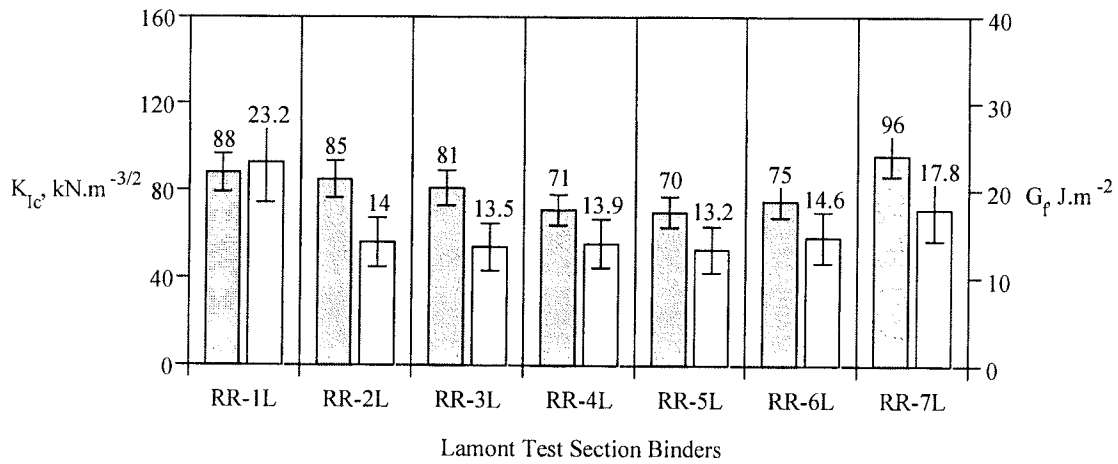
The seven binders from the C-SHRP trial in Lamont, Alberta, which was constructed in 1991, were tested for both their brittle as well as ductile fracture properties. The binders were aged according to standard RTFO and PAV procedures. Samples were conditioned for 24 hours at the test temperature for both the SENB and DENT tests.

The low temperature fracture properties were determined at the temperature at which the AASHTO M320 limiting temperature is reached,  $T_{\text{grade}}$ , and a second temperature that was 6°C warmer,  $T_{\text{grade}} + 6$ . The loading rate in the SENB test was kept constant at 0.01 mm/s. The results are presented in Figures 7 and 8.

The ductile fracture properties were determined at both 0°C and 1 mm/min loading rate and at 25°C and 100 mm/min loading rate. The results are presented in Figures 9 and 10.

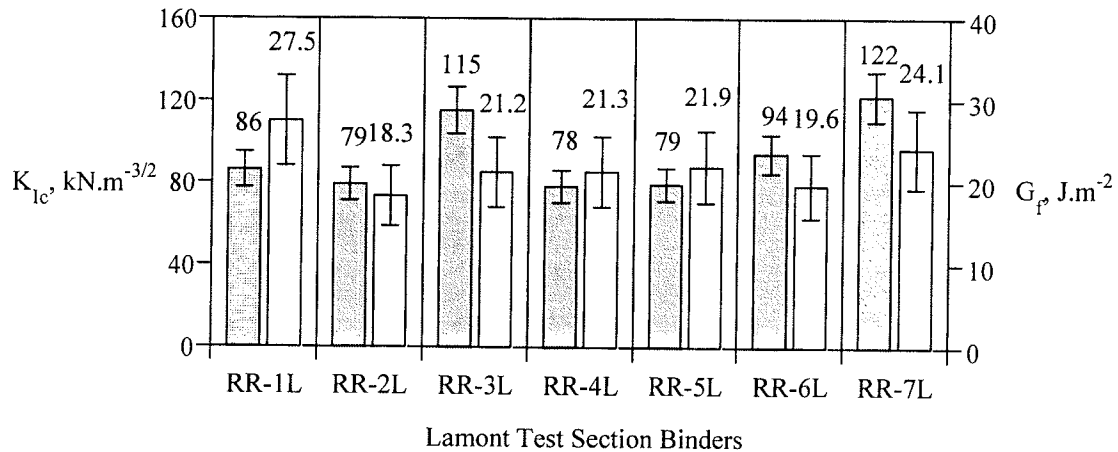
The fracture testing results for the Lamont binders are consistent with the observed field performance as documented in the previous report on reversible ageing in asphalt binders. It was shown that the field performance correlated to a high degree with the limiting stiffness and m-value temperatures as determined after 24 and 72 hours of isothermal conditioning. Hence, this suggests that the fracture properties for this set of binders do not vary by enough to alter the field performance from what is predicted by an extended BBR test. A range of 25°C in the 3-day BBR grades for a set of unmodified binders easily explains the differences in transverse stress cracking performance for this trial without having to resort to the analysis of fracture data.

Figures 7 and 8 show that at the grade temperatures and slightly above, the fracture energies of the binders do not vary by a large amount. There are only small differences between binders in the fracture toughness while the fracture energies are the same within the experimental error.



**Figure 7. Brittle State Fracture Energy Data for Seven Binders used in the Lamont, Alberta Pavement Trial**

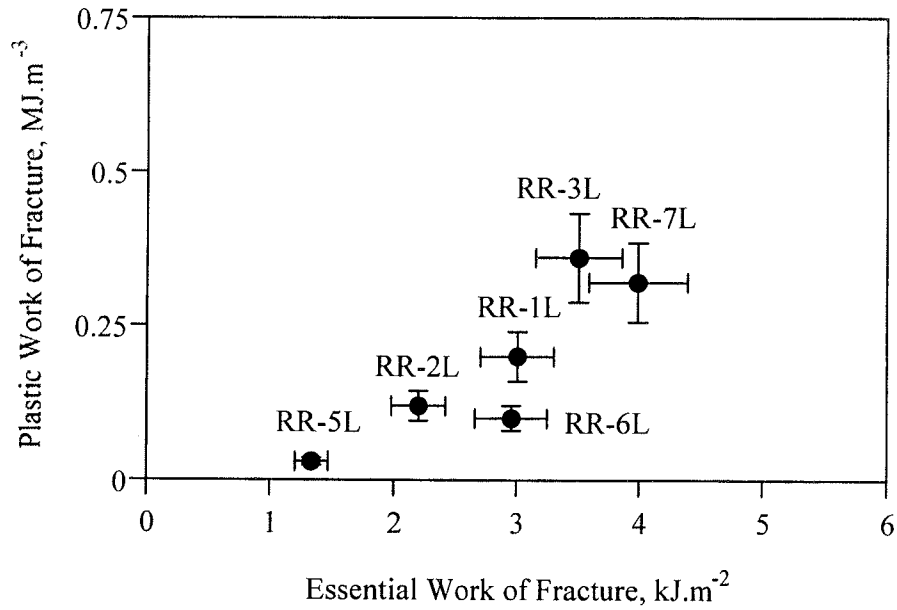
Tested at their PG temperature in SENB at a rate of 0.01 mm/s. Samples were conditioned for 24 hours at the test temperature. Estimated errors are  $\pm 10\%$  for fracture toughness,  $K_{Ic}$ , and  $\pm 20\%$  for fracture energy,  $G_f$ .



**Figure 8. Brittle State Fracture Energy Data for Seven Binders used in the Lamont, Alberta Pavement Trial.**

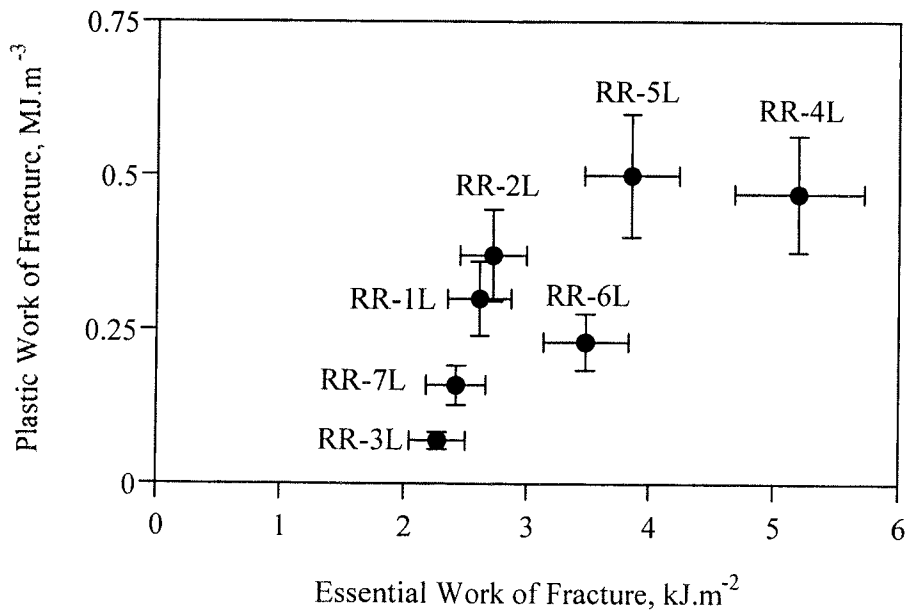
Tested  $6^\circ\text{C}$  above their PG temperature in SENB at a rate of 0.01 mm/s. Samples were conditioned for 24 hours at the test temperature. Estimated errors are  $\pm 10\%$  for fracture toughness,  $K_{Ic}$ , and  $\pm 20\%$  for fracture energy,  $G_f$ .

Figures 9 and 10 indicate that the ductile fracture properties are consistent with the observed field performance. The essential and plastic works of fracture for both the Cold Lake binders (sections RR-3L and RR-7L) are high at  $0^\circ\text{C}$ , which explains in part their superior performance after 12 years in service. Their fracture resistance is low at  $25^\circ\text{C}$  suggesting that at this temperature fatigue could not have been a serious form of distress for these two sections.



**Figure 9. Ductile Works of Fracture for Binders of the Lamont, Alberta Pavement Trial**

Tested at 0°C and a rate of 1 mm/min using the DENT geometry. Samples were conditioned for 24 hours at 0°C. Note that sample RR-4L was too brittle to be tested. Estimated errors are  $\pm 10\%$  for the essential work of fracture,  $w_e$ , and  $\pm 20\%$  for plastic work term,  $\beta w_p$ .



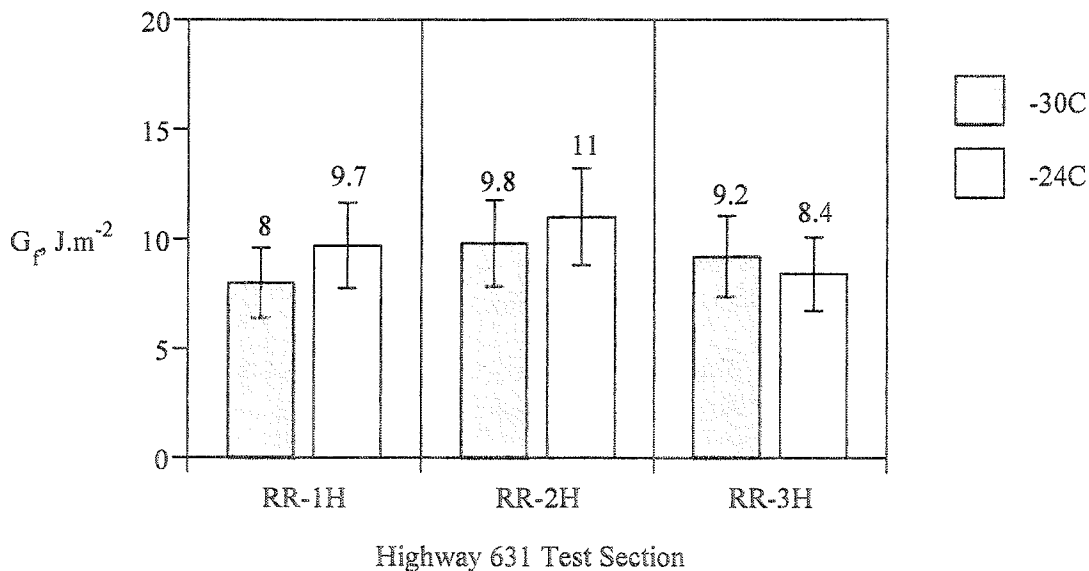
**Figure 10. Ductile Works of Fracture for Binders of the Lamont, Alberta Pavement Trial**

Tested at 25°C and 100 mm/min using the DENT geometry. Samples were conditioned for 24 hours at 25°C. Estimated errors are  $\pm 10\%$  for the essential work,  $w_e$ , and  $\pm 20\%$  for the plastic work term,  $\beta w_p$ .

## HEARST, ONTARIO TEST ROAD MATERIALS

A detailed analysis of the performance of the Hearst, Ontario C-SHRP test road is provided in a previous report on reversible ageing in these and other binders (Hesp (2004c)) as well as in a paper by Iliuta et al. (2004b). The findings indicated that the effects of chemical and reversible ageing fail to explain the difference in cracking severity between Sections RR-2H and RR-3H with 49 and 74 cracks, at performance grades of  $-26.0^{\circ}\text{C}$  and  $-28.6^{\circ}\text{C}$ , respectively, after three days of conditioning. Both binders suffer equally from reversible ageing so there must be other factors to explain the difference in performance.

The analysis of the fracture properties in both the brittle and ductile states suggests that these must have had some impact on the level of transverse cracking. Brittle fracture energies at  $-30^{\circ}\text{C}$  and  $-24^{\circ}\text{C}$  as well as ductile fracture energies at  $0^{\circ}\text{C}$  and  $25^{\circ}\text{C}$  were determined in order to further understand the observed distress levels. The results of the analysis are discussed in detail in the abovementioned sources of which a short review is given herein. Figures 11 and 12 provide the brittle and ductile fracture energies, respectively, for the three Hearst binders as recovered from field cores after 12 years of service.

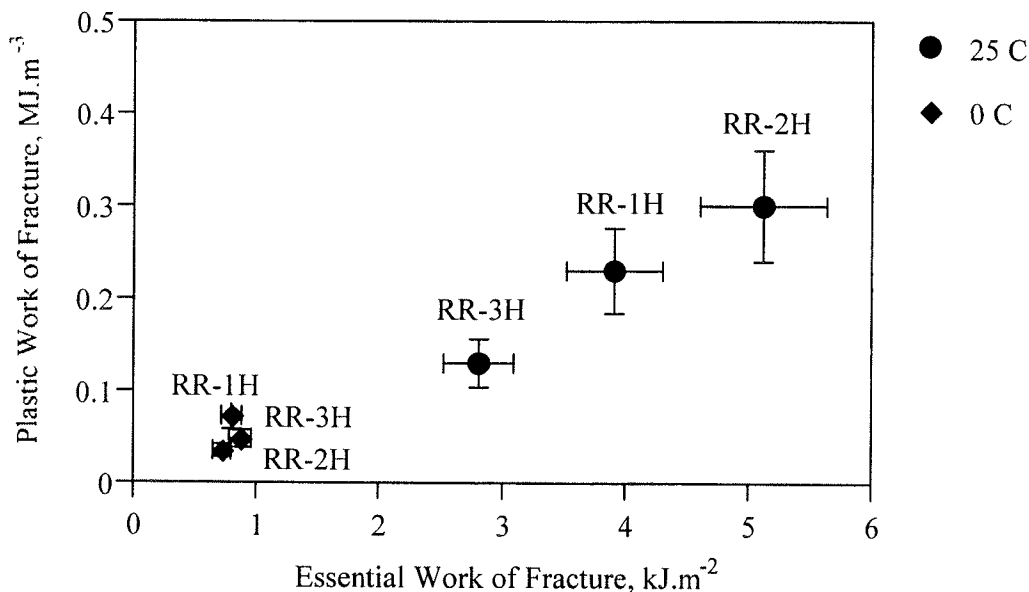


**Figure 11. Brittle State Fracture Energy Data for Three Binders Recovered from the Hearst, Ontario C-SHRP Trial**

Tested at  $-30^{\circ}\text{C}$  and  $-24^{\circ}\text{C}$  in SENB at 0.01 mm/s. Samples were conditioned for 24 hours at the test temperature. Estimated errors are  $\pm 20\%$  for fracture energy,  $G_f$ .

The brittle fracture energies are identical within the error for all three sections. Hence, this provides no explanation for the field performance differences. However, it does appear that these fracture energies are less than half of those of the PAV-aged materials from the Lamont trial (see Figures 7 and 8). This shows that the PAV is unable to accurately replicate the field ageing.





**Figure 12. Ductile Works of Fracture for Binders used in the Hearst, Ontario Trial**

Tested at 25°C and 100 mm/min and at 0°C and 0.1 mm/min using the DENT geometry. Samples were conditioned for 24 hours at the test temperature. Estimated errors are  $\pm 10\%$  for the essential work,  $w_e$ , and  $\pm 20\%$  for the plastic work term,  $\beta w_p$ .

The ductile fracture energies do differ by a significant amount. Section RR-2H, with the lowest number of cracks, has the highest essential and plastic works of fracture. Section RR-3H, with the lowest ductile fracture properties, has nearly 50 percent more cracks than section RR-2H, even though it has a performance grade that is 2.6°C lower and hence would be expected to have been exposed to a significantly lower number of damaging cold days.

RR-1H has ductile fracture properties in between those of the other two but it has lost a significant amount of performance due to both physical and chemical ageing with a recovered performance grade of -20°C after 12 years in service and three days of conditioning at low temperatures. This major difference in low temperature grade can largely explain the severe distress in this section.

## COCHRANE, ONTARIO TEST ROAD MATERIALS

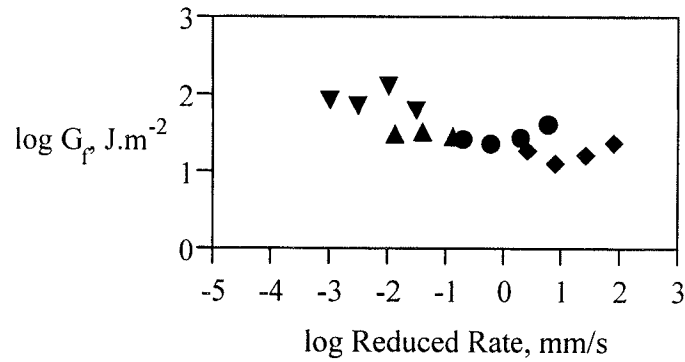
The Cochrane, Ontario pavement trial was constructed on Highway 655 some 65 km north of Timmins, Ontario specifically for this research project in late 2003. The six asphalt suppliers for this trial were chosen based on the anticipation that they would be able to deliver exceptionally tough materials. Research on fracture toughening that started in our laboratory in the mid 1990s has shown that it is possible to produce binders of the exact same Superpave® grade with fracture energies that range from a low of 5 J.m<sup>-2</sup> to a high of 300 J.m<sup>-2</sup>. It was our desire to have at least one or two of the trial sections on Highway 655 constructed with a binder of superior toughness. However, as will be discussed here, this did not entirely happen partially due to the inability of the industry to understand

the factors that make a tough binder. Further, polymer contents currently used to produce PG 64-34 binders are unlikely ever going to provide the superior toughness that is desirable. Asphalt suppliers are currently only rewarded for meeting the Superpave® grade at the lowest possible cost. Hence, the use of air-blown, gelled and acid-modified binders is widespread and there do not appear to be many companies that use innovative materials and techniques to obtain exceptional performance.

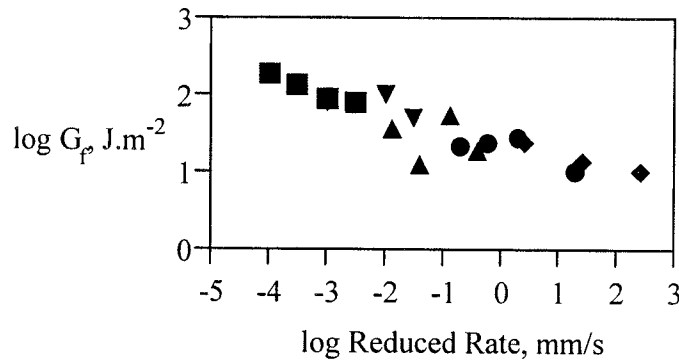
The brittle state fracture properties for the Highway 655 binders were determined at a minimum of four different temperatures and four different rates of loading in order to construct fracture energy master curves. Master curves give a better picture of how fracture performance varies with rate (or time) and temperature. The fracture energy curves at each temperature were shifted in an attempt to form a smooth overall fracture energy master curve. Figures 13-19 provide the master curves for the fracture energies in the brittle state for all seven binders used in the Cochrane trial.

Figure 20 provides the fracture energy at a single rate of 0.01 mm/s and various temperatures for all seven binders. A close inspection of the data suggests that nearly all fracture energies are within the 5-100 J.m<sup>-2</sup> range, except for the binders from Sections 3 and 4 that reach somewhat higher values at temperatures around -16°C. This finding was somewhat disappointing since in prior work it had been possible to produce binders with fracture energies in the 200-300 J.m<sup>-2</sup> range at temperatures as low as -30°C with SBS loadings of 5 weight percent (e.g., Roy and Hesp (2001)). After these results were obtained inquiries were made with the suppliers and it turned out that the highest polymer concentration used was 4 weight percent in the binder for Section 655-5. The binder used for Section 655-3 was only modified with 2 percent polymer. Obviously, for reasons that are unclear, our instruction to put in as much modifier as possible was not followed by any of the suppliers. Fortunately, the Ministry of Transportation of Ontario has approved second and third pavement trials in which this problem will be circumvented by specification of higher grades and/or polymer contents.

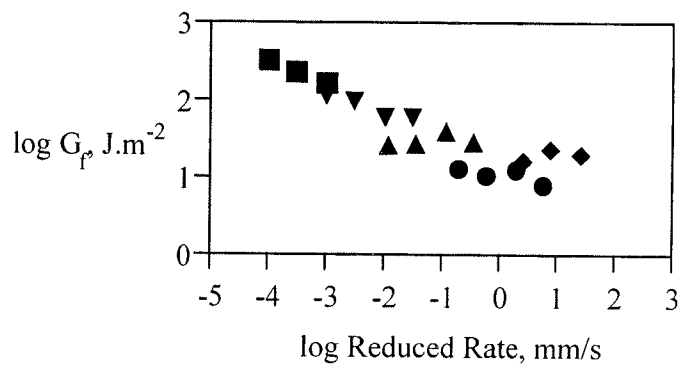
The data from Figures 13-19 show that the behavior of most binders is complex since shifting of the fracture data does not result in the formation of entirely smooth master curves. While the binder from Section 655-5 approaches simple behavior, the one from Section 655-4 is definitely complex. The complexity most likely arises from phase changes within the polymer-modified binder at low temperatures. At warmer temperatures the polymer may become more soluble and thereby toughen the material to a greater extent than what occurs at low temperatures. Further, air-blown binders, such as those used in Sections 655-2 and 655-6, are known to show shear thinning behavior which could manifest itself as lower works of fracture at higher temperatures. This observation has ramifications for the implementation of a simple performance-based specification method.



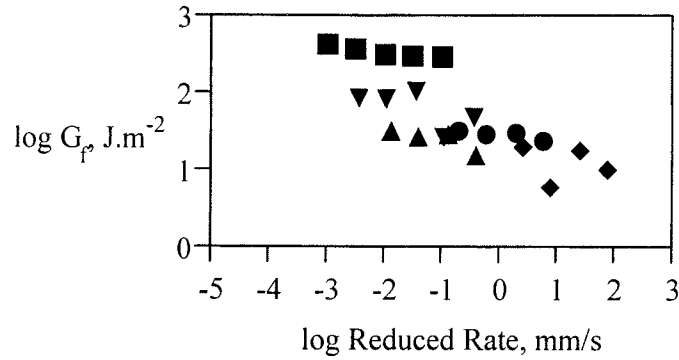
**Figure 13. Fracture Energies for Binder Used in Section 655-1 of the Cochrane Trial**  
 Tested using the SENB geometry ( $\nabla = -34^{\circ}C$ ;  $\blacktriangle = -28^{\circ}C$ ;  $\bullet = -22^{\circ}C$ ; and  $\blacklozenge = -16^{\circ}C$ ).



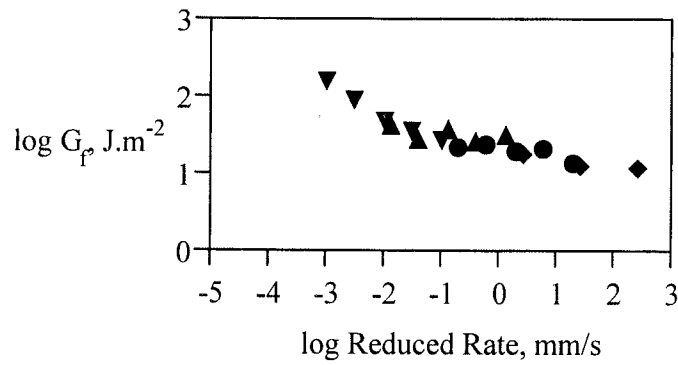
**Figure 14. Fracture Energies for Binder Used in Section 655-2 of the Cochrane Trial**  
 Tested using the SENB geometry ( $\blacksquare = -34^{\circ}C$ ;  $\nabla = -28^{\circ}C$ ;  $\blacktriangle = -22^{\circ}C$ ; and  $\bullet = -16^{\circ}C$ ;  $\blacklozenge = -13^{\circ}C$ ).



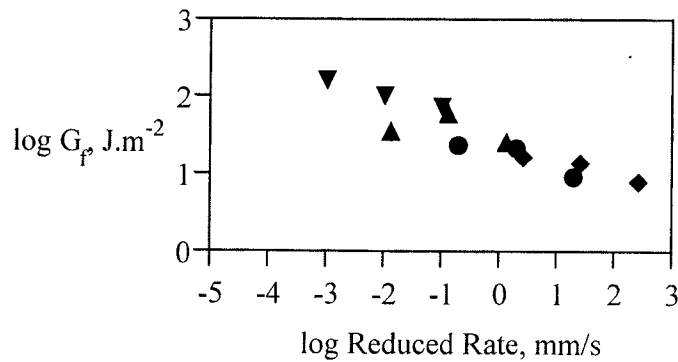
**Figure 15. Fracture Energies for Binder Used in Section 655-3 of the Cochrane Trial**  
 Tested using the SENB geometry ( $\blacksquare = -34^{\circ}C$ ;  $\nabla = -28^{\circ}C$ ;  $\blacktriangle = -22^{\circ}C$ ; and  $\bullet = -16^{\circ}C$ ;  $\blacklozenge = -13^{\circ}C$ ).



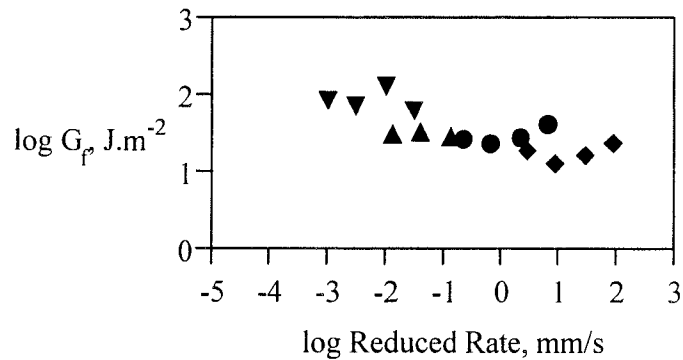
**Figure 16. Fracture Energies for Binder Used in Section 655-4 of the Cochrane Trial**  
 Tested using the SENB geometry (■ = -34°C; ▼ = -28°C; ▲ = -22°C; and ● = -16°C; ◆ = -13°C).



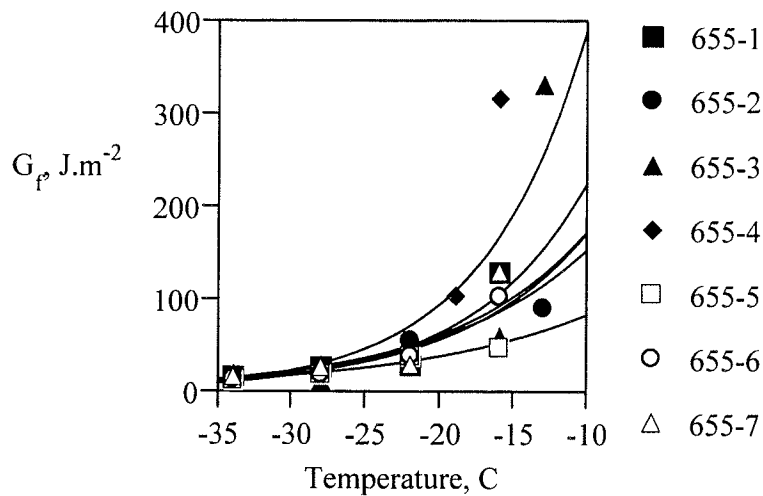
**Figure 17. Fracture Energies for Binder Used in Section 655-5 of the Cochrane Trial**  
 Tested using the SENB geometry (▼ = -34°C; ▲ = -28°C; ● = -22°C; and ◆ = -16°C).



**Figure 18. Fracture Energies for Binder Used in Section 655-6 of the Cochrane Trial**  
 Tested using the SENB geometry (▼ = -34°C; ▲ = -28°C; ● = -22°C; and ◆ = -16°C).



**Figure 19. Fracture Energies for Binder Used in Section 655-7 of the Cochrane Trial**  
Tested using the SENB geometry (▼ = -34°C; ▲ = -28°C; ● = -22°C; and ◆ = -16°C).



**Figure 20. Fracture Energies at 0.01 mm/s as a Function of Temperature for Binders used in the Cochrane, Ontario Trial Tested using the SENB Geometry**

Essential and plastic works of fracture for these seven binders were determined in their ductile state using the double-edge-notched tension (DENT) geometry. The aged binders were tested in the DENT test at four different temperatures and at three to five different rates of loading. The individual essential works of fracture (EWFs) and plastic works of fracture (PWFs) master curves are provided in Figures 21-27.

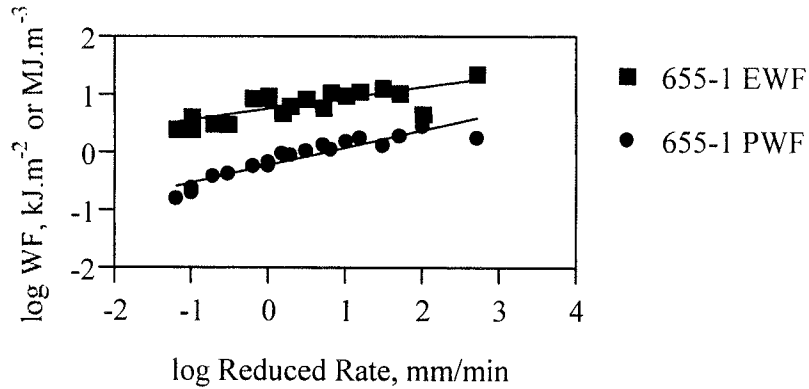


Figure 21. Essential and Plastic Works of Fracture for Binder Used in Section 655-1 of the Cochrane Trial, Tested Using the DENT Geometry

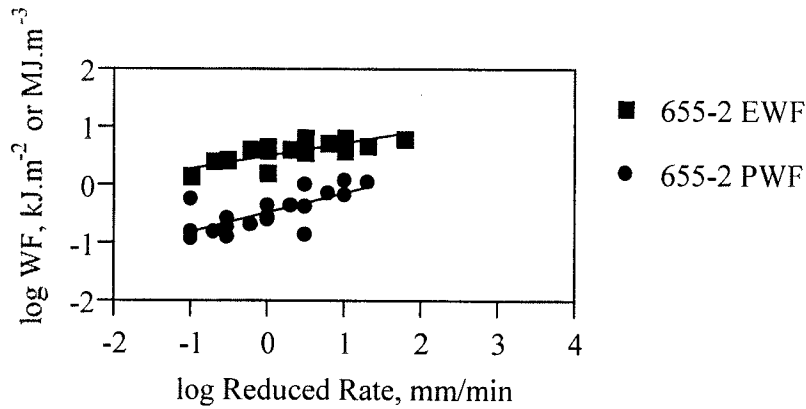


Figure 22. Essential and Plastic Works of Fracture for Binder Used in Section 655-2 of the Cochrane Trial, Tested Using the DENT Geometry

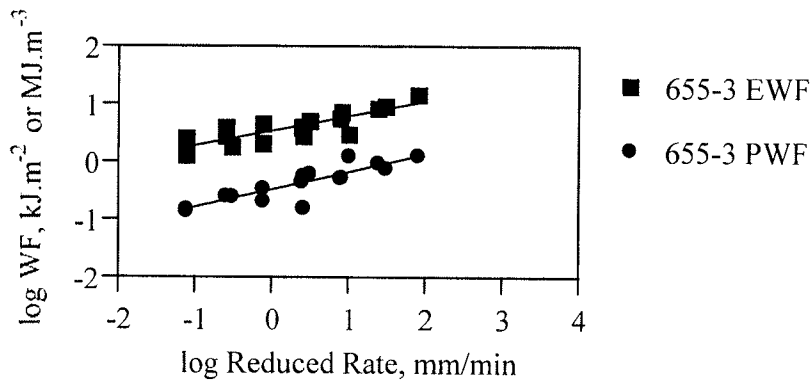


Figure 23. Essential and Plastic Works of Fracture for Binder Used in Section 655-3 of the Cochrane Trial, Tested Using the DENT Geometry

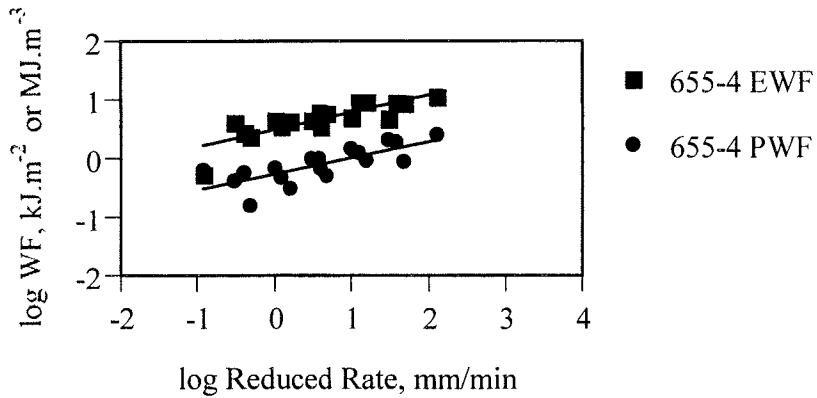


Figure 24. Essential and Plastic Works of Fracture for Binder Used in Section 655-4 of the Cochrane Trial, Tested Using the DENT Geometry

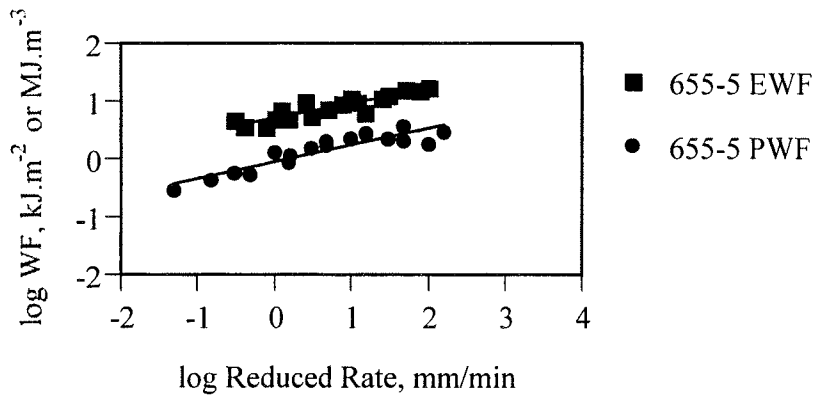


Figure 25. Essential and Plastic Works of Fracture for Binder Used in Section 655-5 of the Cochrane Trial, Tested Using the DENT Geometry

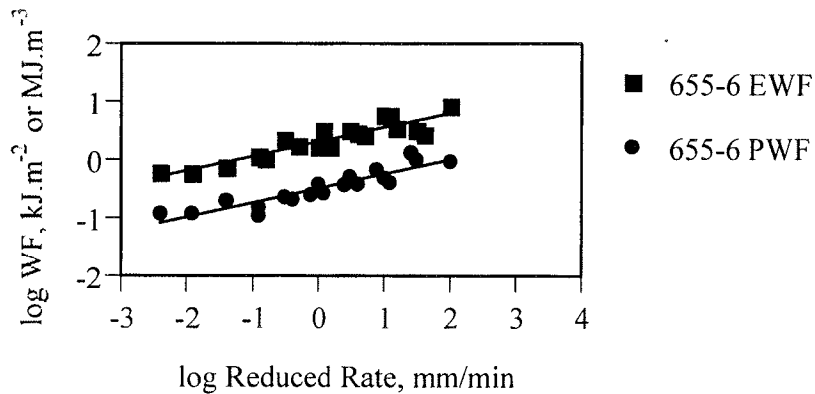
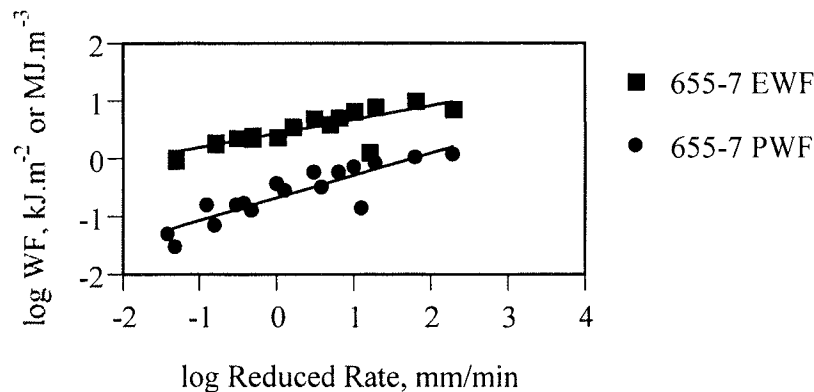


Figure 26. Essential and Plastic Works of Fracture for Binder Used in Section 655-6 of the Cochrane Trial, Tested Using the DENT Geometry



**Figure 27. Essential and Plastic Works of Fracture for Binder Used in Section 655-7 of the Cochrane Trial, Tested Using the DENT Geometry**

The data show that under ductile conditions the works of fracture can be shifted to form reasonably smooth master curves. Hence, it appears that the same mechanisms operate during the failure process irrespective of the temperature and rate of loading. In this temperature range no anomalous phase transitions appear to take place that would have provided complex graphs.

In order to provide a somewhat easier comparison between the ductile fracture data for the seven binders, the essential and plastic works of fracture at 0.1 mm/min and 0°C are plotted in Figure 28. This graph shows that there are significant differences in both the essential and plastic works of fracture for the different materials. The binders used in Sections 655-1 and 655-5 show superior ductile fracture properties and this was reflected in the field performance as discussed in the previous report.

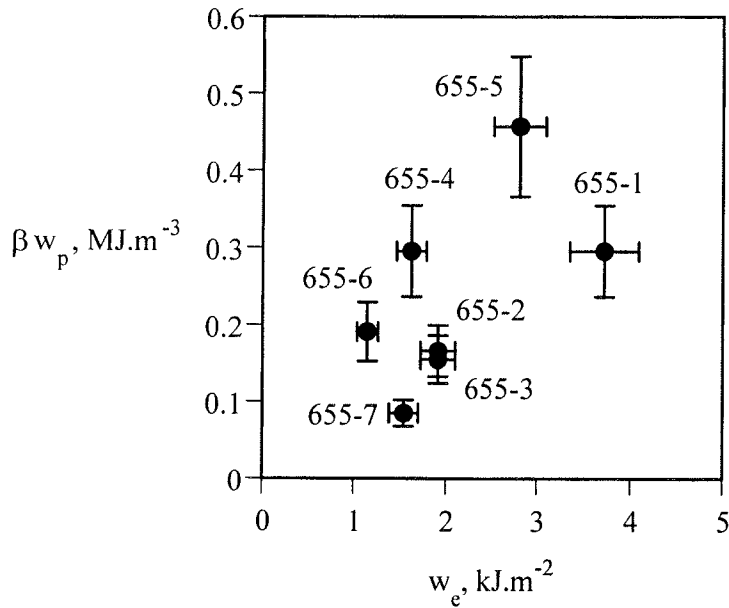
Section 655-5 reversible aged by a moderate amount but still shows no cracking today, which is likely due to the high toughness in the ductile state. Sections 6 and 7 also show little or no cracking despite their low ductile fracture properties but this is probably due to the lower voids contents in these two sections (Iliuta et al. (2004b)). Sections 2, 3, and 4 showed the most cracking which is due to their low ductile fracture properties and their tendency to reversibly age during cold storage.

Figure 29 shows the essential work of fracture and critical crack opening displacement ( $CTOD = w_e/\sigma_y$ ) at 25°C and 100 mm/s, which is again consistent with the observed field performance. However, this data shows that the binder of section 655-5 is clearly superior from a fatigue perspective with both the essential work of fracture *and* the crack opening displacement being the highest for this set of binders. This superior performance is likely due to the high polymer content. The section 655-1 material appears to have obtained a high essential work of fracture from a high stiffness/strength which lowers the critical crack opening displacement and hence is expected to be less favorable compared to the performance of section 655-5.

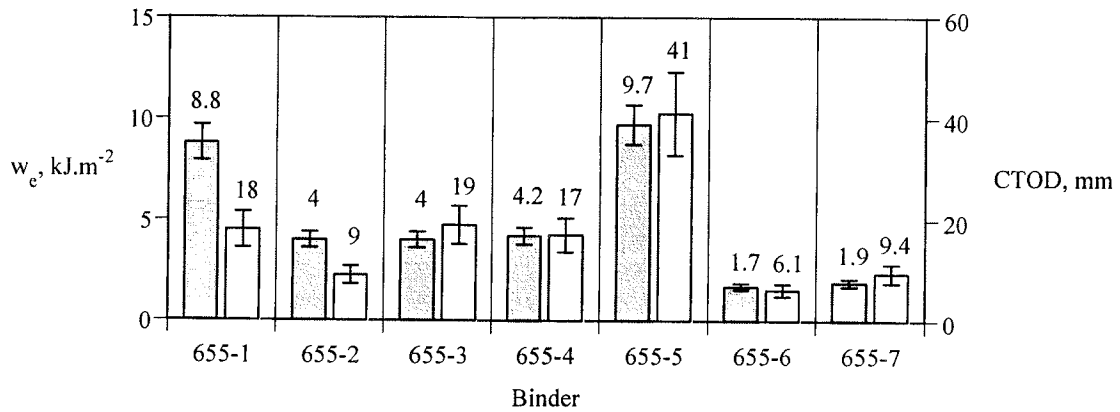
It is clear from these early performance data that the developed fracture grading approach is consistent with the field performance in all cases studied thus far. In later years deviations may occur depending on the rates at which these binders chemically age and how this differs from the way the



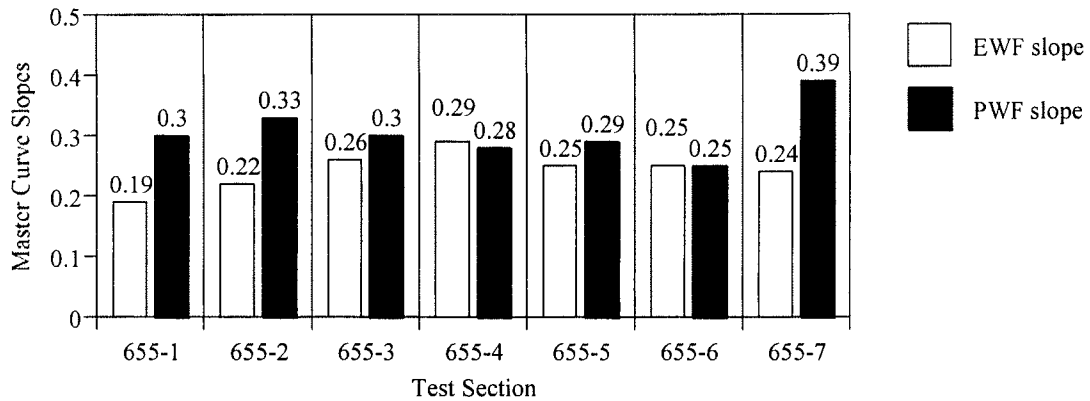
materials were aged in the RTFO and PAV in this study.



**Figure 28. Ductile Essential and Plastic Works of Fracture for the Seven Binders Used in the Cochrane trial, Tested using the DENT Geometry at 0°C and 0.1 mm/min**  
 Estimated errors are ± 10 % for the essential work,  $w_e$ , and ± 20 % for the plastic work term,  $\beta w_p$ .



**Figure 29. Ductile Essential Works of Fracture and Critical Crack Opening Displacement (CTOD =  $w_e/\sigma_y$ ) for the Seven Binders Used in the Cochrane Trial, Tested Using the DENT Geometry at 25°C and 100 mm/min**  
 Estimated errors are ± 10 % for the essential work (shaded bars),  $w_e$ , and ± 20 % for the critical crack opening displacement (open bars), CTOD. Note: Tensile yield stresses were approximated by the net section yield stresses for the shortest ligament length of 5 mm.

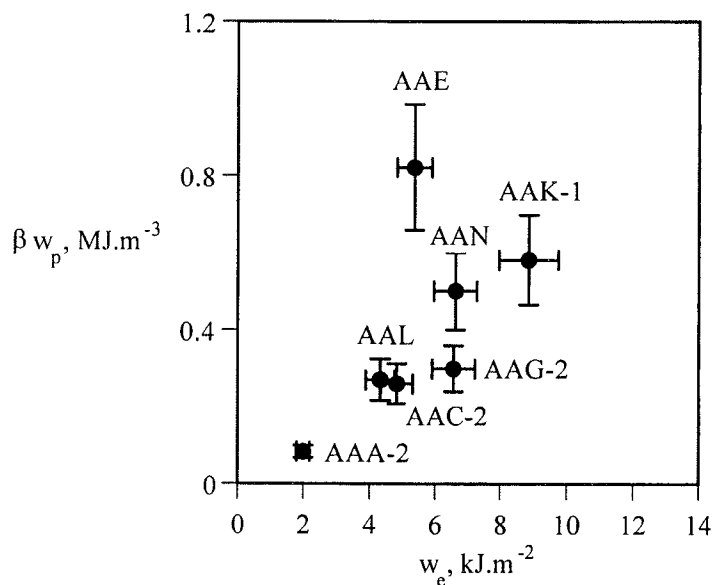


**Figure 30. Slopes of the Essential and Plastic Works of Fracture Master Curves for the Seven Binders Used in the Cochrane Trial**

Figure 30 shows the slopes of the individual master curves for both the essential and plastic works of fracture. The data shows that there are no large differences between these seven binders. Since the slope indicates the way in which a binder approaches the brittle state it would be beneficial to have low slopes associated with high fracture energies. The analysis shows that only Section 655-1 has the best balance in this respect. However, if other binders with higher thermal sensitivity were to be tested the slopes may change much beyond what was found in this study and hence the issue of what rate and temperature of testing is best chosen for binder grading becomes more difficult to settle.

## SHRP MRL MATERIALS

A number of the SHRP MRL binders were tested in order to obtain more insight into how the fracture properties change depending on the binder composition. The data are presented in Figure 31 for the ductile state and Figures 32 and 33 for the brittle state.

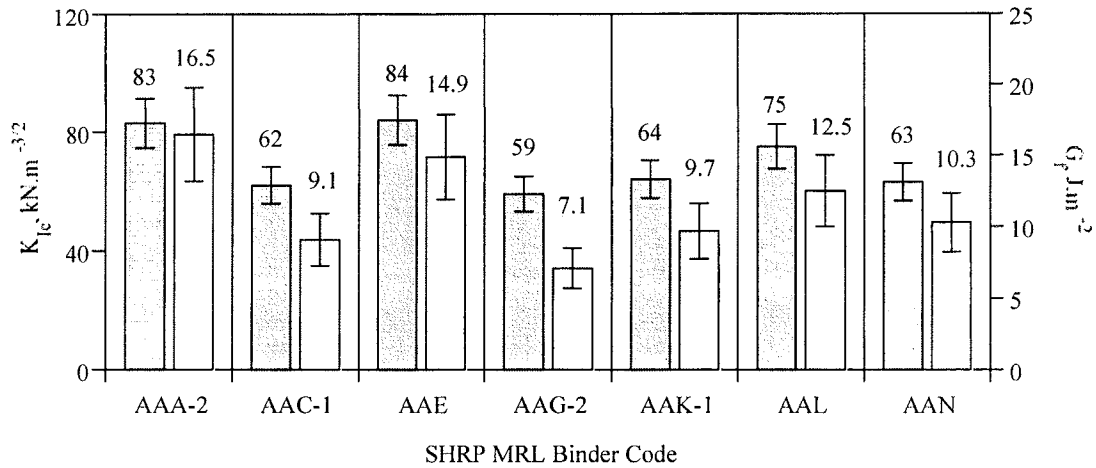


**Figure 31. Ductile State Fracture Energy Data for Selected SHRP MRL Binders, Tested Using the DENT Geometry at 25°C and 100 mm/min**

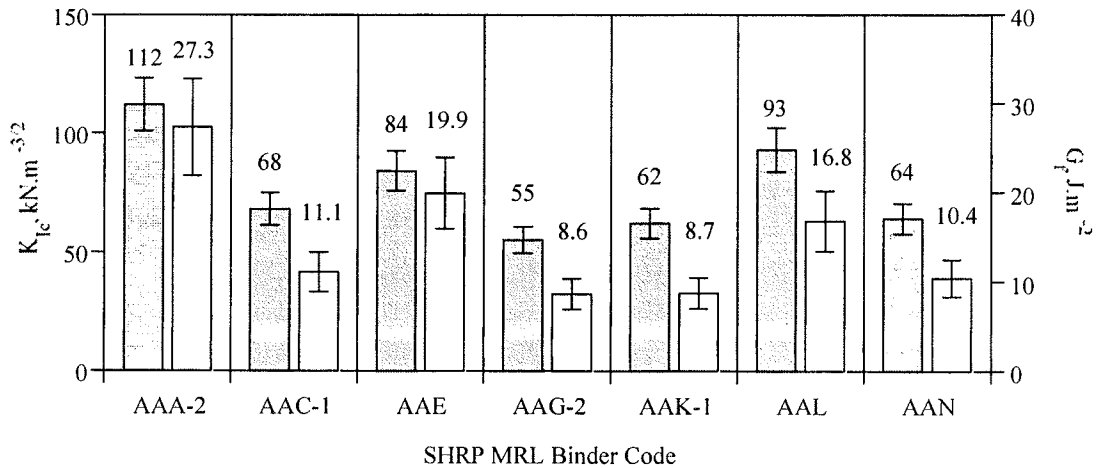
Estimated errors are  $\pm 10\%$  for the essential work,  $w_e$ , and  $\pm 20\%$  for the plastic work term,  $\beta w_p$ .

The ductile state works of fracture show that harder binders have essential and plastic works of fracture that are higher than those of softer binders. The one oxidized binder appears to have a plastic work of fracture that is quite high but it may also indicate that at 25°C and 100 mm/min it is close to the brittle state. Hence, these data need to be interpreted with caution and usually it is safe to say that binders of different grades need to be compared only to judge gross differences.

The brittle fracture energies are all very similar and not very different from those determined for the Lamont, Alberta C-SHRP binders (see Figures 7 and 8). Possible exceptions are binders AAC-1, AAG-2 and AAK-1, which could be due to their high wax, low asphaltene and high asphaltene contents, respectively.



**Figure 32. Brittle State Fracture Energy Data for Binders Used in the SHRP Materials Reference Library, Tested at their PG Temperature in SENB at a Rate of 0.01 mm/s**  
 Samples were conditioned for 24 hours at the test temperature. Estimated errors are ± 10 % for fracture toughness,  $K_{Ic}$ , and ± 20 % for fracture energy,  $G_f$ .

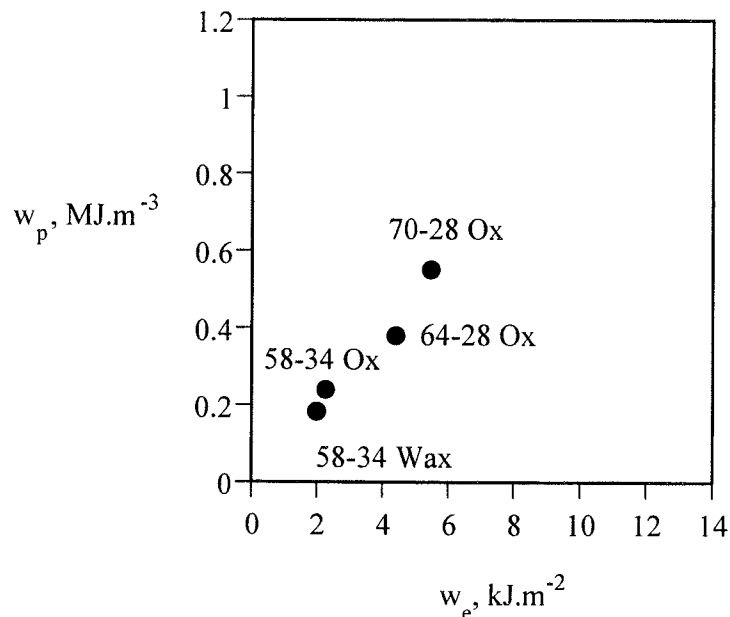


**Figure 33. Brittle Fracture Energy Data for Binders Used in the SHRP Materials Reference Library, Tested 6°C above their PG Temperature in SENB at a Rate of 0.01 mm/s**  
 Samples were conditioned for 24 hours at the test temperature. Estimated errors are ± 10 % for fracture toughness,  $K_{Ic}$ , and ± 20 % for fracture energy,  $G_f$ .

## WAX-MODIFIED AND OXIDIZED MATERIALS

The wax modified and oxidized binders were tested because there is a suspicion in the industry that such materials perform poorly yet are able to obtain very respectable SHRP Superpave® grades due to their modification methods.

Figure 34 provides the ductile fracture properties for these binders in the same scale as for the previous sets of ductile fracture data. The brittle state properties of these binders were not determined. However, because these materials are known to suffer significantly from reversible aging processes it is reasonable to expect that the brittle fracture properties will also deteriorate in time.



**Figure 34. Ductile State Works of Fracture for Selected Oxidized and a Single Wax Modified Asphalt Binder, Tested Using the DENT Geometry at 25°C and 100 mm/min**

Estimated errors are  $\pm 10\%$  for the essential work,  $w_e$ , and  $\pm 20\%$  for the plastic work term,  $\beta w_p$ .

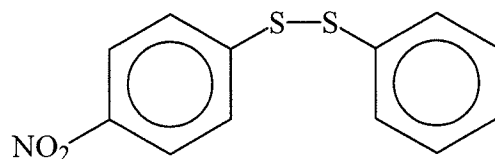
The ductile works of fracture show once more that at 25°C and 100 mm/min the harder binders show higher works of fracture. This makes sense since the harder binders are able to build up more strength and thereby increase the work of fracture. However, what this figure does not show is how close or far the binders are from their ductile-to-brittle transition. Had these binders been tested at higher rates and/or lower temperatures then perhaps the ranking would have been different. This once more reinforces the observation that it is very important to consider all factors when grading binders for fracture performance.

## NEW BINDERS WITH IMPROVED TOUGHNESS

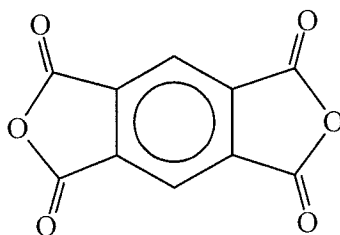
A small part of this study was focused on the development of improved asphalt binders. A Cold Lake base asphalt of 300/400 penetration grade was obtained for this purpose from the Strathcona, Alberta refinery of Imperial Oil of Canada. This base asphalt was chosen, as it is known to provide a challenge in terms of polymer modification and hence toughening. Due to its moderately high asphaltene and resins contents, most styrene-butadiene type polymers fail to dissolve in this binder unless sophisticated chemistry is employed. However, Cold Lake bitumen also has one of the lowest tendencies to reversibly age harden and therefore provides a potentially valuable starting point for producing superior performing modified binders.

The methods that were considered to compatibilize 5 weight percent of styrene-butadiene-styrene linear triblock copolymer (Kraton D-1192 SBS) or 5 weight percent of styrene-butadiene diblock copolymer (Finaprene 1205) in this project included the use of (1) regular sulfur (S), (2) 4-nitrophenyl disulfide (NPDS), and (3) 1,2,4,5-benzenetetracarboxylic anhydride (DANH) as compatibilizers. The structures of the latter two compounds are given below. The sulfur was added either prior to or along with the polymer modifier. The dianhydride was added either alone or with a small amount of polyphosphoric acid (PPA). The nitro compound is thought to associate with the asphaltene fraction of the asphalt and hence deliver the sulfur radicals where they are most needed. The dianhydride is thought to react with the asphaltene fraction which then partially or completely precipitates and hence this makes the polymer more compatible with the oily phase.

4-Nitrophenyl disulfide (NPDS)

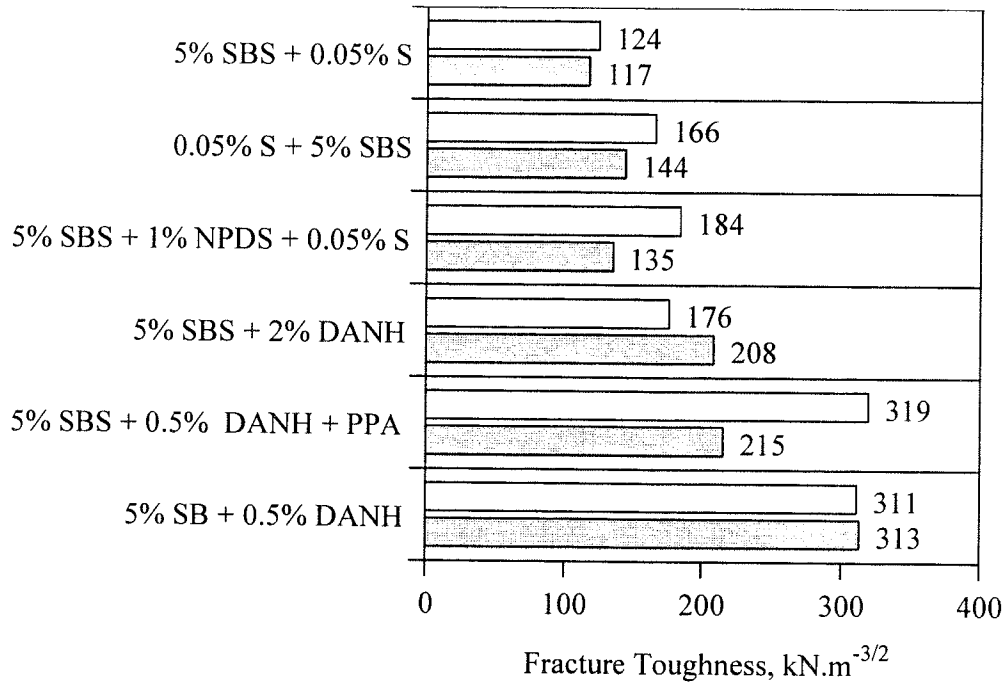


1,2,4,5-Benzenetetracarboxylic anhydride (DANH)



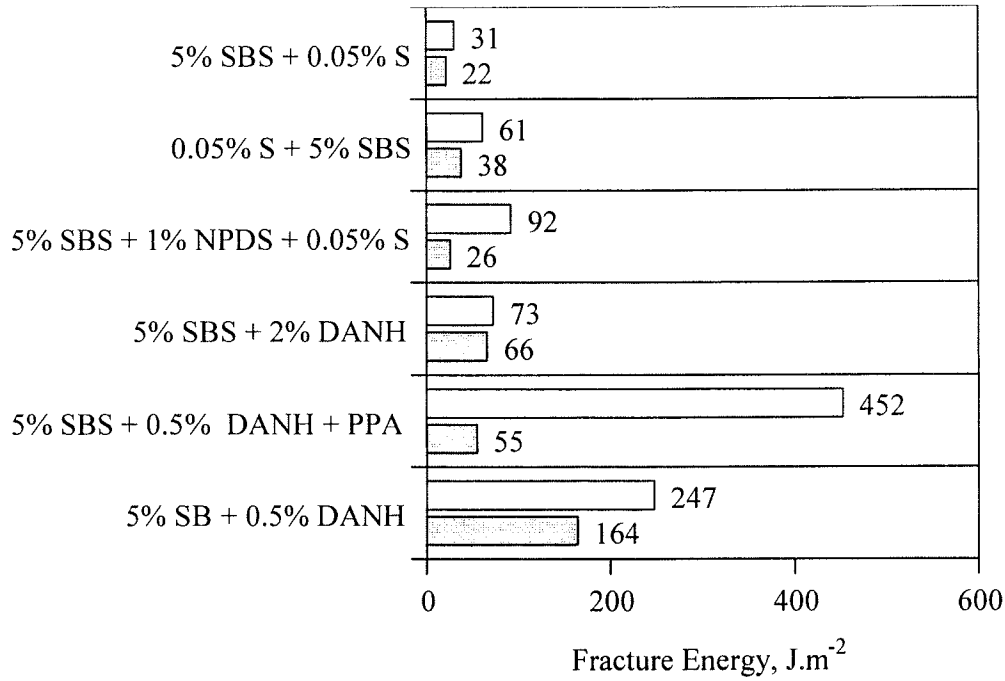
**Figure 35. Structures of Compatibilizers Investigated for Incorporating SBS into Cold Lake 300/400 Base Asphalt**

The stiffness,  $E$ , fracture toughness,  $K_{Ic}$ , and fracture energy,  $G_f$ , were determined at both  $-32^\circ\text{C}$  and  $-38^\circ\text{C}$  since the base asphalt grades as a PG 46-38. The results are provided in Figures 36-38. The data shows that the fracture energy in the brittle state can vary by a great deal for binders with identical polymer content and nearly identical stiffness. This validates our earlier findings that binders of the same grade can show a vast range of toughness and this is likely going to show up as differences in performance in service. For a detailed discussion of these results the reader is referred to the doctoral dissertation of Iliuta (2006).

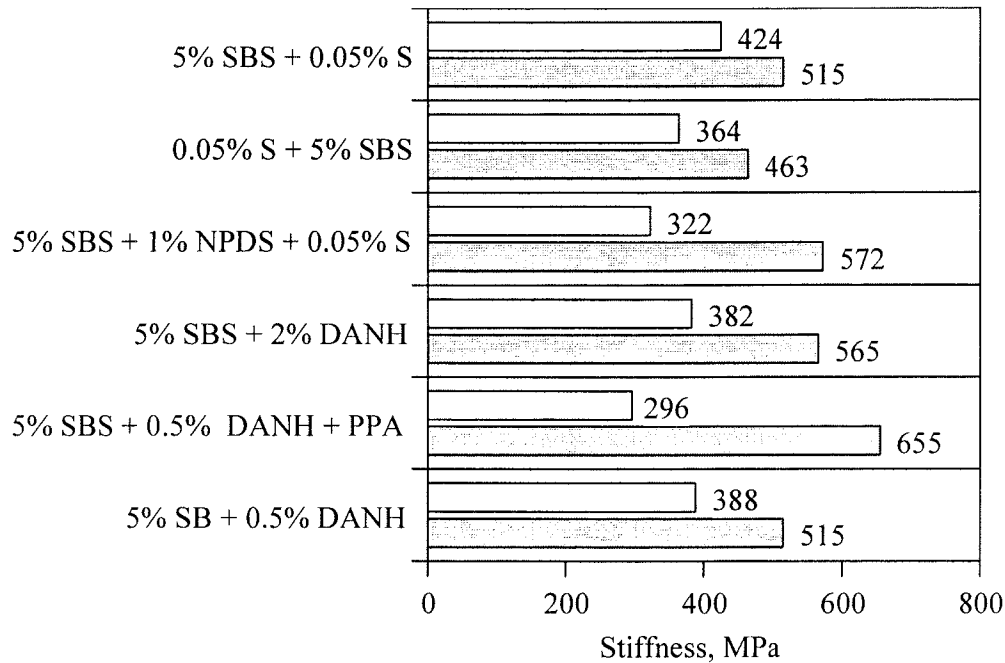


**Figure 36. Brittle State Fracture Toughness for Investigated Binders**  
(open bars give toughness at  $-32^\circ\text{C}$  while shaded bars give toughness at  $-38^\circ\text{C}$ )

Fortunately, the Ministry of Transportation of Ontario has approved a third pavement trial north of Timmins, Ontario on Highway 655. This trial will be designed with a number of test sections that contain high polymer contents in the same Cold Lake 300/400 base asphalt. Long term pavement monitoring will allow us to determine if the high toughness in the laboratory tests does in fact translate into reduced low temperature and fatigue cracking.



**Figure 37. Brittle State Fracture Energies for Investigated Binders**  
 (open bars give energy at -32°C while shaded bars give energy at -38°C)



**Figure 38. Brittle State Stiffness for Investigated Binders**  
 (open bars give stiffness at -32°C while shaded bars give stiffness at -38°C)



## COMPACT TENSION TESTING OF ASPHALT BINDERS

A small portion of this project was dedicated to the development of the compact tension (CT) geometry for the testing of asphalt binders in their brittle and brittle-to-ductile states. This geometry is preferred over the three-point bend specimen since it allows for the simultaneous measurement of the crack opening displacement and the fracture toughness or fracture energy. It is our belief that eventually the critical crack opening displacement is the more relevant property since it provides a measure of strain tolerance in the presence of sharp flaws. For a detailed review of the background to this method and results obtained, the reader is referred to Edwards and Hesp (2006), a brief synopsis of which follows here.

The CT tests were conducted on specimens with different widths,  $B$ , and notch depths,  $a$ . Table 2 shows that the fracture toughness,  $K_{Ic}$ , was found to be reasonably constant, irrespective of the notch depth or specimen width for both straight and modified binder. Hence, this verified that the plane-strain condition was met in the specimen geometries and sizes tested. The only exception was when AAN binder was tested at  $-16^{\circ}\text{C}$ , which is close to the ductile state for this material. Here the fracture toughness was found to be slightly higher for narrow samples ( $B = 12.7$  mm) with deep notched ( $a/W = 0.6$ ) likely due to a mixed mode fracture event. However, data for the same two binders in Table 3 shows that the fracture energy,  $G_f$ , was found to consistently decrease with notch depth, which is thought to be due to energy absorbing mechanisms away from the crack tip.

**Table 2. Effect of Notch Depth and Specimen Width on Fracture Toughness ( $K_{Ic}$  in  $\text{kN}\cdot\text{m}^{-3/2}$ ) of AAN and AAG-2 + 5% SBS**

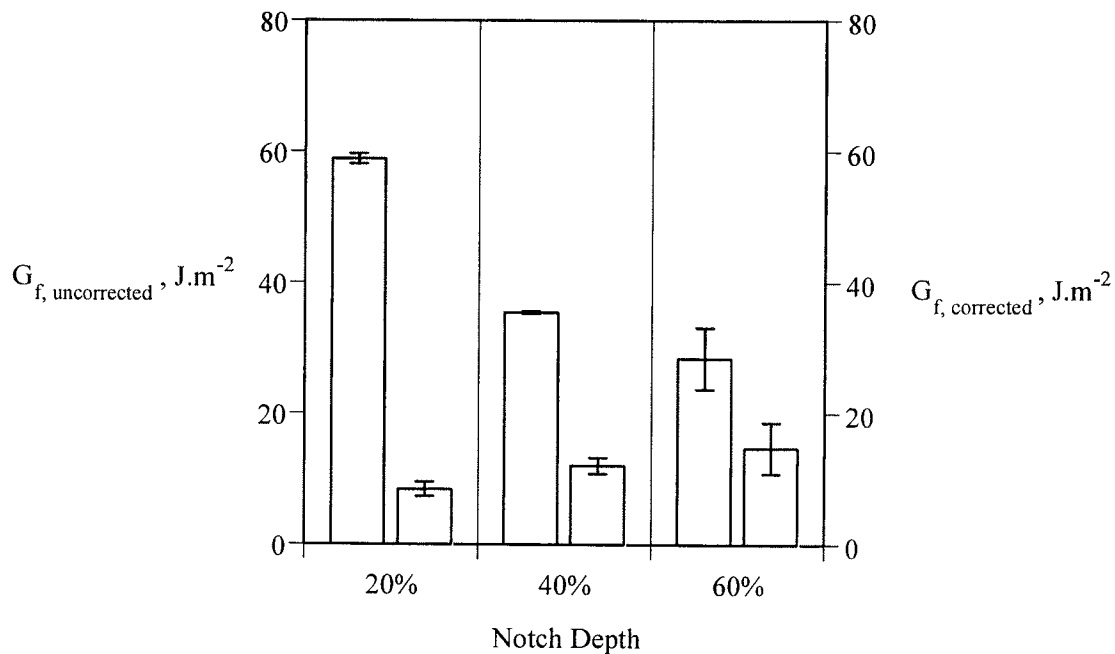
Binder	T, $^{\circ}\text{C}$	a/W	Specimen Width (B), mm		
			12.7	19.1	25.4
AAN	-16 †	0.2	48	40	47
		0.4	31	57	51
		0.6	86	54	- *
AAN	-22	0.2	42	39	41
		0.4	53	47	50
		0.6	52	46	42
AAG-2 + 5% SBS	-22	0.2	120	135	120
		0.4	145	155	136
		0.6	144	154	148
AAG-2 + 5% SBS	-28	0.2	172	144	121
		0.4	168	178	158
		0.6	159	160	157

Note:  $a$  = notch depth,  $W$  = sample height, and  $B$  = sample width, † AAN is close to the ductile state at  $-16^{\circ}\text{C}$  so this set of data was not the most reproducible and some of these results may reflect a mixed-mode fracture toughness. \* Not all samples were tested in all nine configurations. (After Edwards and Hesp (2006).)

**Table 3. Effect of Notch Depth and Specimen Width on Fracture Energy ( $G_f$  in  $J.m^{-2}$ ) of AAN and AAG-2 + 5% SBS**

Binder	T, °C	a/W	Specimen Width (B), mm		
			12.7	19.1	25.4
AAN	-16	0.2	13.5	9.9	15.1
		0.4	5.4	9.5	8.2
		0.6	7.7	5.4	- †
AAN	-22	0.2	8.2	10.9	8.9
		0.4	5.8	4.5	5.7
		0.6	3.7	4.4	3.3
AAG-2 + 5% SBS	-22	0.2	54.0	51.3	58.8
		0.4	37.7	43.5	35.3
		0.6	25.7	30.5	30.1
AAG-2 + 5% SBS	-28	0.2	89.4	73.8	78.3
		0.4	39.3	52.1	44.7
		0.6	27.5	32.0	30.9

Note: † Not all data was obtained for all 36 combinations. (After Edwards and Hesp (2006).)

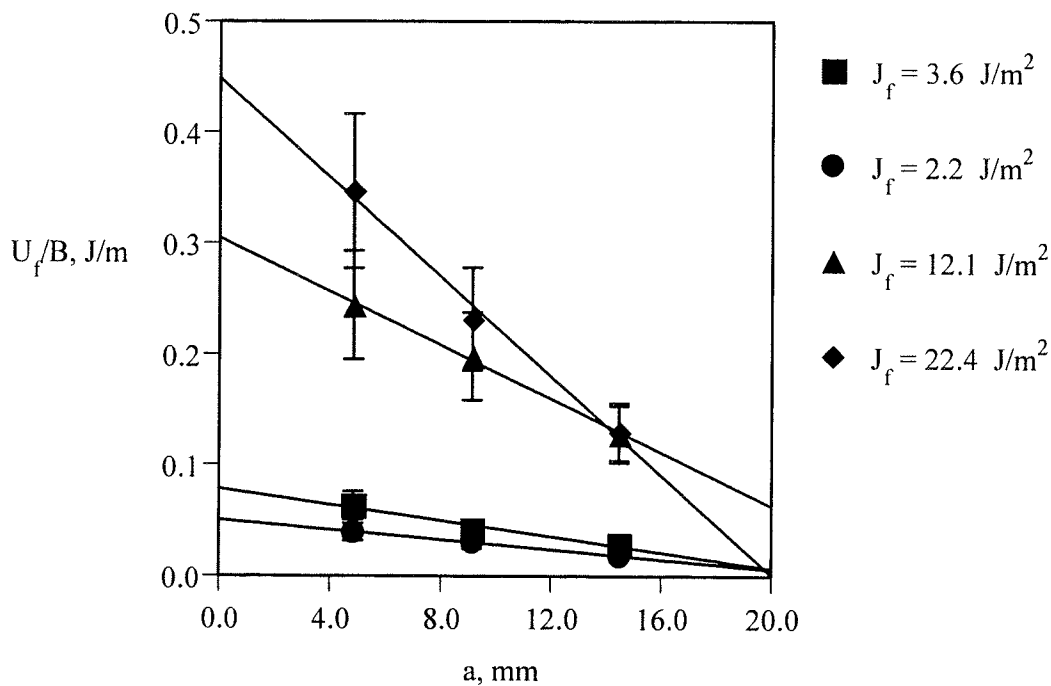


**Figure 39. Effect of Energy Correction on Fracture Energy of AAG-2 + 5% SBS at  $-22^{\circ}C$**   
 Note: Relative errors were calculated from differences in replicate measurements. (After Edwards and Hesp (2006).)

Deeper notches or an energy correction are able to account for this issue. Figure 39 shows the

fracture energies corrected by subtracting the energy absorbed in an unnotched sample up to the same load within the same time for which the notched samples failed. The reader is referred to Edwards and Hesp (2006) for a detailed discussion. However, the figure shows that the obtained fracture energy is the same within the experimental error for all three  $a/W$  ratios (notch depths).

A slightly different fracture energy,  $J_f$ , was obtained in a more direct fashion from the slope of a plot of the normalized failure energy,  $U_f/B$ , versus notch depth,  $a$ . The fracture energy obtained according to this generalized locus method,  $J_f$ , provides results closer to those obtained with an energy correction from a single notch depth.



**Figure 40. Generalized Locus Approach to Determine Fracture Energy,  $J_f$**   
 (■ = AAN at  $-16^{\circ}\text{C}$ ; ● = AAN at  $-22^{\circ}\text{C}$ ; ▲ = AAG-2 + 5% SBS at  $-22^{\circ}\text{C}$ ; ◆ = AAG-2 + 5% SBS at  $-28^{\circ}\text{C}$ ;  
 error bars give  $\pm 20$  percent of the mean.)

Figure 40 shows that for the AAG-2 + 5% SBS sample at  $-22^{\circ}\text{C}$  the fracture energy is calculated to be  $12.1 \text{ J}\cdot\text{m}^{-2}$ , which is statistically the same as the corrected fracture energy from Figure 39 for this binder at the same temperature. Reproducibility of the test was found to be good with pooled standard deviations of 5-10 percent for  $K_{Ic}$  and 15-20 percent for  $G_f$ , which is typical. Given the fact that brittle fracture properties can vary by orders of magnitude for binders of the same Superpave® grade, it is concluded that the test method has a high ability to reveal statistically significant differences in toughness.

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# Conclusions

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Given the review of the literature and the results presented in this report, the following conclusions are provided:

- The fracture toughness and fracture energy in the brittle state do not vary by much for binders that are unmodified or modified with only low levels of additives when the materials are tested at approximately equal stiffness. Exceptions to this are binders with very low asphaltene contents that tend to be more brittle (e.g., California Valley). Hence, binder grading for unmodified binders should be reasonably accurate based on BBR properties alone provided the effects of reversible aging (physical hardening) are taken into consideration.
- Brittle state fracture energies measured at different rates and temperatures can be shifted to form master curves. For some binders this shifting produces smooth master curves. However, most binders fail to produce entirely smooth master curves likely due to subtle phase changes in the temperature range of testing.
- The essential and plastic works of fracture for a given binder are largely determined by the viscous flow and yield characteristics of the material under ductile conditions. For very soft binders, the ductile fracture properties can be low which is largely due to a generally low yield stress. However, the crack opening displacement property for such binders, given by  $\delta_t = w_e/\sigma_y$ , could still be acceptable in part due to this low yield stress.
- Ductile works of fracture measured at different rates and temperatures can be shifted to form master curves. For the binders investigated in this study the shifting produces smooth curves suggesting no phase changes occur over the range of rates and temperatures employed.
- Two test sections on Highway 655 that were constructed with polymer-modified binders that show particularly high essential and plastic works of fracture survived the particularly harsh winter of 2004 unscathed while other sections with inferior properties show a considerable amount of wheelpath distress. For the C-SHRP trial in Hearst, Ontario, essential and plastic works of fracture were able to explain the observed differences in transverse stress cracking for the three different binders. The binder with the lowest ductile fracture properties showed the highest amount of distress while the binder with the highest ductile fracture properties showed the least amount of distress. These observations provide some validation of the essential work of fracture approach to asphalt binder grading.
- Modified binders for which the polymer-rich phase forms a continuous network can show significantly increased fracture properties in the brittle state. This research shows that it is possible to produce an exceptionally tough binder from what is generally considered to be a very difficult to modify base asphalts (Cold Lake).
- The compact tension geometry can provide accurate fracture measurements. While fracture toughness,  $K_{Ic}$ , is found to be independent of specimen size and geometry, the fracture energy,  $G_f$ , is found to depend on notch depth, which is thought to arise from energy absorbing mechanisms away from the fracture zone. An energy correction or an analysis according to the so-called generalized locus method is able to account for this issue.

How the above findings relate to field performance remains to be investigated with well-designed pavement trials that are to be followed until sufficient distress is observed to draw meaningful further conclusions. The main hypothesis that tougher binders are better at resisting fracture distress has been validated with pavement trials in Lamont, Hearst and Cochrane. However, further validation is desirable since these existing trials did not contain any test sections with superior toughness binders.

## Recommendations

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Given the review of the literature and the results presented in this report, the following recommendations are provided:

- It would be very helpful for the further development of the two fracture tests (DENT and CT) if other laboratories start implementing the methods developed. Once the data in this report are reproduced in the laboratories of both users and producers of asphalt cement, it may become feasible to design a limited number of pavement trials that are to be constructed with binders of varying degrees of toughness. However, such trials will have to be carefully designed since it will be difficult to exclude all possible confounding factors. The observed distress in such trial can then be used to further validate the hypothesis that tougher binders reduce the amount of fracture in service and that the fracture mechanics approach to binder grading provides improved performance prediction over the currently used rheology approach (AASHTO M320).
- It is proposed that the critical crack opening displacement, CTOD or  $\delta_t$ , be further investigated for performance grading. A large number of binders need to be tested at varying temperatures and rates of loading to assess this property. It can then be compared with measured fatigue and low temperature fracture performance. However, once again, such studies would need to pay particular attention to the large number of likely confounding factors. The CTOD or  $\delta_t$  property is easily obtained from the crack mouth opening displacement in the CT test and from the ratio of essential work over yield stress in the DENT test,  $w_e/\sigma_y$ . A preliminary study on materials from the latest Federal Highway Administration Accelerated Loading Facility trial has shown that the CTOD is better at predicting observed fatigue severity than binder loss modulus (Andriescu et al. (2006)).

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**Development of an Improved Asphalt  
Binder Specification Testing Approach  
*Part III. Asphalt Mixture Testing  
and Field Validation***

**August 1, 2006**

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<b>Publication Title</b>	<b>Development of an Improved Asphalt Binder Specification Testing Approach</b> <b><i>Part III. Asphalt Mixture Testing and Field Validation</i></b>
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<b>Publication Date</b>	March 28, 2006
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<b>Abstract</b>	<p>Part III of this final report is to document and discuss an investigation of the failure performance properties of asphalt mixtures that were used in the seven pavement trial sections on Highway 655 just north of Timmins, Ontario, and to validate the general approach to asphalt binder and mixture grading with early field performance data.</p> <p>Asphalt mixtures were tested using the following methods: (1) dynamic compression to determine the complex modulus and phase angle master curves; (2) dynamic fatigue in four-point bending; (3) dynamic creep fracture in compact tension; (4) monotonic rate tests on double-edge-notched tension specimens to determine the essential and plastic works of fracture as well as an approximate critical crack opening displacement; and (5) dynamic compression to determine failure numbers without confinement.</p> <p>An analysis of the findings suggests that only the monotonic double-edge-notched tension test results correlated reasonably well with field distress. It was found that the ductile failure properties needed to be supplemented with reversible aging tendencies to provide a complete interpretation of the distress variation experienced within the trial.</p>
<b>Key Words</b>	Asphalt Performance Grading, Low Temperature Cracking, Fatigue Cracking, Fracture Energy, Essential Work of Fracture, Plastic Work of Fracture, Critical Crack Opening Displacement; Field Validation

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## Executive Summary

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This report documents and discusses an investigation of the ductile state properties of selected asphalt mixtures that were used in pavement trial sections on Highway 655 just north of Timmins, Ontario. Ductile properties were determined with the following methods: (1) dynamic compression to determine the complex modulus and phase angle master curves; (2) dynamic fatigue in four-point bending; (3) dynamic creep fracture in compact tension; (4) monotonic rate tests on double-edge-notched tension specimens to determine the essential and plastic works of failure as well as an approximate critical crack opening displacement; and (5) dynamic compression to determine failure numbers without confinement.

The complex modulus and phase angle master curves varied by relatively small amounts. Only the mixture from section 2 showed a significantly higher complex modulus and lower phase angle, which could in part explain the high level of distress for this section in the field. Section 7 had a slightly lower complex modulus and higher phase angle, and this could explain to some extent the absence of distress in this section. However, the remaining five sections showed very similar master curves while two were cracked severely and three others survived the first three years largely without any significant distress.

Both the dynamic fatigue tests in four-point bending and the dynamic creep fracture tests in compact tension showed little correlation with the field performance data. Softer binders typically performed more poorly in the constant load tests while harder binders did better. However, no relationship between field and lab data could be found for any of the several ways in which the data were analysed.

The essential work of fracture method uses a monotonic tensile double-edge-notched (DENT) geometry. By performing the DENT test on a range of specimens with varying notch depths (i.e., ligament lengths) it is possible to partition the essential work,  $w_e$ , needed to form two new surfaces, from the non-essential plastic work,  $w_p$ , dissipated in areas away from the fracture process zone. This separation of energies is important since in the pavement the failure process is often localized around flaws and notches or in areas of high stress concentration under the edges of tires. Hence, a regular direct tension test on a mixture may overestimate the performance of some materials and is therefore expected to provide a less accurate prediction of in-service performance. It is hypothesised that both the essential and plastic works of fracture need to be high to assure good performance. In addition, for mixtures with equal or similar fracture energies, the ones with low yield stresses are preferred over those with higher yield stresses. The critical crack tip opening displacement,  $\delta_t$  or CTOD, can be obtained from the ratio of essential work over the net section yield stress,  $w_e/\sigma_y$ , and provides a measure of strain tolerance in the ductile and brittle-to-ductile states in the presence of sharp flaws (cracks). It is believed that this property will be able to provide an improved grading for asphalt in terms of fatigue resistance.

The ductile fracture properties for the set of Highway 655 mixtures varied significantly.



Essential works of fracture were found to vary by a factor of nearly 2.4, plastic works varied by a factor of nearly 3.5, and crack tip opening displacements varied by a factor of 2. The binders used to make these mixtures had Superpave® grades that ranged from a low of PG 52-34 to a high of PG 64-34. The four mixtures from Highway 655 that showed the highest crack opening displacements survived the particularly severe winter of 2004 largely unscathed while the three with inferior properties incurred a considerable amount of wheel path distress. A complete explanation of the ranking in this trial was obtained by considering the variation in reversible ageing between the different binders (see figure 11 in part I of this report), which likely related to the varying degrees of crack initiation during cold spells.

It was found that there is a reasonable correlation between mixture and binder essential works of fracture but less so for the plastic works of fracture and the crack opening displacements. This suggests that a binder test may be able to predict mixture performance thus greatly simplifying the design process. However, the reason(s) for the differences in binder and mixture CTOD ranking remain to be investigated and dealt with.

The dynamic creep life in compression was also determined and, as expected, it was found that the correlation with in-service performance was poor. The mixture with the highest laboratory creep life was made with an oxidized binder. In the field this was also the material that showed the highest amount of wheel path cracking after the severe winter of 2004. The mixture with the lowest laboratory creep life was found to have survived unscathed. However, as for the DENT tests, the dynamic creep tests showed some degree of variability in test results.

# Introduction and Background

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Failure in asphalt pavements at low temperatures is often a complex process and can involve a number of different factors that lead to an increase in cracking severity with time. Low-temperature cracks are nearly always found transverse to the driving direction because this is how the bulk of the thermal strain energy is stored during cold periods in winter. The longitudinal direction is restrained whereas the transverse direction can often shrink enough to prevent longitudinal thermal cracks. However, this observation does not say anything about how and when each individual transverse crack started. Evidence from thin yet conventionally designed pavement trials in northern Ontario and elsewhere suggests that the onset and severity of low-temperature transverse cracking distress can be intricately linked to not only the binder's low-temperature stiffness and relaxation ability but also its fatigue characteristics, asphalt mixture design, traffic levels, structure of the pavement, and sub-grade characteristics.

This report assesses the ambient temperature failure characteristics of seven different asphalt mixtures that were used in Ontario's most recent pavement trial on Highway 655 north of Timmins which was constructed in the summer of 2003 (Iliuta et al., 2004). Dynamic compression tests were used to determine complex modulus and phase angle master curves, while four-point bending and compact tension tests were conducted to determine fatigue lives and crack growth rates. The essential work of fracture approach was used to measure the resistance of the respective mixtures to failure in uniaxial tension. The results obtained in the mixture are compared with those in the asphalt binders. In addition to the Highway 655 results a number of other binder and mixture data are used for comparison. The dynamic compressive creep test was used to determine the resistance of the respective mixtures to compressive failure. Since it was hypothesized that different mechanisms operate in tension and compression it was expected that the results would differ for the different failure tests. The findings of this research will be of use to those individuals currently developing a simple performance test which is essentially a restrained version of the test used in this project.

The laboratory observations are compared with the early distress as found in the field sections. The test road was subjected to one particularly severe cold spell in January 2004 during which the surface temperature of the road dropped below  $-30^{\circ}\text{C}$  on eight separate days with lows of  $-34^{\circ}\text{C}$  on two occasions (Iliuta et al., 2004). Days after this cold spell there were only about four transverse cracks visible. However, a visit in April 2004, several weeks into the spring-thaw, revealed that three test sections had been badly damaged in the south bound lane on their left wheel paths. The total distress amounted to approximately 150 m of longitudinal cracks. Two other sections had survived the winter unscathed while another two had only minor damage. The data obtained in the respective mixture tests is compared with the early cracking in these trial sections.

Using the laboratory to field correlation a discussion on an improved specification for asphalt binders is provided.

# Experimental

## MATERIALS

The materials investigated in this project were obtained directly from the contractor prior to and during construction. The seven binders were obtained from the low-temperature pavement trial north of Timmins, Ontario. The binders were sampled during the paving of each test section from the line used to transfer the material from their storage tank into the asphalt plant. Aggregates were sampled according to standard procedures and transferred in 40 kg bags to Queen's University for fabrication of gyratory plugs and slabs.

A listing of the binders and their pertinent information is given in table 1.

**Table 1 Pertinent Properties of Binders Investigated for Fracture Performance**

Binder	Crude Source	Modification	Grade
655-1	Lloydminster	RET	PG 64-37
655-2	Unknown	Oxidized/SBS	PG 64-35
655-3	Unknown	SBS	PG 64-36
655-4	Unknown	SBS	PG 64-35
655-5	Unknown	SBS	PG 64-35
655-6	Unknown	Oxidized	PG 58-35
655-7	Unknown	Unmodified	PG 52-35

Note: RET = reactive ethylene terpolymer of ethylene, butyl acrylate, and glycidyl methacrylate; and SBS = styrene-butadiene-styrene linear block copolymer.

The surface course mixture was used for all tests in this project. It consisted of a Superpave® 12.5 mm design with an optimum asphalt binder content of 5.2 weight percent. The mixture contained coarse aggregate and screenings of a hard volcanic rock and a sand fraction from a local river source.

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## EXPERIMENTAL METHODS

### Asphalt Mixture Preparation

Asphalt mix cylinders and slabs using the seven Highway 655 binders were made with a hard volcanic rock-based Superpave® design with a maximum aggregate size of 12.5 mm. All materials were heated in a forced-convection oven at temperatures between 150°C and 160°C for at least four hours prior to use.

Asphalt mix cylinders were compacted to a constant density by using a Rainhart gyratory compactor. For the dynamic creep tests the gyratory plugs were cored to produce 82 mm × 185 mm cylindrical specimens which were subsequently polished on both ends to produce ~150 mm high specimens to a flatness of ± 0.2 mm. Gyratory plugs were also cut with a diamond-tipped saw to obtain prismatic samples of approximately 85 mm × 36 mm × 180 mm in size for double-edge-notched tension testing. Notches were cut on both sides in the middle of the samples in order to obtain ligaments in the 30 mm to 120 mm range.

For the four-point bending and compact tension tests the samples were cut from slabs made with a laboratory compactor. Slabs were 60 cm in length by 40 cm in width and either 10 cm or 13 cm in height. The ends of the specimens were no closer than 25 mm from the compacted slab face, to assure uniform air void distribution.

Void contents were determined after cutting by weighing dry, wet/surface dry, and under water. Most samples tested had voids within a 2 percent range around the average. All mixture numbers in this paper correspond to section numbers in the trial (i.e., mixture 1 was made with identical materials and composition as those for section 1).

### Simple Performance Testing for Dynamic Modulus and Phase Angle

The dynamic modulus and phase angle master curves were determined for all mixtures except section 3 at the Pavement Materials and Construction Laboratory, Turner-Fairbank Highway Research Center, Federal Highway Administration, McLean, Virginia. The following testing procedure was kindly provided by Nelson Gibson of FHWA.

A simple performance tester (SPT) manufactured by IPC Global was utilized for determining the complex modulus and phase angle master curves. Cylindrical SPT specimens were cored from laboratory compacted slabs. The cylindrical specimens were 100 mm in diameter and sawed at both ends to 150 mm in height with smooth and parallel cut faces as described in the AASHTO Provisional Standard for Dynamic Modulus Testing, TP62-03, and protocol from the Simple Performance Tester for Superpave® Mix Design (Bonaquist et al., 2003). The ends of the specimen were no closer than 25 mm from the compacted slab face, which exceeded the protocol of trimming a minimum of 7.5 mm from each end of a gyratory specimen to ideally achieve uniform air void distribution. Three axial LVDTs were mounted for dynamic modulus testing at 120° apart on studs having a gauge length of 75 mm attached to the sides of the SPT specimen with epoxy.

Prior to SPT testing, the SPT specimens were placed in an environmental chamber to stabilize their temperature with the testing temperature. After the SPT specimen reached equilibrium temperature, two Teflon pads were used on top and beneath the SPT specimen to reduce friction for dynamic modulus testing.

The SPT dynamic modulus tests deviated slightly from established procedures (AASHTO TP62-03, 2005; Witzak et al., 2002) at a sweep of temperatures of 4, 12, 24, and 36°C and at frequencies of 10, 1, 0.1, and 0.02 Hz. Nevertheless, sufficient dynamic modulus data were acquired to obtain overlap at different temperatures for master curves of interest. Two specimens with three LVDT's were tested and yielded similar estimated limits of accuracy when using 2 LVDT's and 4 replicates: 13.1% and 13.4%, respectively. Before applying the dynamic load to the specimen, a contact load was applied that was equal to 5 percent of the dynamic load. After that, a compressive haversine dynamic load was applied to the specimen and dynamically controlled by the SPT so that the resulted axial strain was in the range of 75 to 125  $\mu\text{m/m}$  (this varies from mixture to mixture depending on the stiffness and temperature). Following pre-programmed protocols, trial dynamic loads were applied for 10 pre-loading cycles to determine the stress required to maintain 75-125  $\mu\text{m/m}$  dynamic strains, followed by 10 formal cycles to measure the dynamic modulus and phase angle. Details of data reduction and sinusoidal fitting for phase angle and dynamic modulus calculation can be found in Bonaquist et al. (2003).

### **Dynamic Fatigue in Four-Point Bending and Compact Tension**

Dynamic fatigue in bending was done on an MTS 810 servo-hydraulic test frame. The instrument was equipped with a four-point bending fixture accommodating beams of asphalt concrete 76 mm by 76 mm by 381 mm. Tests were conducted in constant force and constant displacement modes. Repeated haversine pulses of 0.1 s duration were applied without rest periods until the samples failed. All tests were done at room temperature,  $25 \pm 1^\circ\text{C}$ . The data were logged with the TestStar IIs software.

The controlled force fatigue data were analysed according to the following failure criteria: (1) a conventional 90 percent reduction in stiffness; (2) a change in the dissipated energy ratio (Pronk, 1995); (3) a peak in the plot of (stiffness  $\times$  number of cycles) versus number of cycles (Rowe and Bouldin, 2000); and (4) a change in the slope of a two-stage Weibull plot (Tsai et al., 2002). The controlled displacement fatigue data were analysed according to: (1) a conventional 50 percent reduction in stiffness; (2) a change in the dissipated energy ratio (Pronk, 1995); (3) a peak in the plot of (stiffness  $\times$  number of cycles) versus number of cycles (Rowe and Bouldin, 2000); and (4) a change in the slope of a two-stage Weibull plot (Tsai et al., 2002).

The dynamic creep/fatigue tests on compact tension (CT) specimens were done on an MTS 810 system. Haversine load pulses of 150 N and 0.1 s duration were applied continuously on rectangular specimens of an effective width of 154 mm (W), effective thickness of 40 mm (B), and effective height of 150 mm (H). The initial notch depth was approximately 20 mm (a). Loading was done through aluminium end blocks. Hence, the initial  $a/W$  ratio was approximately 0.13. A shallow groove was cut on either side of the specimen to a depth of 5 mm to prevent wandering of the crack.

The groove facing the front of the machine was covered with a thin layer of plaster of Paris to facilitate visual observation of crack length. The number of cycles to reach a certain crack length was recorded at 5 mm intervals. The crack growth rate was determined by fitting the raw data to an exponential and then plotting the differential (i.e., smoothed crack growth rate) in a double logarithmic graph according to the Paris law:

$$\frac{da}{dN} = A(\Delta K)^n \quad [1]$$

where  $a$  is the crack length (m),  $N$  is the number of cycles,  $\Delta K$  is the stress intensity factor ( $\text{Pa m}^{0.5}$ ), and  $A$  and  $n$  are Paris law constants. The stress intensity factor was determined according to standard equations available elsewhere (McCrum et al., 1995).

### Ductile State Essential Work of Fracture Testing

The essential work of fracture,  $w_e$ , and plastic work of fracture,  $w_p$ , were determined in double-edge-notched tension (DENT).

In brief, samples were cut from gyratory cores and subsequently glued with aluminum end pieces for ready attachment to the test frame. The notch depth in the double-edge-notched tension sample was varied to provide ligament lengths of 30-120 mm. Figure 1 provides a photograph of two samples, one just after the peak load was reached and the other after the test was stopped. The arrows in the left photograph show large aggregate particles that started breaking just prior to the peak load was reached. The arrows in the right photograph show the elliptical shape of the fracture zone.

Samples were tested in duplicate and the total works of fracture,  $W_t$ , were used to determine the specific total works of fracture,  $w_t$ , by dividing through the cross sectional area for each ligament,  $LB$ . The specific total work of fracture was plotted against the ligament length to provide a measure of the essential and plastic works of fracture. The essential and plastic works of fracture were determined at 25°C, and a loading rate of 5.4 mm/min (strain rate of  $5 \times 10^{-4}$  m/m/s). For a detailed discussion on the development of this test method, and the rheological characterization of these binders, the reader is referred to the doctoral dissertation of Andriescu (2006).

The force-displacement diagrams were all found to be more or less self-similar, the force-displacement diagrams showed a level portion before the samples failed through macro cracking (i.e., full ligament yielding occurred before fracture), and the volume of the plastic zone was found to be proportional to the square of the ligament length multiplied by the sample thickness (i.e., Equation 2 was found to fit the experimental data (Andriescu et al., 2004):

$$w_t = w_e + \beta w_p L \quad [2]$$

where

$w_t$  = specific total work of fracture ( $\text{J/m}^2$ );

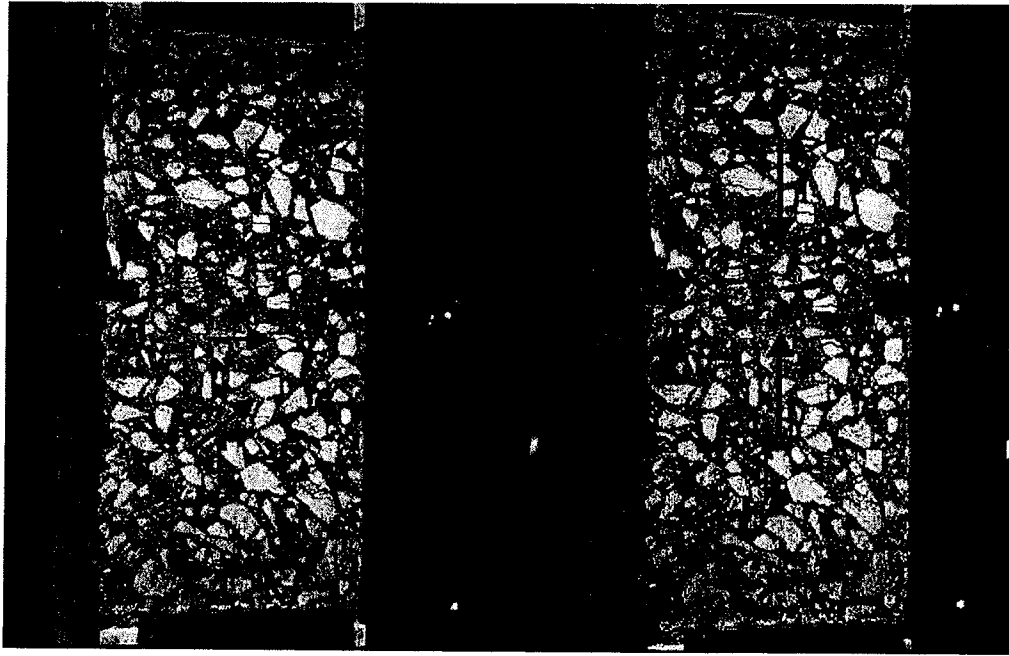
$w_e$  = specific essential work of fracture ( $\text{J/m}^2$ );

$w_p$  = specific plastic work of fracture ( $J/m^3$ );

$B$  = sample thickness (m);

$L$  = ligament length (m); and

$\beta$  = scaling factor describing the shape of the plastic zone (Mai et al., 2000).



**Figure 1. Double-Edge-Notched Tension Specimens after Failure (Andriescu et al., 2004).**

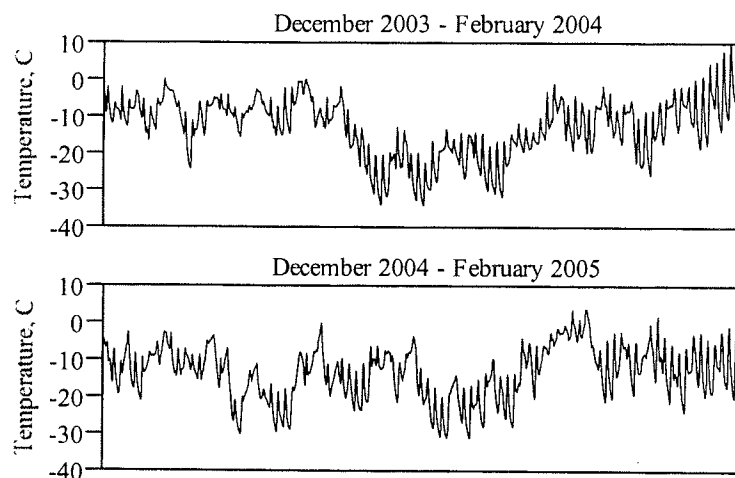
### **Dynamic Creep Testing in Unconfined Compression**

The dynamic creep response was obtained for all seven mixtures in unconfined compression tests at both 25°C and 40°C. The loads applied produced stress levels of 400 kPa at 40°C and 2000 kPa at 25°C. All samples were subjected to 0.1 s haversine loading pulses followed by 0.9 s rest periods until complete failure occurred. In order to reduce friction between the loading platens two thin Teflon® sheets with grease in between were inserted at either side of the sample.

# Results and Discussions

## WEATHER DATA AND FIELD PERFORMANCE SURVEYS

Temperature loggers were retrieved from the site in April 2004, April 2005, and June 2006. Figure 2 provides pavement surface temperatures for the first two winters. Useable pavement surface temperatures were obtained from one logger for both 2004 and 2005 while useable air temperature data were obtained from two loggers for all three winters.



**Figure 2. Pavement Surface Temperatures**

Note: Pavement temperatures are averaged over four thermocouples.  
The curves provide temperatures at ~5 mm depth.

The temperatures reached extremes in the first winter with a record low air temperature of  $-47^{\circ}\text{C}$  on January 9, 2004. The pavement surface temperature (at 5 mm depth) reached  $-34^{\circ}\text{C}$  on two occasions during January 2004 and reached below  $-30^{\circ}\text{C}$  on eight separate occasions. During early 2005 the air temperatures reached around  $-40^{\circ}\text{C}$  on six occasions, while the pavement at 5 mm depth reached below  $-30^{\circ}\text{C}$  on five occasions. In early 2006 the two lowest air temperatures recorded were around  $-39^{\circ}\text{C}$  with what would have been corresponding surface temperatures of around  $-30^{\circ}\text{C}$  or slightly lower.

Hence, this trial is situated in an ideal location for low temperature specification test validation, since temperatures regularly reach close to the design value. The distress observed after the first winter provided us with insight into which factors are of importance for the mitigation of transverse and wheel path cracking.

The cracking distress was surveyed shortly after the severe cold spell of early January 2004 and during the spring thaw in late April 2004. A more cursory survey was conducted in April 2005,

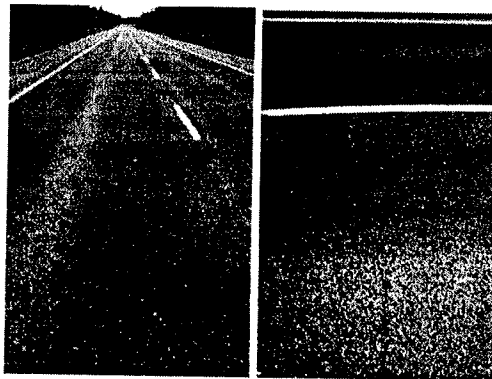


followed by a more detailed one in July 2006. Figure 3 shows typical photographs of the wheel path distress found in sections 2, 3, and 4.

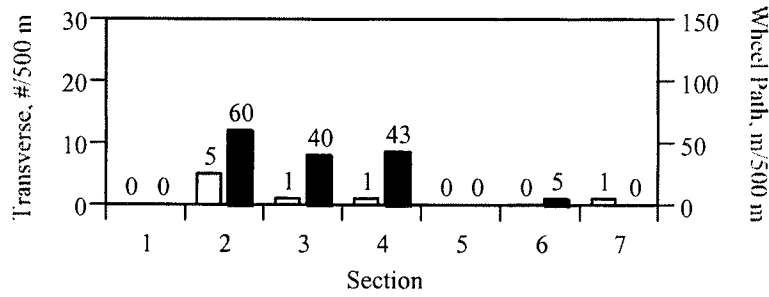
Figure 4 shows the cracking severity for all sections in terms of wheel path and transverse cracking for early 2004, while figure 5 shows this for the summer of 2006. It is obvious from the data that for binders of nearly identical grades, there is a considerable variation in terms of distress. Two sections survived the first winter unscathed, while three others were badly damaged. Sections 2, 3, and 4 sustained 60.2 m, 40.1 m, and 43.5 m of wheel path cracking, respectively, making them badly damaged at a very early stage. Section 6 sustained a single wheel path crack of 5.3 m, while section 7 sustained a single transverse crack about half the width of the lane. Sections 1 and 5 survived the first winter unscathed.

From the data in figures 4 and 5 it is evident that the binders in sections 2-4 performed poorly compared to those in sections 1 and 5-7. Section 4 exhibits 50 m of mid-lane cracking, likely due to paver-induced segregation, in addition to the 92 m of wheel path cracks, making it the worst performer. A detailed analysis of end result specification data and of a FWD investigation excluded construction and sub-grade variability as possible causes for the observed distress variation within the trial (Bodley et al., Submitted, AAPT 2007).

Most of the longitudinal cracks were located in the southbound lane in the left wheel path. This is believed to have been because this lane carried loaded logging trucks to Timmins, while they returned empty on the northbound lane. Furthermore, the left wheel path is thought to be under more thermal restraint than the right wheel path, which is closer to the edge of the pavement. Hence, the predominantly triaxial state of stress is thought to have caused the severe distress in this location. How different material properties have contributed to the differences between the seven sections is discussed next.

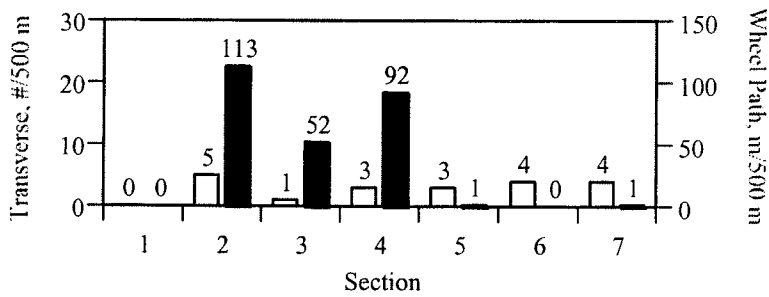


**Figure 3. Spring 2006 Wheel Path Cracking in Section 3 (left) and Transverse Crack Initiation in Section 4 (right)**



**Figure 4. Spring 2004 Crack Survey**

Note: Open bars for transverse cracking and solid bars for wheel path cracking.

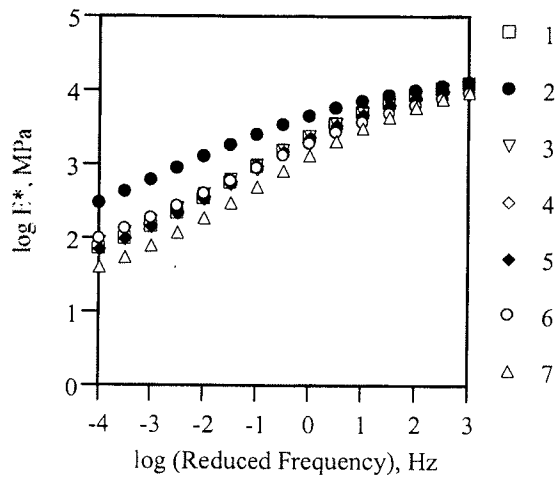


**Figure 5. Summer 2006 Crack Survey**

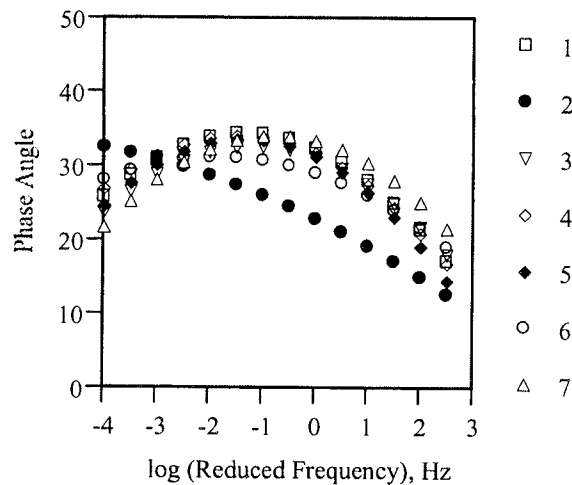
Note: Open bars for transverse cracking and solid bars for wheel path cracking.

## COMPLEX MODULUS AND PHASE ANGLE MASTER CURVES

The complex modulus (figure 6) and phase angle (figure 7) master curves in dynamic compression were determined according to standard procedures as described in the experimental section.



**Figure 6. Dynamic Modulus Master Curves in Compression**



**Figure 7. Phase Angle Master Curves in Compression**

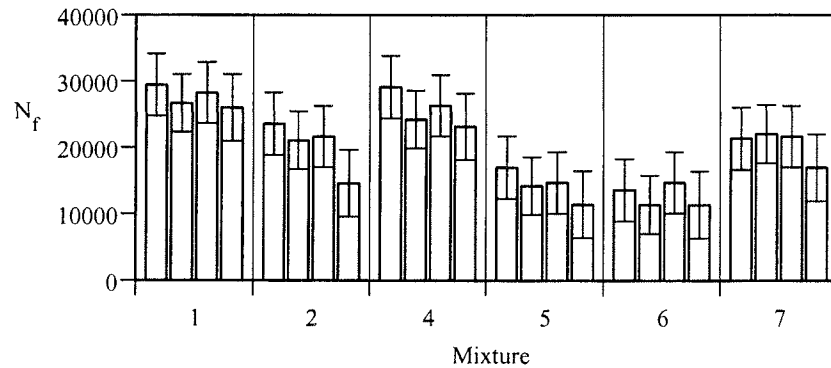
The results were largely unremarkable except for the tests on mixture 2 which showed both a higher complex modulus and lower phase angle at all frequencies investigated. It is unclear at this moment what if any significance this has with respect to field performance since the other systems were all very similar yet showed very different performance in service. Mixtures 1 and 3-6 have nearly identical master curves while two are cracked severely and three are almost without any cracks.

It is likely that the dynamic modulus and phase angle have only limited use on their own for the prediction of asphalt performance in terms of the type of wheel path distress observed in this trial. The low strain properties as measured in the dynamic modulus protocol do not relate well with the high strain fracture behaviour of the mixtures when they were subjected to critical stresses during the 2004 cold spell and subsequent spring thaw.

## DYNAMIC FATIGUE IN FOUR-POINT BENDING

Both the displacement- and force-controlled tests were conducted at room temperature,  $25 \pm 1^\circ\text{C}$ , a loading frequency of 10 Hz, and by using haversine pulses. The displacement-controlled tests were done at 600 micro strain, which rendered failure for all mixtures within a reasonable time. Since these tests typically take a long time, only one strain level was investigated thus limiting the information that can be obtained from the data.

The following failure criteria were used: (1) a 50 percent stiffness reduction; (2) a change in dissipated energy ratio (Pronk, 1995); (3) a peak in the plot of (stiffness  $\times$  number of cycles) versus number of cycles (Rowe and Bouldin, 2000); and (4) a change in slope of the two-stage Weibull plot (Tsai et al., 2002). The average number of cycles to failure for each mixture is given in figure 8, in the above order for each criterion.

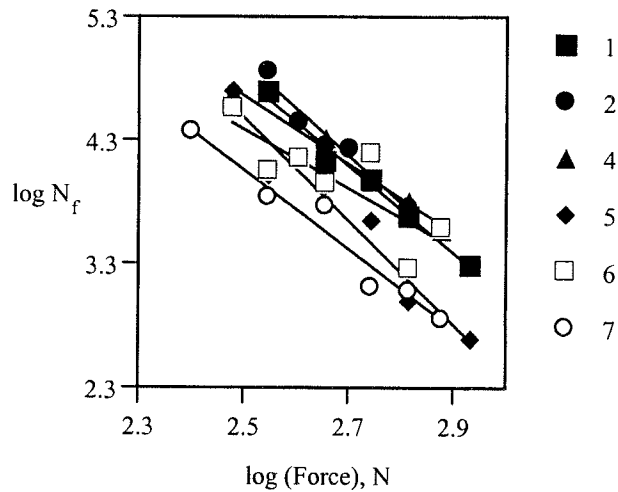


**Figure 8. Number of Cycles to Failure in Displacement-Controlled Tests at 600  $\mu\text{m}/\text{m}$**

Note: Mixture 3 was not tested due to materials and time limitations. Averages of between two and three samples are provided for each mixture. First bar provides failure according to a 50 percent stiffness reduction criterion; second bar according to the dissipated energy ratio; third bar according to the peak in stiffness  $\times$  number of cycles plot; and fourth bar for change of slope in two-stage Weibull plot. Error bars provide  $\pm$  one pooled standard deviation.

The data shows that a change in the failure criterion does not make a significant difference in the ranking for any of the mixtures. Only two mixtures, with very similar failure numbers, rank differently in the 50 percent stiffness reduction (first bar) compared to all the other rankings, which are consistent with each other. Further, there appears to be little if any relationship between the endurance limits in this test and the field performance. Mixtures 5 and 6 rank last while in the field they have yet to show significant cracking. Mixture 4 ranks second best in figure 8 while the field performance is worst. Hence, it can be concluded that the controlled-displacement test is unable to predict the relative field performance for these mixtures. The reason for this failure is not entirely clear but may be related to differences between test and field in terms of loading mode, loading rate, stress state, and degree of stress concentration.

Using a 90 percent stiffness reduction failure criterion, the Wöhler curves for the force-controlled four-point bending tests are provided in figure 9. The data shows that the softer systems fail faster, which is as expected for a force-controlled test. However, once more there appears to be no direct relation with the performance in the field (figures 4 and 5).



**Figure 9. Wöhler Curves for Force Control**

Note: Number of cycles to failure was determined at a 90 percent stiffness reduction. Mixture 3 was not tested due to materials and time limitations.

The load-controlled test data were also analysed according to the same criteria as listed above and once more the findings were not significantly altered by switching to a different definition of failure.

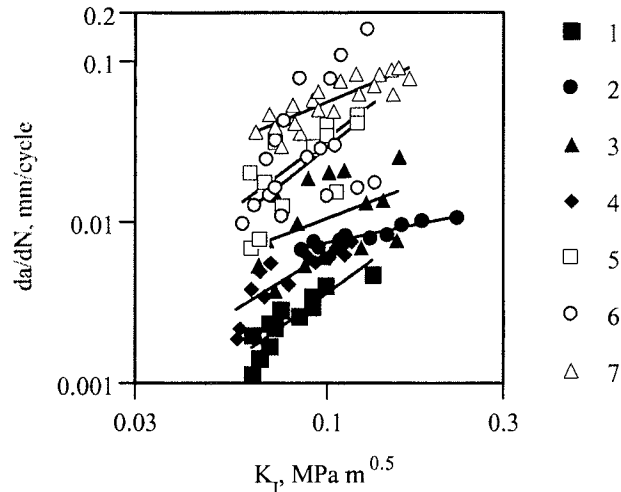
It is difficult to conceive that with heavy trucks driving over the pavement the loading can be anything else than a constant force situation. However, the stress state may be complex with a change from compressive to tensile components depending on the location relative to the position right underneath the tire. Further, the critical area where cracking initiates may be highly localized, making the situation more difficult to reproduce in a four-point bending experiment. For the relatively thin pavement used on Highway 655 the displacement reaches a certain level that depends on the composite pavement stiffness. Whether surface cracks appear will likely depend on the displacement, and hence the load applied and the pavement stiffness, but also on the strain tolerance of the materials. In order to draw meaningful conclusions from the four-point bending data, it may be necessary to involve a more sophisticated structural, mechanics, and energy analysis. However, this type of model development was beyond the scope of this work.

## DYNAMIC CREEP FRACTURE IN COMPACT TENSION

The dynamic creep fracture tests were conducted in compact tension with continuous 0.1 s haversine load pulses of 150 N. All tests were done at room temperature,  $25 \pm 1^\circ\text{C}$ . The number of load pulses was recorded at regular crack length intervals, and the data were fitted to an exponential. The exponential was numerically differentiated to obtain a smoothed crack growth rate, which was subsequently plotted versus the stress intensity factor,  $K_I$ , on a logarithmic scale. The results for all tests are given in figure 10.

The data shows that for all mixtures except the mixture representing section 2 there is a large degree of scatter which is similar to what is reported by others (Collop et al., 2004). The scatter can be attributed to the fact that this test was done on relatively small samples for a 12.5 mm maximum

aggregate size. Moreover, the nature of dynamic crack growth tests is such that large degrees of scatter are unavoidable.



**Figure 10. Dynamic Creep Crack Growth in Compact Tension**

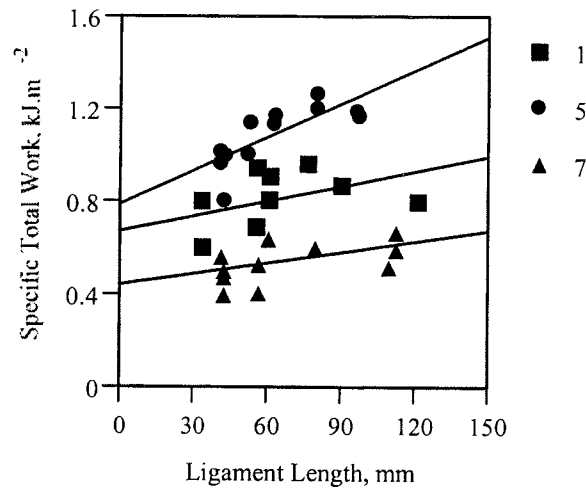
The relative rankings show somewhat expected trends similar to the load-controlled four-point bend results in that the softer systems (mixtures 7, 6, and 5) respond with high rates of crack growth compared to the stiffer systems (mixtures 1, 2, and 4). However, there appears to be no simple relationship between the rate of crack growth and the cracking severity in the field (figures 4 and 5). In order to draw meaningful conclusions from this data, it may be necessary to involve a more sophisticated structural analysis. Additional measurements of crack tip opening displacement in the fatigue test would be helpful to differentiate between good and not so good systems. However, such measurements are either difficult to perform or would involve a number of assumptions. Hence, our efforts have considered the essential work of fracture approach that provides measures of critical failure properties that are more easily obtained.

## DOUBLE-EDGE-NOTCHED TENSION

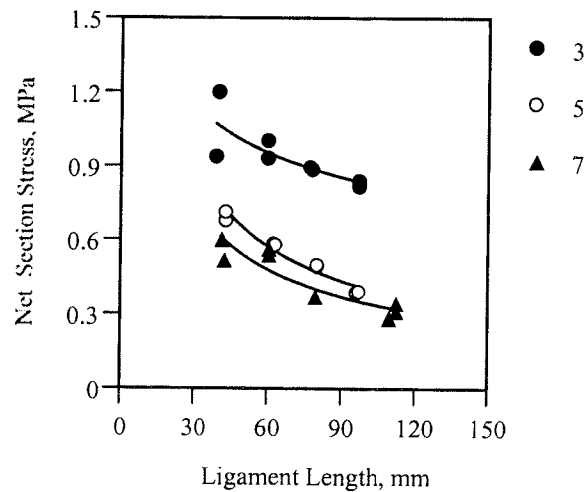
The DENT tests provide a measure of the essential and plastic works of fracture,  $w_e$  and  $\beta w_p$ , as well as an approximation of the critical crack opening displacement from the ratio of  $w_e$  over the net section stress of the smallest ligament specimen,  $\delta_t = w_e/\sigma_{net}$ .

Samples were all tested at  $25 \pm 1^\circ\text{C}$  and 5.4 mm/min, which guaranteed ductile failure in all systems. For most tests the reproducibility was found to be good although not as good as for the binder tests. The only exception was for the mixture representing section 4, which showed a somewhat higher variability. The force versus displacement curves were all found to be self-similar for different ligament lengths (i.e., were similarly shaped), and the data fit equation 2 with reasonable accuracy, thus providing some degree of validity to the essential work of fracture approach.

Representative results for the analysis according to equation 2 are given for mixtures 1, 5, and 7 in figure 11, while typical net section stresses as a function of ligament length are given for mixtures 3, 5, and 7 in figure 12. (Other mixtures gave similar results.) The data are interesting in several respects. First, the specific total works of fracture vary by a substantial amount between mixtures, although the scatter is larger than in the binder tests. Second, the net section stress for mixture 3 is significantly higher than that of mixtures 5 and 7. This causes the mixture to be less strain tolerant and explains to some degree the fact that section 3 was severely cracked after just the first winter. The same high net section stresses were found for mixtures 2 and 4, thus providing a simple explanation of why these performed so poorly in the field.

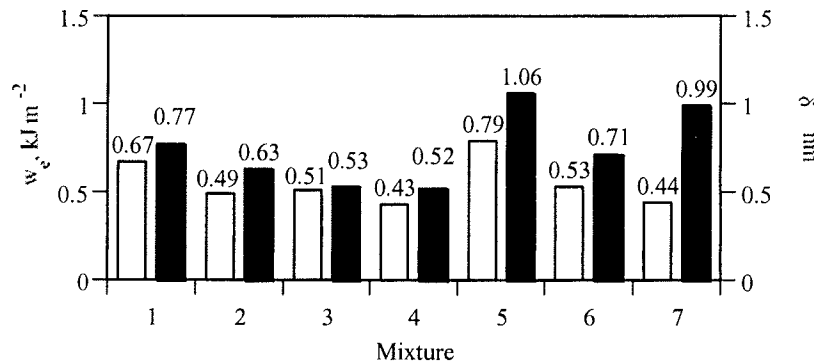


**Figure 11. Essential Works of Fracture for Mixtures 1, 5, and 7**  
 Note: Other systems gave similar results.



**Figure 12. Net Section Stresses as Function of Ligament Length for Mixtures 3, 5, and 7**  
 Note: Other systems gave similar results. Only mixture 4 gave somewhat higher variability.

The critical crack opening displacement was approximated by dividing the essential work of fracture by the next section stress in the smallest ligament length. The results for  $w_e$  and  $\delta_t$  for all mixtures are given in figure 13.



**Figure 13. Essential Works of Fracture and Approximate Critical Crack Opening Displacements for Mixtures 1-7**

Note: Open bars for essential works of fracture and solid bars for critical crack opening displacement.

The results in figure 13 are in general agreement with the severity of distress noted within the trial sections. Sections 2-4 have the lowest critical crack opening displacements and are the most severely cracked. Sections 1 and 5-7 have the highest critical crack opening displacements and have performed very well without showing signs of early distress.

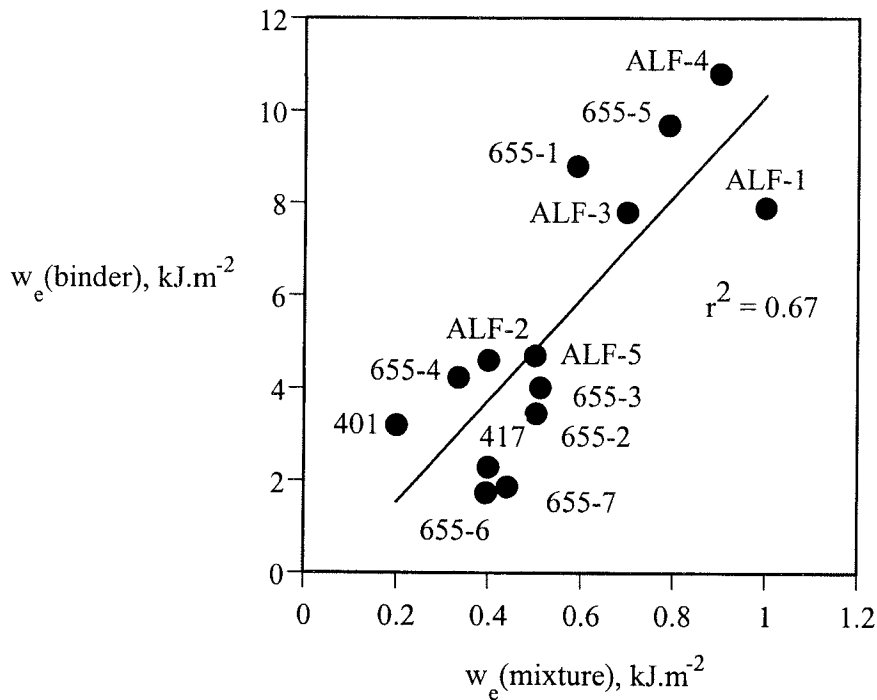
The fact that section 2 has cracked more than section 3 can likely be explained by the fact that binder 2 reversibly ages much more (figure 11 in part I of this report), which would have initiated more micro cracks during the excursions at low temperatures. Moreover, binder 3 reversibly ages less than binder 4, and appears to be of lower viscosity, and thus will heal cracks more readily during summer months, which could in part explain the lower amount of distress.

The future will likely show that sections 1 and 5 outperform sections 6 and 7, since they have significantly higher essential works of fracture and high temperature performance grades. In long-life pavements the rutting distress will undoubtedly feed the surface initiated cracking, making the picture more complex than what it appears after just three years of service.

It is interesting to note that sections 3-5 were all made with SBS-modified materials but the amount of polymer in sections 3 and 4 must have been much less than that used in section 5. The performance of this trial shows that not all PG 64-34 grades perform the same and that the use of acid and other non-polymer (chemical) modifiers may lead to premature cracking in service. (Section 4 was found to contain polyphosphoric acid in addition to SBS (Bodley et al., Submitted, AAPT 2007).)

Figures 14 and 15 show that there is a reasonable correlation between mixture and binder essential work of fracture but less so for the respective plastic works for the systems studied. This is reassuring since it is planned that the binder essential work will eventually replace the mixture test.

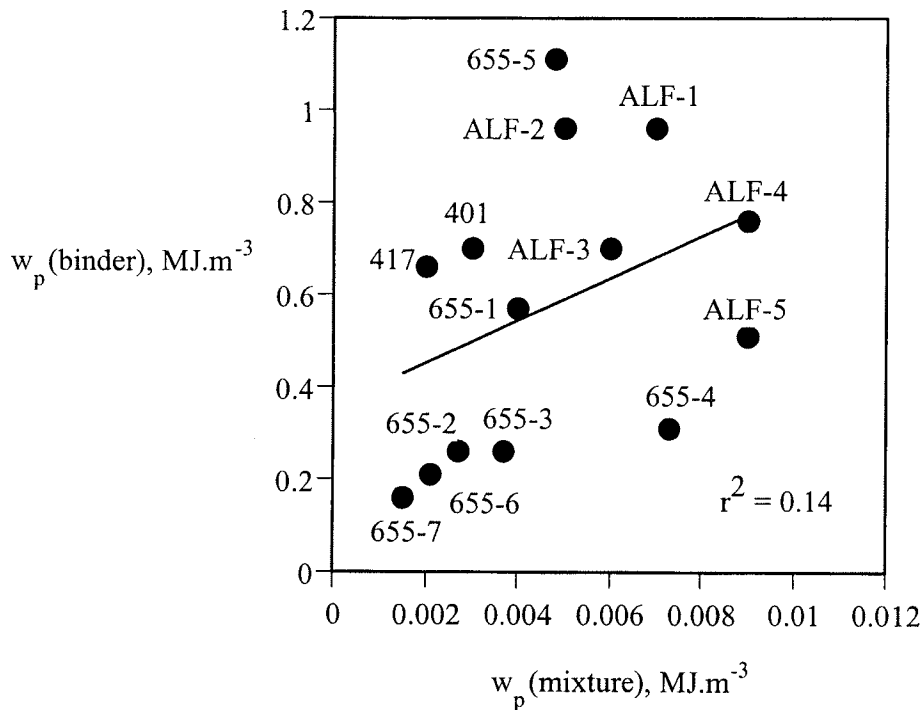




**Figure 14. Correlation between Binder and Mixture Essential Works of Fracture for Highway 655, FHWA ALF Materials and Hwys 401 and 417 Contracts**  
(For experimental details on ALF and 401/417 materials refer to Andriescu et al., 2004.)

The binder essential works of fracture are all about ten times higher than the mixture essential works. This is due to the fact that the stretching rates were different in the binder from those in the mix (100 mm/min versus 5.4 mm/min), the mixture was also stiffer due to the presence of large amounts of aggregate (fracture energy is inversely proportional to stiffness), and the mixture contained significant amounts of voids and weak interfaces which facilitate the propagation of cracks.

The fact that the binder plastic works of fracture correlate less with the mixture data stems from the fact that the plastic work represents the ability of the system to spread the energy dissipation to areas away from the fracture process zone. In the mixture the interfaces between binder and coarse aggregate experience a high level of triaxial tension and therefore fail prior to the mastic and binder regions. Hence, the plastic work is often less in systems prepared with tough polymer modified binders. However, recent work with materials from the FHWA accelerated loading facility have indicated that the essential works provide a reasonably good prediction for fatigue resistance through the crack tip opening displacement (CTOD) (Andriescu et al., 2006).



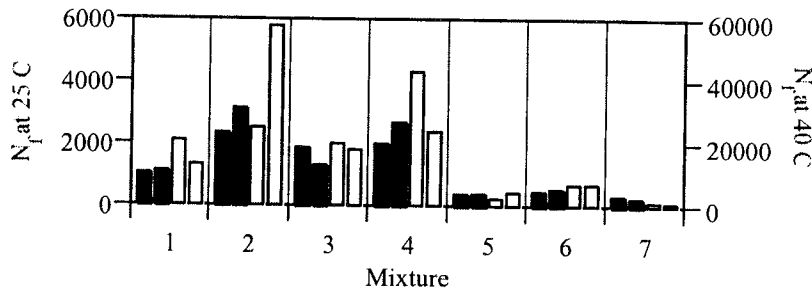
**Figure 15. Comparison between Binder and Mixture Plastic Works of Fracture for Highway 655, FHWA ALF Materials and Highways 401 and 417 Contracts**  
(For experimental details on ALF and 401/417 materials refer to Andriescu et al., 2004.)

It is also interesting to note that the mixtures with low plastic works of fracture in the binder (655-2, 655-3, and 655-4) showed a significant amount of wheel path distress after their first winter (70 m, 50 m, and 50 m, respectively). While those with higher plastic works (655-1 and 655-5) survived the first harsh winter unscathed (figures 4 and 5).

The large difference in ranking between binder CTOD (figure 29 in part II of this report) and mixture CTOD (figure 13 above) for sections 1, 3, 4, and 5 may have something to do with the aging differences between RTFO/PAV for the binder and short term oven aging for the mixture. Perhaps the interface in these polymer modified binders is weaker than it is for the oxidized and straight run systems in sections 2, 6 and 7. Another explanation may come from the fact that the net section stress used to calculate the binder CTOD might not have been appropriate in that the confinement in the binder films in the mixture could have been considerably higher than what it was for the 5 mm ligament DENT specimens. Finally, there could have been differences in oil absorption by the coarse aggregate. Clearly this issue needs to be further investigated with well controlled laboratory tests and eventually field trials.

## DYNAMIC CREEP IN UNCONFINED COMPRESSION

In addition to the previous tests, a number of dynamic compression experiments were done in order to compare the relative performance for the two different types of loading. Figure 16 provides the load pulses to failure for the seven mixtures at both 25°C and 40°C. The data show that the best performing mixtures in this test were made with the oxidized binder 655-2 and the two SBS-modified binders 655-3 and 655-4. These also happen to be the worst performing in the field with 70 m, 50 m and 50 m of wheel path cracks after just the first winter, respectively. In contrast, the best performing section 5, shows the second-worst performance in the dynamic creep test. These findings confirm that the mechanism in this test has little relevance for the field performance of these mixtures.



**Figure 16. Dynamic Creep Test Results for Highway 655 Mixtures at 25°C and 2,000 kPa (duplicates, solid bars) and 40°C and 400 kPa (duplicates, open bars).**

However, long-term fatigue performance may be confounded by the effects of rutting during summer months. Hence, it will be interesting to follow the relative performance of these sections to see if the harder mixtures will derive any benefits from their increased rut resistance or if they continue to perform poorly due to their lower strain tolerance during spring thaw.

## Conclusions

Given the results presented in this report, the following conclusions are provided:

- Binders with high essential works of fracture yield mixtures with high essential works of fracture. High essential works are likely a beneficial attribute to prevent fatigue distress in service.
- High plastic works of fracture are likely beneficial for the prevention of fatigue distress in service provided the essential work is acceptable.
- Mixtures with high crack opening displacements show less fatigue distress compared to stiffer mixtures with low crack opening displacements in thin pavements such as the one used for the Highway 655 trial.
- Binder crack opening displacement correlates poorly with mixture crack opening displacement and this is likely due to differences in RTFO/PAV aging of the binders and short term oven aging for the mixture, variations in interfacial strength, variations in oil absorption by the coarse aggregate (exudation), and/or factors related to the strain rate sensitivity of the binders.

How the above findings relate to long term field performance remains to be investigated with the Highway 655 trial and other well-designed test roads that are to be followed until sufficient distress is observed to draw meaningful further conclusions.

## Recommendations

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Given the review of the literature and the results presented in this report, the following recommendations are provided:

- The field trial on Highway 655 will need to be followed for at least the next 10-15 years. It is likely that the binders will age harden at different rates which will confound the analysis. However, recovered binders and tests on mixture samples cut from the field will provide insight into the long-term importance of the various fracture parameters.
- The construction of two new pavement trials, on Highway 417 (2006) and Highway 655 (2007), with binders that are more heavily modified will hopefully provide evidence for the hypothesis that tougher binders can reduce fatigue and low temperature cracking to an insignificant level.
- The reason for the poor correlation between binder and mixture crack opening displacement needs to be further investigated. The difference appears to be due to the large variations in net section stresses between binder and mixture and between various mixtures. This could be due to differences in RTFO/PAV versus short term oven aging, interfacial strength, oil absorption by the aggregate (exudation), and/or strain rate sensitivity. Hence, the development of a DENT test for the mastic is a priority.

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