# IMPORTANCE OF BITUMEN PHYSICAL HARDENING FOR THERMAL STRESS BUILDUP AND RELAXATION IN ASPHALT

Hussain Bahia<sup>1</sup>, Hassan Tabatabaee<sup>2</sup>, and Raul Velasquez<sup>3</sup>

<sup>1</sup> University of Wisconsin-Madison, 3350 Engineering Hall, 1415 Engineering Drive, Madison, WI, 53706, USA, hubahia@facstaff.wisc.edu

<sup>2</sup> University of Wisconsin-Madison, 3535 Engineering Hall, 1415 Engineering Drive, Madison, WI, 53706, USA, tabatabaee@wisc.edu

<sup>3</sup> University of Wisconsin-Madison, 3352 Engineering Hall, 1415 Engineering Drive, Madison, WI, 53706, USA, ravelasquez@wisc.edu

#### ABSTRACT

Bitumen is known to undergo significant time-dependent stiffening when stored at low temperatures. This phenomenon, often referred to as physical hardening, has been shown to have significant impact on the laboratory performance of bitumen. However, the importance of isothermal conditioning for asphalt and its effect on thermal cracking performance has been a subject of debate.

This paper investigates the effects of cooling rate and isothermal conditioning on thermal stress buildup and relaxation of different asphalt materials obtained from field pavement sections in MnROAD, Minnesota, USA. A relatively simple device was developed and used to simultaneously test two asphalts beams: a unrestrained beam from which the change in volumetric properties with temperature, and consequently the glass transition temperature  $(T_g)$  and coefficients of expansion/contraction, are measured; and a restrained beam used to measure thermal stress buildup.

Test performed at different cooling rates and various relaxation periods showed that at high cooling rates, isothermal contraction significantly affects the rate of thermal stress buildup. Furthermore, contrary to the expectation of full stress relaxation for visco-elastic materials, the thermal stress in restrained samples held at isothermal conditions for extended periods of time did not completely relax, converging to a constant residual stress. Fracture of specimens held isothermally occurred at a temperature higher than fracture temperature measured when specimen is monotonically cooled down. These results indicate that physical hardening may be responsible for unexpected thermal cracking observed in the field. The findings show the need to measure the glass transition of bitumen, their behavior under isothermal conditioning, and the use of realistic cooling rates for better prediction of thermal cracking.

**Keywords:** Low Temperature Performance, Thermal Stress, Stress Relaxation, Glass Transition, Isothermal Conditioning, MnROAD.

#### 1. INTRODUCTION

Thermal cracking is the most important distress in regions subjected to cold temperatures. It is believed that the excessive brittleness of the bitumen due to the increase in stiffness and decrease in the ability to relax stress lead to the buildup of thermally induced stress and ultimately cracking of pavements.

Visco-elastic materials such as asphalt can relax stress by viscous flow. Asphalt pavements are restrained from significant movement, thus thermally induced contraction can lead to significant stress build-up in the pavement. Due to the time-dependent behavior of visco-elastic materials, the higher the capability of the material to relax stress, the lower the thermal stress buildup, and consequently the lower the potential for low temperature cracking [1, 2]. Thus, stress relaxation has been considered an important factor in cracking resistance of asphalt pavements [3]. Researchers also consider factors such as the rate of cooling, coefficients of expansion/contraction, glass transition temperature, shape of master curve at low temperatures and the tensile strength to affect the critical cracking temperature [2].

The change in behavior near or below the glass transition temperature has been noted by many researchers in recent years [4, 5, 6, 7, 8, 9, 10]; the increase in brittleness as well as the time-dependent behavior of the material in this temperature range can have a detrimental effect on actual bitumen performance during its service life. Bouldin et al. [2] reported that the midpoint of the bitumen's glass transition, typically referred to as the "glass transition temperature", is in the vicinity of the pavement critical cracking temperature. Kriz et al. [9] showed that physical hardening can affect the position of the glass transition temperature in bitumen. Change in relaxation properties has been noted in bitumen and many polymers during physical hardening [8, 9, 11].

Researchers have noted the effect of isothermal conditioning, typically referred to as "physical hardening" or "physical aging", in amorphous material for many years. Struik described physical hardening in polymers as a type of thermo-reversible relaxation process, taking place in the glass transition region of amorphous materials [11]. The first comprehensive study on physical hardening in bitumen was reported during SHRP [4, 5]. Physical hardening is usually explained by the free volume theory proposed by Struik [11] and Ferry [12]. However, some researchers have also associated physical hardening with the crystalline domain and wax fraction of the bitumen [6, 7, 9]. Bahia and Anderson found that the approach used to account for the effect of changing temperature in viscoelastic materials (i.e., time-temperature superposition principle) can be applied to the stiffening effect of physical hardening with conditioning time by using a shift factor on the time scale [4].

Recently Hesp and Subramani [13] observed better correlations between Bending Beam Rheometer (BBR) results and field performance when bitumen samples were conditioned for 72 hours. It was also noted that bitumen whose Superpave grade was controlled by the m-value had a more significant loss of performance after isothermal conditioning, while those that were controlled by stiffness lost less.

Romero et al. [14] investigated the effect of different mineral fillers and different volumetric properties on the physical hardening of asphalt using the Thermal Stress Restrained Specimen Test (TSRST). The authors concluded that fracture rather than strength properties are affected by physical hardening. They also concluded that it cannot be inferred that a mixture will show the effects of physical hardening solely based on the bitumen exhibiting changes during testing after low-temperature isothermal storage. It must be noted that the researchers conditioned the specimens in unrestrained conditions, thus the stress buildup due to isothermal contraction was not considered in the conclusions. Evan and Hesp [15] suggested that inconclusive results pertaining to the importance of physical hardening in asphalt, is due to shortcomings and difficulties associated with the current TSRST test method used for low temperature performance assessment.

Researchers such as Shenoy [16] have claimed that stress relaxation in the bitumen can cancel out any effect of physical hardening in the asphalt, thus believing the phenomenon to be of no practical importance. Recent studies by others such as Falchetto et al., [17], Falchetto and Marasteanu [18] and Evans and Hesp [15] have concluded otherwise. Falchetto and his co-workers measured the increase in stiffness in both bitumen and asphalt BBR beams, showing that the semi-empirical Hirsch model can be used to predict the hardening of the asphalt beams based on the bitumen hardening [17, 18]. Evan and Hesp [15] showed that bitumen that had higher BBR grade loss after 72 hrs of conditioning retained more residual stress after relaxation.

In this paper, experimental results on asphalt beams obtained from field sections are used to show the importance of physical hardening for accurate estimation of thermal stress buildup, relaxation capabilities, and in general the thermal cracking susceptibility of asphalt. A new device that allows measuring thermal strain and stress as a function of time and temperature was used to show the consequences of physical hardening of bitumen on strain and stress of asphalt.

## 2. MATERIALS AND TESTING PROCEDURE

#### 2.1 Materials

The description of the materials used in this study are presented in Table 1. Loose asphalt were collected from field pavement sections in Minnesota, USA. Loose asphalt was compacted using Superpave Gyratory compactor

targeting design parameters provided by MnROAD engineers. The bitumen and aggregates used for asphalt preparation represent typical materials placed in pavements in the Mid-West region of the USA. As indicated in Table 1, bitumen used in these sections were modified with commonly used chemical and polymer additives.

Asphalt	Location	Description
PG58-34 PPA	MnROAD 33	Modified with Polyphosphoric Acid (PPA)
PG58-34 SBS+Acid	MnROAD 34	Modified with Styrene-Butadiene Styrene (SBS) and PPA
PG58-34 SBS	MnROAD 35	Modified with SBS
PG58-34 Elvaloy +Acid	MnROAD 77	Modified with PPA and Elvaloy
PG58-28	MnROAD 20	Neat
PG58-34	MnROAD 22	Unknown modification
Wisconsin	Wisconsin	Binder used in construction of SMA pavement
PG 58-34 (CAN)	MN CR 112	Canadian blend, Elvaloy modified
PG 58-28 (VAL)	MN CR 112	Valero binder
PG 58-28 (MTN)	MN CR 112	Marathon binder
PG 58-28 (CIT)	MN CR 112	CITGO binder

#### Table 1: Description of Asphalt Tested.

# 2.2 Asphalt Thermal Cracking Analyzer (ATCA)

In an effort to measure the effect of isothermal conditioning on thermal stress response of asphalt, a device was developed that simultaneously tests two beams; one unrestrained, and the other with restrained ends. The unrestrained beam is used to measure thermo-volumetric properties such as the glass transition temperature ( $T_g$ ) and coefficients of expansion/contraction ( $\alpha_l$  and  $\alpha_g$ ). The restrained beam is used to capture the induced thermal stress due to prevented contraction in the sample when subjected to low temperatures. This device is currently being referred to as the Asphalt Thermal Cracking Analyzer (ATCA). The system is schematically shown in Figure 1.



Restrained sample





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# Figure 1: a) Illustration of the Asphalt Thermal Cracking Analyzer (ATCA) system; (b) restrained beam setup, (c) unrestrained beam setup, and (d) restrained beam after failure.

The unrestrained and restrained samples are produced from one Superpave gyratory compacted sample. Using a masonry saw, four prismatic beams of 5 by 5-cm in cross section and 15 cm long are cut from 17 cm sample. Two of these beams are sawed in half to produce four 7.5 cm blocks. By gluing a 7.5 cm block to each end of the two 15 cm blocks, two 30 cm beams are produced. As both beams are produced from the same sample and both are exposed to the same thermal history, the stress buildup, glass transition temperature, coefficients of thermal contraction/expansion can be used to get a comprehensive picture of the low temperature performance of the asphalt. Figure 2 shows typical results obtained from the ATCA system when temperature is decreased at the rate of  $1^{\circ}C/min$  from + 30 to -70°C.



Figure 2: Typical ATCA result for asphalt (a) Tg (b) stress buildup.

Using the ATCA system, many complex experiments, such as thermal cycling with isothermal steps and measurement of thermal stress relaxation are possible. One such test using the ATCA is a thermal stress relaxation experiment in which the chamber temperature is reduced to a predefined low temperature at a controlled cooling rate (0.1 to 1 °C/min), continuously monitored using temperature probes placed within the core of the asphalt beams. The temperature is then kept at the predefined temperature for prolonged periods ranging between 2 and 10 hrs. The stress buildup in the restrained specimen, and the strain in the unrestrained sample, are measured continuously. The results are used to plot curves of thermal stress as a function of core temperature and test time during the extended isothermal condition.

# 3. ANALYSIS AND RESULTS

Using the ATCA, cooling and relaxation experiments were carried out at different rates, isothermal temperatures and isothermal relaxation times. For all experiments thermal stress was observed to build up as the temperature decreases in the restrained beam. Temperature reduction was stopped at a predefined temperature to start the isothermal stage. Although the temperature measured at the core of the asphalt sample was kept constant, the sample stress continued to build up even after the core temperature had stabilized. Isothermal contraction was also observed simultaneously in the unrestrained beam. As the isothermal conditions continued, the stress gradually started to relax. This trend was observed for all asphalt samples tested, as shown in Figure 3 in which two identical asphalt samples were cooled to -20°C and then held isothermally for 5 and 10 hrs, respectively. It is observed that after the initial isothermal stress buildup, the rate of build-up gradually decreases, followed by a relaxation of stress until stabilizing at a constant value over time.



Figure 3: Isothermal stress behaviour in asphalt after 5 and 10 hrs of isothermal conditions.

Furthermore, it was noted that the amount of isothermal stress (Figure 4b) and strain (Figure 4a) buildup was dependent on the rate of cooling. When a rate of  $0.1^{\circ}$ C/min (6°C/hr) was used as much as 10% of the ultimate stress continued to build up after achieving a stable core temperature (Figure 4b). When rate was increased to an extremely fast cooling rate of 1°C/min, most of the ultimate stress is reached after achieving a stable core temperature, although the specimen ultimately reached the same stress levels as with the slower rate as indicated in Figure 4. Tests performed at an intermediate rate of 0.5°C/min resulted in isothermal stress buildup in between the two extreme cooling rates.

Researchers in earlier studies have used many cooling rates to test and model field conditions. Bouldin et al. [2] used  $3^{\circ}$ C/hr to match studied field sections, while suggesting that resulting cracking temperature may be "bumped" up or down for faster and slower rates, respectively. Modeling by Bahia et al. [8] for rates lower than  $10^{\circ}$ C/hr showed that reducing cooling rate corresponded to a shift in thermal stress buildup toward lower temperatures. SHRP researchers reported that although typical field cooling rates seldom exceed 2.7°C/hr, most TSRST tests are done at rates of  $10^{\circ}$ C/hr or higher to reduce testing time [1]. Tests conducted during the SHRP study showed a decrease in fracture temperature as the cooling rate varied between 1 to 5°C/hr, while the effect on tensile strength varied for different samples. They noted that previous researchers reported little or no effect on fracture temperature and tensile strength for different cooling rates higher than 5°C/hr. They concluded that although these cooling rates do not necessarily match typical field conditions, they are sufficient to assess relative performance of specimens.

In this study, it is hypothesized that the observed isothermal behavior is due to the time-dependent nature of thermal contraction as temperature approaches the glass transition region. The complete explanation of the mechanism of glass transition and physical hardening is beyond scope of this paper and can be found in Bahia and Anderson [21], Tabatabaee et al. [22] and Bahia and Velasquez [10].



Figure 4: Comparison of thermal stress and strain during cooling and isothermal conditions at 0.1 and 1°C/min cooling rates.

If cooling rate is sufficiently slow, the specimen has ample time to fully contract; but as the cooling rate increases, although core of samples can reach conditioning temperature, the amount of delayed contraction increases. The delayed contraction takes place over time after sample core reaches isothermal contraction, hence causing the specimen to buildup thermal stress while at a constant temperature. After sufficient time has passed, all samples achieve full contraction, thus ultimately building up the same amount of thermal stress. An example of this behavior is shown in Figures 4(a) and 4(b).

An important consequence of the observed behavior is that thermal stress will build up at slower rate during cooling if the rate is high enough to not allow for complete contraction during the cooling period, as shown in Figure 4(c). Although at first glance this seems counter intuitive, it must be pointed out that for sufficiently slow cooling rates in which full contraction is taking place during cooling, the trend will be opposite, as the slower cooling rates will allow for more thermal stress relaxation and consequently a lower rate of stress build up during cooling.

Figure 5 highlights a noteworthy observation during sample thermal stress buildup with and without isothermal conditioning. It can be seen that the trend of stress buildup when the temperature was decreased at a constant rate down to the point of cracking varies significantly from the sample which is cooled at the same rate but held isothermally at a certain temperature. The asphalt beam held isothermally built up stress over time at a constant temperature to stress

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levels achieved at temperatures more than 10°C lower when the sample was continually cooled. This important observation points out the possibility of pavements cracking at temperatures well above what is expected, if held isothermally at low temperatures for a period of time.



Figure 5: Typical stress build up in restrained beam using the ATCA, with and without the isothermal conditioning step.

Another important observation made during isothermal conditioning of various asphalts is shown in Figures 6 and 7. It is observed that during isothermal conditions, asphalt samples can reach a critical value of thermal stress that result in sample fracture. The importance of this observation becomes more apparent when considering that under continuous constant cooling these samples would not have built up this level of stress until temperatures well below the current fracture temperature. This may easily explain discrepancy between predicted low temperature cracking temperatures and observed under- performance in the field, underscoring the importance of considering the potential of isothermal contraction and time-dependent strain in asphalt material when selecting appropriate material for specific climatic conditions.



Figure 6: ATCA restrained beam fracture during isothermal conditions (MN County Road 112-Valero).



Figure 7: ATCA restrained beam fracture under isothermal conditions (MN County Road 112-CITGO).

The possible effect of different levels of isothermal physical hardening of bitumens in asphalts was observed when comparing the performance of field sections constructed recently in a county road in Minnesota. Asphalts using bitumen of identical Superpave performance grades (PG 58-28) were used in the construction of the test sections. After being exposed to identical climatic conditions, one of the sections was observed to have cracked two times more than the others. Although various performance tests failed to differentiate the bitumen significantly, physical hardening tests using both the Bending Beam Rheometer for the bitumen and the ATCA for the asphalt showed that one of the bitumen has considerably higher susceptibility to isothermal contraction than the other. This bitumen corresponds to the worse field performance, as indicated in Figure 8. Further information about this field study will be presented in a future publication.



Figure 8: Comparison of physical hardening susceptibility of two bitumen of identical Superpave performance grades. The field section using asphalt of source "A" cracked two times more than asphalt of source "B".

#### 4. CONCLUSIONS

This paper shows asphalt can undergo isothermal contraction that significantly affects the rate of thermal stress buildup and relaxation. Contrary to the expectation of full stress relaxation for asphalt materials after terminating the cooling step, the samples held at isothermal conditions for extended periods of time converged to a constant residual stress value. It was also observed that specimens held isothermally can fracture at a temperature higher than the fracture temperature measured when specimen is cooled at a constant rate. Based on these observations, it is believed that physical hardening may be responsible for unexpected thermal cracking observed in the lab and the field. Time-dependent contraction is important in asphalt and has a continuous effect during thermal stress buildup, most prominently in or below the glass transition region. Effect of isothermal contraction becomes very important when using laboratory tests at faster cooling rates to predict field conditions. The results of this paper suggest that it is critical to measure the glass transition of asphalt, and that asphalt behavior under isothermal conditioning is a key factor in <u>5th Eurasphalt & Eurobitume Congress</u>, 13-15th June 2012, Istanbul

accurate prediction of thermal cracking in the field. The results also show that for better prediction of thermal cracking it is necessary to account for the effect of physical hardening when using laboratory cooling rates higher than cooling rates experienced in the field.

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