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Nanoindentation Characterization of Aging in Different Phases of an Asphalt Concrete

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<td>The research project is funded by SOLARIS in cooperation with the NMDOT</td>
<td>To this day, asphalt researchers have studied only bulk state (liquid or semisolid) of an asphalt binder and using traditional rheometers. In roadway pavements, an asphalt binder never exists in a liquid or semisolid state, rather it exists as a solid, as one of the three phases/components (asphalt, aggregate, voids) of an asphalt concrete. While rheometric techniques used mostly bulk asphalt material, recently nanoindentation technique has brought an opportunity to indent an asphalt binder while it resides in an asphalt concrete as a solid phase. One of the problems of indentation is, it is difficult to indent a specimen phase and it is always difficult to identify whether the indenter is hitting a void or binder or mastic. In this study, using a nanoindentation creep test, two Performance Grade (PG) binders: PG 70-22 and PG 64-22 are studied for their behavior at a bulk state and at a phase state. Indentation load-displacement data was analyzed by the readily available Oliver-Pharr method to obtain elastic modulus and hardness for the aggregate phase of AC. However, Oliver-Pharr is not applicable to binder and mastic data due to their viscous nature. Therefore, viscoelastic spring-dashpot- rigid body model is used to obtain and compare viscoelastic material properties of binder and mastic phase of AC for different aging conditions.</td>
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Introduction

It is known that an asphalt concrete consists of asphalt binder and aggregate. Aggregate consists of: coarse aggregate and fines. In this study, fines are defined as aggregate materials that pass through a #200 sieve. Asphalt binder creates a coating or film around the coarse aggregate, which is defined as the binder phase of AC. Fines are believed to be trapped inside an asphalt film or mixed with asphalt binder, creating a composite material called mastic. Thus, AC has three phases: mastic, asphalt film binder, and coarse aggregate, as shown in fig. 1. The test methods developed, to this day, are mostly rheological shear and bending beam tests performed on the bulk volume of binders and mastic samples. No studies have been performed to determine the stiffness and hardness of binder and mastic when they reside in asphalt concrete (AC) as phase state. Because the existing tests used in the asphalt area cannot be performed on binder and mastic while they are an integral part of AC. Recently, nanoindentation has brought an opportunity to conduct tests on the binder, mastic and aggregate while they are integral parts of AC (Faisal et al. 2014, 2015, Tarefder and Faisal 2014, Veytskin et al. 2016). Because, in nanoindentation test, a nanometer size tip, which is smaller than binder film thickness as well as mastic phase, can be used in these phases to indent them. There is an urgent need for fundamental research that would provide a clear understanding of “different phases” of asphalt. However, the problem with nanoindentation is, it is difficult to identify whether it is hitting on a binder or mastic or aggregate phase of AC. The current study attempts to develop a technique using the viscoelastic creep response of the AC phases.

![Figure 1. Different phases of Asphalt Concrete (AC).](image)

However, how and to what extent these phases play roles in comparison with bulk state different phases of AC are not known yet. In the proposed study, nanoindentation test will be used to measure mechanical properties such as stiffness and hardness of asphalt binder, mastic, and aggregate while they are in phase state of AC sample. The use of the nanoindentation technique in the field of asphalt is rather new (Faisal et al. 2014, 2015, Tarefder and Faisal 2013a, 2013b, 2014, Veytskin et al. 2015, 2016, Ossa et al. 2005, Ossa and Collop 2007, Jager et al. 2010, Faisal et al. 2015). Asphalt is known to be a viscoelastic material that exhibits creep behavior.
(Ossa and Collop 2007, Tarefder and Faisal 2013, Faisal et al. 2015). Tarefder et al. (2010) developed a range of indentation derived stiffness and hardness values of aged asphalt. In another study, Faisal et al. (2014, 2015) conducted a study of nanoindentation on moisture damaged AC mixes of New Mexico. Jager et al. (2010) studied the thermal effects on the mechanical properties of the asphalt binder. In previous of the authors, aging characterization of mastic and aggregate phases were conducted by visual observation and using elasto-plastic model (Tarefder and Faisal 2013b). However, during nanoindentation AC phases can only be identified by using viscoelastic, which has been adopted in the current study. In addition, visco-elasto-plastic analysis of the nanoindentation data extracted three different attractive material parameters, like stiffness, hardness and indentation viscosity, when they were an integral part of AC body.

To understand, how and to what extent AC phases play role compared to bulk state, current study uses the aging phenomena to compare different conditions. Aging is a complex phenomenon and involves physical and chemical modifications to asphalt molecules. These modifications, or changes, have dramatic effects on asphalt binder and asphalt concrete properties such as viscoelasticity (stiffness, hardness), and adhesion/cohesion (Newcomb et al. 2015, Veytskin et al. 2015). As asphalt binder ages the nonpolar molecule part decreases and polar part increases (Petersen 2009). The increased amounts of polar molecules change the nature of the asphalt and lead to increased stiffness and slower stress-relaxation rates. Currently, aging in asphalt is one of the major concerns in the asphalt research field, as aging can lead to the development of distresses like fatigue or thermal cracking in asphalt pavement (Raquel et al. 2011, Huet 1965). Because aging phenomena has not be understood well yet, several models such as top-down cracking model, fatigue model, used in the newly developed pavement ME design software, are incomplete or underdevelopment. There is a need for a mechanical understanding of aging in asphalt. Therefore, an understanding of the phenomena of aging can be benefited or enhanced by measuring the stiffness and hardness of asphalt binder using the nanoindentation technique, which is done here.

**Objectives**

This study was conducted to provide a comprehensive evaluation of the material characteristics three different phases of AC and comparative aging evaluations. The major tasks carried out in this research were:

- Identification of specific AC phases based on materials softness and indentation depth.
- Generation of a database of unaged vs. aged modulus and hardness of binder, aggregate, and mastic.
- Comparison of aging in different phases of AC will provide a relative increase in aging index, defined by the stiffness of aged by the unaged property.
A model to analyze nanoindentation data to determine and compare creep behavior of aged and unaged asphalt.

**Task 1: Sample Preparation for Nanoindentation Testing**

**Aggregate and Asphalt Binder Collection**

Performance Grade (PG) asphalt binder samples (PG 64-22, PG 70-22) were collected from the local contractor associated with the New Mexico Department of Transportation (NMDOT). Limestone aggregate samples were collected from I-40 instrumented section construction site in New Mexico. In this report, PG 70-22 samples are defined as Binder1, and PG 64-22 samples are defined as Binder2.

**Asphalt Concrete Sample Preparation**

A Superpave gyratory compactor was used to compact the loose asphalt concrete mix. Figure 2 shows the gradation curve of aggregate used for making SP-III mix according to NMDOT specification. PG 64-22 and PG 70-22 were used to make asphalt concrete samples. The initial dimensions of the cylindrical specimens were 150 mm in diameter and around 170 mm in height. These specimens are then cored and sawed to have finished specimens of diameter 100 mm and of height 150 mm. To achieve consistency in the test results the target air void was set at 5.5±0.5 for the finished specimen, and therefore, several trial samples were compacted at the beginning to reach the target air void. To determine the bulk specific gravity of the finished cylindrical specimens and thus the air void content, AASHTO T 166 standard specification was used.

![Figure 2. Gradation curve of the asphalt concrete mix.](image-url)

Coring was performed to prepare nanoindentation sample from the middle part of the gyratory sample. The middle part of the gyratory compacted sample is used because this part has the most
uniform air void distribution. The required sample size to conduct nanoindentation test can be as small as a few millimeters. In this study, a 25-mm diameter sample was prepared. Next, the 25-mm diameter sample was polished to produce a smooth surface. Surface smoothness is crucial for nanoindentation test since the contact area of the nanoindenter is measured indirectly from the depth of penetration (Tarefder et al. 2010). Polishing of nanoindentation sample is performed using the water-cooled polishing machine. To produce a smooth surface without dislodging any small aggregate form the 25 mm diameter sample, a sequence of water-resistant silicon carbide polishing papers with decreasing abrasiveness are utilized. The set of polishing papers are 100, 200, 400, 600, 800, 1,200 and 1,500 grit sizes each for the duration of 150 seconds. Then the polished nanoindentation sample is washed to remove dust particles. Ideally, one can argue that damage has occurred on the sample surface during polishing. Therefore, such sample is not appropriate for aging study. The fact is that both the unaged and aged conditioned samples are subjected to polishing, which is required for the indentation tip to locate the surface during testing. Therefore, it can be counter-argued that both samples were subjected to the same damage during polishing. Figure 3 shows polished AC sample for nanoindentation testing.

![Figure 3. Asphalt Concrete Sample for Nanoindentation.](image)

**Asphalt Binder Sample Preparation**

PG 64-22 and PG 70-22 binder samples were used in this study. Figure 4 shows a laboratory prepared asphalt binder film on glass substrate. As the first step, a glass slide surface 12.7 mm × 12.7 mm was selected and weighed in scale up to 4 significant decimal digits of grams. Next, the glass slide was wrapped with high-temperature resistant tape. The tape was placed so that it formed the 6.35 mm square gap area previously outlined in the binder. Then, hot polymer modified liquid asphalt binder was poured into the gap between the tape strips. The polymer-modified binders were melted by heating them to 163 °C for an hour. The asphalt coated surface was placed in the oven at 163 °C for 10 min in order to have a smooth surface, cooled at room temperature and the tapes were removed. Finally, the glass slide with the asphalt coating was weighed again to measure the amount of asphalt binder. From the known area, the density of the asphalt binder and mass the thickness of the binder film was measured. The film thickness varied...
within a range of 40 µm to 80 µm to avoid substrate effect on test results. Three replicated samples are used to capture the variability within one each set.

![Asphalt Binder Sample for Nanoindentation](image)

**Figure 4: Asphalt Binder Sample for Nanoindentation.**

**Task 2: Sample Conditioning (Aged and Unaged)**

To simulate long-term aging, the fabricated AC specimens of task 1 were conditioned according to the AASHTO R30 standard practice. The standard requires that the specimen should be kept in a forced draft oven at a temperature of 185 °F (85 °C) for 5 days. According to, the AASHTO R30 standard is expected to simulate long-term aging of a mix in the field over a period of 5 to 7 years. Assuming, the conditioning will simulate an aged specimen corresponding to a 5-year aging period and successive long-term aging by AASHTO R30 will simulate a field aging period which is a progressive linear multiple of 5-7 years period, this study prepared a total of 3 cylindrical samples, simulated aging period of zero year unaged/ level zero aged, 2 years/2 days in draft oven, 5-7 years/ 5 days in draft oven. During the conditioning period, the specimens were handled carefully to avoid any disturbance which could cause damage to the specimens.

**Task 3: Nanoindentation Testing**

**Preliminaries**

In a nanoindentation test, an indenter tip of a known modulus of elasticity and geometry is loaded to penetrate a sample surface and then unloaded. Modulus of elasticity ($E$) of the sample is determined from the load-displacement data. The area of contact at full load is determined from the measured depth of penetration and the known geometry of the indenter tip. Sample hardness ($H$) is calculated by dividing the maximum load by the contact area. A typical load-displacement curve is shown in figure 5(a). A sitting load is typically applied initially to facilitate a contact between the tip and sample surface. Next, the load is increased gradually from point a to b. The tip is unloaded at the maximum load point b. The unloading path is assumed to be
elastic for most of the elastoplastic material. The unloading curve does not come back to point a due to plastic deformation in elastoplastic materials. The slope of the unloading curve at point b is usually equal to the slope of the loading curve at point a.

\[ h_c = \text{vertical depth along which contact is made}; \]
\[ h_e = \text{elastic depth recovery during unloading}; \]
\[ h_p = \text{final depth after unloading}; \]
\[ h_{\text{max}} = \text{depth at maximum load}; \]
\[ h_s = \text{displacement of the surface at the perimeter of the contact}. \]

(a) Load-Displacement Curve

(b) Indentation Depth
Figure 5: Schematic of the Indentation Test.

Figure 5(b) shows the surface profile as a function of the penetration depth during loading and unloading. Here, $h_{max}$ is the total depth of indentation at a maximum load, $h_{p}$ is the total depth of indentation that is unrecovered, $h_{s}$ is the depth of the surface at the perimeter of the indenter contact and $h_{c}$ is the vertical depth along which the contact is made between the indenter and the sample. Therefore,

$$h_{c} = h_{max} - h_{s}$$  \hspace{1cm} (1)

The depth of impression that is recovered is,

$$h_{e} = h_{max} - h_{p}$$  \hspace{1cm} (2)

**Oliver-Pharr Method**

The Oliver and Pharr method are based on the elastic contact between a rigid sphere (tip) and a flat surface (sample) (Oliver and Pharr 1992). If the indentation load $P$, penetration depth $h$ is recorded as the load-displacement curve, the reduced elastic modulus $E^*$ can be found from the load-displacement curve. However, the equation also relates to the indentation radius. The elastic modulus of the indented sample can be inferred from following equation, where indenter radius $R$ and contact radius $a$:

$$\frac{dP}{dh} = 2E^* \sqrt{Rh} = 2E^* a$$  \hspace{1cm} (3)

The projected area at the maximum load can be defined as $A = \pi a^2$. Therefore,

$$S = \frac{dP}{dh} = \frac{2}{\sqrt{\pi}} E^* \sqrt{A}$$  \hspace{1cm} (4)

where $S$ is the unloading stiffness or slope of the unloading curve;
How to Find $S$

Oliver and Pharr used a power law function to fit the unloading path of the load-displacement curve (23). The power law function used by Oliver-Pharr is shown in Eq. (6):

$$P = \alpha (h - h_f)^m$$

where $h$ is the depth of penetration, $h_f$ is plastic depth, $\alpha$ and $m$ are curve fitting parameters related to tip geometry. $m$ is equal to 1 for the flat-ended cylindrical tip, $m$ is 1.5 for the spherical tip, and $m$ is 2 for the conical tip (Berkovich tip). The slope is measured by differentiation in the above Eq. (6) at the onset of unloading.

How to Find $A$

Oliver and Pharr defined the projected area $A$ as a function of $h_c$ defined in Eq. (1). Oliver and Pharr extrapolated the tangent line to the unloading curve at the maximum loading point down to zero loads. This yields an intercept value for depth which estimates the $h_s$ by:

$$h_s = \varepsilon \frac{P_{\text{max}}}{S}$$

Therefore,

$$h_c = h_{\text{max}} - \varepsilon \frac{P_{\text{max}}}{S}$$

where $\varepsilon$ is a geometric constant. $\varepsilon = 0.72$ for conical tip, $\varepsilon = 0.75$ for Berkovich tip, and $\varepsilon = 0.72$ for spherical tip. The project area is measured by:

$$A = \pi a^2 = \pi (Rh_c)$$

where $R$ is known and $h_c$ is calculated using the above Eq. (9).

How to Find $E$

Timoshenko and Goodier found the reduced elastic modulus, $E^*$ is related to the modulus of the indenter and the specimen and given by (Oliver and Pharr 1992):

$$\frac{1}{E^*} = \frac{1 - \nu^2}{E} + \frac{1 - \nu_i^2}{E_i}$$

where $E$ is Young’s modulus of the material, $\nu$ is Poisson’s ratio of the material, $E_i$ is Young’s modulus of the indenter and $\nu_i$ is Poisson’s ratio of the indenter, $E^*$ is the reduced modulus. One can find the elastic modulus of the sample, $E$ using Eq. (10).

How to Find Hardness, $H$
Hardness, \( H \), is defined by the maximum load divided by the projected area:

\[
H = \frac{P_{\text{max}}}{A}
\]  

where \( P_{\text{max}} \) is peak load and \( A \) is projected area of contact at the peak load. The unit of hardness is given in N/m\(^2\)=Pa.

**Overview of SDR Model**

Till today, nanoindentation testing is mostly performed on elasto-plastic materials, which show well-defined loading (elasto-plastic) and unloading (elastic) behavior suitable for analyzing using the well-established Oliver-Pharr method (Oliver and Pharr 1992, Oyen and Cook 2002, Oyen and Co 2007). The Oliver-Pharr method of analysis uses the slope of the unloading (elastic) curve in the modulus calculation as discussed above. Nanoindentation has recently been used to understand time-dependent creep behavior of viscoelastic materials such as asphalt (Tarefder and Faisal 2013, Faisal et al. 2015). In case of viscoelastic materials such as asphalt, the slope of the unloading curve becomes negative due to the continuous viscous flow. The material is essentially unloaded visco-elastically, instead of elastically. Therefore, use of such unloading data in the Oliver-Pharr method results in an inaccurate value of modulus of elasticity (Tarefder and Faisal 2013a).

To extract the creep response of the material the displacement during loading and unloading are ignored. It is assumed the loading and unloading are very fast compared to creep loading time of the material. The creep response of the material is modeled using linear viscoelastic (LVE) analysis of the material. According to LVE theory, the load-displacement response of an indenter is expressed by a quadratic elastic load-displacement relationship, shown in Eq. (12) (Faisal and Tarefder 2013b, Oliver and Pharr 1992, Oyen and Cook 2002, Oyen and Co 2007).

\[
h^2(t) = \frac{\pi}{2} \cot \psi \int_0^t D(t-u) \frac{dP_0(u)}{du} du
\]

where \( P_0 \) = indentation load
\( h \) = displacement due to applied load in a material
\( \psi \) = include a half angle of Berkovich indenter and
\( D(t) = 1/E \), is creep compliance and \( u \), is a dummy variable for integration and delayed response is presented by \((t-u)\) variable instead of time variable \( t \).

According to Generalized Power Law (GPL), the creep compliance of thin film material can be expressed as,

\[
D(t) = D_0 + D_1 t^n
\]

In the study, generalized power law has been used to represent the creep compliance of the thin film material. Here, \( D_0, D_1, \) and \( n \) are regression coefficients. \( D_0 \) represents the short term behavior while \( D_1 \) represents the long-term behavior of the material. The current study uses the creep compliance parameters of bulk asphalt binder and mastic material to identify the specific
phases from the phase state of AC, as within LVE limit material creep compliance parameters does not depend on the state of stress. After phase identification, the load-displacement curves were introduced in Spring-Dashpot-Rigid body (SDR) model. Figure 6 shows a three element SDR model, which was found to be the most suitable model for asphalt in a previous study by the Tarefder and Faisal (2013b). Though the creep displacement remains within the LVE region of the material, however, the complex loading pattern of nanoindentation test on asphalt is analogous to non-linear visco-elasto-plastic behavior. Therefore, in SDR model all the model elements are shown proportional to the non-linear quadratic function of the element to render the non-linear behavior of load-displacement curve.

![Figure 6. Spring-Dashpot-Rigid (SDR) element model.](image)

The final form of the SDR can be found in Tarefder and Faisal and is given below:

**Loading Curve**—The slope of the loading curve is expressed as:

\[ P(t) = kt \]  
\[ \frac{dP}{dt} = \frac{dP_e}{dt} = \frac{dP_v}{dt} = \frac{dP_p}{dt} = k; \quad 0 \leq t \leq t_R \]  

where \( k \) represents the loading and unloading rate, \( t \) is a time variable, \( t_R \) represents loading and unloading time, \( t_C \) represents the dwell time, \( P \) represents load, \( P_e \) represents elastic load, \( P_v \) represents viscous load, \( P_p \) represents plastic load and \( P_{\text{max}} \) is the maximum indenter load.

Substituting the slope value in Eq. (12):

\[ \frac{dh}{dt} = \frac{(kt)^{1/2}}{(\alpha_2 \eta)^{1/2}} + \frac{1}{(kt)^{1/2}} \frac{k}{2(\alpha_1 E'')^{1/2}} + \frac{1}{(kt)^{1/2}} \frac{k}{2(\alpha_3 H)^{1/2}} \]  

where \( H \) is the plastic deformation resistance or hardness of the material, and \( \alpha_3 = \pi \tan^2 \psi \) is a dimensionless geometry parameter for sharp indentation with effective included angle \( 2 \psi \). The dimensionless constants are for the Berkovich tip as: \( \alpha_3 = \pi \tan^2 \psi = 24.5 \) and geometric constant.
\( \alpha_1 = \alpha_2 = 4.4 \). In Eq. (16), \( h \) represents indentation depth, \( E' \) is the plane strain modulus of asphalt and \( \eta \) represents indentation viscosity. By integrating Eq. (16):

\[
h^{\text{LOAD}}(t) = (kt)^{1/2} \left( \frac{2t}{3(\alpha_2 \eta)^{1/2}} + \frac{1}{(\alpha_1 E')^{1/2}} \right) + \frac{1}{(\alpha_1 H)^{1/2}}
\]

(17)

Creep Curve—Slope during holding time (see Figure 2) can be expressed as:

\[
\frac{dP}{dt} = 0; \quad t_R \leq t \leq t_e + t_R
\]

(18)

Substituting this in Eq. (16) and integrating the resulting equation gives:

\[
h^{\text{CREEP}}(t) = \int_{t_R}^{t_{e+R}} \frac{(P_{\text{max}})^{1/2}}{(\alpha_2 \eta)^{1/2}} dt
\]

(19)

\[
h^{\text{CREEP}}(t) = \frac{(P_{\text{max}})^{1/2}}{(\alpha_2 \eta)^{1/2}} (t - t_R) + h^{\text{LOAD}}(t_R)
\]

(20)

Unloading Curve—Slope of the unloading curve (see Figure 2) can be expressed as:

\[
\frac{dP}{dt} = -k; \quad t_R + t_C \leq t \leq 2t_R + t_C
\]

(21)

Thus the unloading rate can be defined as:

\[
\frac{dh}{dt} = \left[ \frac{k(2t_R + t_C - t)^{1/2}}{(\alpha_2 \eta)^{1/2}} \right] - \frac{1}{[k(2t_R + t_C - t)]^{1/2}} \frac{k}{2(\alpha_1 E')^{1/2}}
\]

(22)

The solution for unloading portion is given by:

\[
h^{\text{UNLOAD}}(t) = (k)^{1/2} \left( \frac{t_R^{3/2} - (2t_R + t_C - t)^{3/2}}{3} + \frac{(2t_R + t_C - t)^{1/2} - t_R^{1/2}}{(\alpha_1 E')^{1/2}} \right) + h^{\text{CREEP}}(t_R + t_C)
\]

(22)

Eqs. (17), (20) and (23) define the entire displacement-time history of a nanoindentation test of asphalt.
Nanoindentation Test

The nanoindenter device at the University of New Mexico (UNM) nano test laboratory was used for indentation. Figure 7 shows the nanoindentation test setup with the Berkovich indenter tip and sample indenting in AC. In the nanoindentation test, the AC sample was mounted on a polymer substrate and the sample substrate system held by a sample stub. The pendulum in the system was used to adjust the bridge box output for the Berkovich indenter tip. In the previous study by the Tarefder and Faisal (2013a and 2013b), both spherical and Berkovich tips were used on asphalt. However, their study concluded that the Berkovich tips are more suitable than the spherical tips for asphalt binder testing. Spherical tips tend to adhere to the unaged asphalt sample surface at room temperature. As a result, system compliance can be lost during indentation on asphalt. Based on the previous experience, a Berkovich tip was used in the current study. A Berkovich tip consists of three-sided pyramidal Berkovich tip with a semiangle of 65.27°. It has sharp and well-defined (pyramid defined by face angle 65.3°) tip geometry. In this study, a maximum load of 0.52 mN was applied with an unloading rate of 0.02 mN/sec. A sitting load of 0.02 mN was used for all the samples. A creep time of 200 seconds was applied after reaching the maximum load. The viscous effects of the test results are reduced by using a fast unloading rate and applying an extended dwell time. Tarefder and Faisal (2013a), have shown that a dwell time of 100-200 seconds (long) can minimize the viscous effect of asphalt. Based on the previous study by the authors, a loading rate of 0.02 mN/sec, and a dwell time of 200 seconds were chosen for the nanoindentation test of AC in this study (Tarefder and Faisal 2013a). Each AC sample was indented at 100 locations to deal with the variability of nanoindentation results due to material heterogeneity in the asphalt or binder.

Figure 7. Nanoindentation test setup for AC.
Task 4: Load-Displacement Data Analysis

As discussed in the previous sections, the current study used creep response to identify the AC phases, while in the previous study by Faisal et al. (2014, 2015) identified the nanoscale phases of AC were identified with respect to the degree of softness. In those studies following assumptions were made to identify binder, mastic and aggregate phases: for a maximum nanoindentation load of 0.51 mN and a dwell time of 200 s: (1) if the indentation depth remains within 1000 nm, the indented phase is defined as the aggregate phase of AC; (2) if the indentation depth remains between 1000 nm to 3000 nm for AC, the indented phase is defined as the mastic phase of AC; and (3) if the indentation depth is higher than 3000 nm for AC, the indented phase is defined as the binder phase of AC. In the current study, the integral AC phases are identified by adopting the two different approaches. One is related to the degree of softness as mentioned above and secondly with respect to the creep behavior of each phase.

Figure 8. Load-displacement Curves of Nanoindentation Tests on Binder1 and Binder2 Samples.

Nanoindentation on Binder at Bulk State

Figure 8(a) shows the load-displacement curves of nanoindentation tests on bulk state binder sample prepared on glass slides. 20 nanoindentation tests were done on binder sample. Binder1 samples showed a maximum displacement of 7040 nm. As discussed earlier, initially slopes of the unloading curves were used to determine the elastic modulus of the material. However, the unloading curves of the load-displacement response of Binder1 show negligible dissimilar response, it might be due to the asphaltic component distribution in asphalt binder. PG 64-22 binder showed a maximum displacement of 7040 nm, whereas PG 70-22 binder showed a maximum displacement of 4423 nm. As discussed earlier, initially slope of the unloading curves was used to determine the elastic modulus of the material. Figure 8(b) shows PG 70-22 has a higher amount of elastic recovery compared to PG 64-22 binder. However, the unloading curves of the load-displacement response of PG 70-22 show negligible dissimilar response, it might be due to the heterogeneous component distribution in asphalt binder.
The load-displacement curves were further analyzed using SDR analysis method to determine the elastic modulus and the hardness of the material. Table 1 shows the elastic modulus and hardness of asphalt binder, it also incorporates the elastic modulus and hardness of RTFO and PAV aged asphalt binder

Table 1 Elastic Modulus and Hardness of Asphalt Binder for Three Different Aging Conditions.

<table>
<thead>
<tr>
<th>Binder</th>
<th>Elastic Modulus (MPa)</th>
<th>Hardness (kPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Unaged</td>
<td>RTFO</td>
</tr>
<tr>
<td>PG 64-22</td>
<td>3.15±0.27</td>
<td>3.97±0.48</td>
</tr>
<tr>
<td>PG 70-22</td>
<td>3.8±0.43</td>
<td>3.83±0.11</td>
</tr>
</tbody>
</table>

Aging in asphalt binder increases the variability in nanoscale elastic modulus and hardness of the asphalt binder, as it can be found in the increased standard deviation. Previous studies show the asphalt aging increases the asphaltene content in asphalt binder. During nanoindentation, the possibility of hitting the asphaltene fraction of asphalt binder increases, which also increases the stiffness as well as the stiffness variability of the thin film binder. However, due to viscoelasticity in asphalt binder, the analysis requires viscoelastic creep analysis of the material, which is done in the later part of the paper.

**Nanoinindentation on Mastic at Bulk State**

Figure 9(a) shows load-displacement curves on mastic samples using the binder1 sample. Maximum depth was found within 2000 to 3000 nm, which means the inclusion of fines with asphalt binder creates a stiffer sample to indent using the nanoindenter. In the current study, asphalt mastic sample was prepared using a dust to binder ratio of 1.2. Figure 9(b) shows the load-displacement curves of nanoindentation on asphalt mastic sample. The load-displacement curve shows the maximum displacement ranges from 1000 to 3500 nm.

![Load-displacement curves for PG 64-22 Mastic](image1)

![Load-displacement curves for PG 70-22 Mastic](image2)
Figure 9. Load-Displacement Curves of Nanoindentation Tests on PG 64-22 and PG 70-22 Mastic.

Comparative analysis between Figure 8(a) and 8(b) show, the inclusion of fines with the asphalt binder sample to create asphalt mastic creates stiffer material, where indentation depths are lower compared to the maximum depth of nanoindentations on asphalt binder sample. However, comparison of PG 64-22 and PG 70-22 did not show a significant difference, except the indentation response of PG 70-22 mastic samples were followed a range of maximum depth from 1000 nm to 3500 nm.

**Nanoindentation on AC Sample**

Asphalt concrete is a heterogeneous material. In case of nanoindentation, a maximum load of 0.51 and dwell time of 200 sec always showed a maximum load within 9000 nm. Figure 10(a) shows the load-displacement response of PG 64-22 AC sample.

![Load-Displacement Curves of Nanoindentations on PG 64-22 AC Sample](image)

(a) PG 64-22 AC Sample

![Load-Displacement Curves of Nanoindentations on PG 70-22 AC Sample](image)

(b) PG 70-22AC Sample

Figure. 10 Load-Displacement Curves of Nanoindentations on PG 64-22 and PG 70-22 AC Sample.

The load-displacement curve distribution shows, most of the load-displacement curves are distributed in the first 1000 nm region, which means most of the indentations were done on mastic and aggregate phase of asphalt. In the hot mix asphalt about 95% of the material is aggregate and only about five percent is binder, when binder is mixed with aggregate it makes a thin coat of binder around the aggregate and when the sample is cut from the sample it creates a surface mostly consists of aggregate phases with very few places of interface between aggregates, the interface is mostly consists of mastic material and small amount of binder phase. As nanoindentation requires a smooth perfectly level surface for indentation, the polishing process washes away some of some binder thin coats from AC sample. However, the interface remains the only source of finding binder phase in AC. In the study, all the indentation curves
were further analyzed regarding their principal parameters as discussed before to preliminary identification of binder, mastic and aggregate phase of AC. Afterwards, their creep responses were extracted and compared with independent material creep response. According to principal component analysis from 80 valid indentations only nine indentations were found on binder phase of AC, 10 indentations on mastic phase and 63 indentations on the aggregate phase of AC.

**Task 5: Modeling Creep Behavior**

**Asphaltic Phase Identification**

As discussed in the previous sections, the current study used creep response to identify the AC phases, while in the previous study by Faisal et al. (2014, 2015) identified the nanoscale phases of AC were identified with respect to the degree of softness. In those studies following assumptions were made to identify binder, mastic and aggregate phases: for a maximum nanoindentation load of 0.51 mN and a dwell time of 200 s: (1) if the indentation depth remains within 1000 nm, the indented phase is defined as the aggregate phase of AC; (2) if the indentation depth remains between 1000 nm to 3000 nm for AC, the indented phase is defined as the mastic phase of AC; and (3) if the indentation depth is higher than 3000 nm for AC, the indented phase is defined as the binder phase of AC. However, the assumptions did not consider nanoindenter hitting a void space in AC concrete samples, which is a quite common scenario for AC. Nano/Microvoids in AC shows sudden high micro scale displacement of the indenter for a specific load, which invalidates the specific phase identification according to the degree of softness of the material. Therefore, current study identifies those three distinct phases of AC according to the creep response GPL model parameters of the material. However, fitting all the indentation data for three replicated sample with 100 indentations on each sample is a time-consuming process. Therefore, current study uses the principal component analysis using statistical software R initially to identify the soft phases of AC according to maximum depth and elastic modulus analysis. Afterwards, the soft phases of AC are analyzed further to determine the GPL model parameters of Eq (13). The GPL parameters of soft phases are compared with those of bulk states of binder and mastic samples to identify specific phase.
Figure 11. Load-displacement, Creep Response and GPL Model Fitting of Binder Sample.
Binder Phase of Asphalt Concrete

Figure 11(a) shows the binder phase load-displacement curves of asphalt concrete. As discussed before all the binder phase was preliminarily identified using principal component analysis with the help of statistical software R. The maximum displacements of all the indentations were ranged from 2738 nm to 9607 nm. As discussed previously, binder phase of AC is predominantly a viscoelastic material only the creep response/dwell time of the load-displacement curve was further analyzed to validate the phase identification work. Figure 11(b) shows the extracted creep responses of the load-displacement curves. The figure 11(b) shows displacement-time plot of all eight creep responses extracted from nanoindentation data. For creep analysis, it is assumed the load and unloading time negligible compared to creep response time. Therefore, the creep start time and load were designated as zero. Among all the creep response plots only the first plot showed a displacement of 6.8 µm compared to other curves plots average of 2.3 µm. As the first curves maximum displacement is three times of the average, it is deducted as an outlier from the analysis. Figure 11(c) shows average creep response of all the binder phase data of asphalt concrete. The current study uses generalized power law to represent the creep compliance of the material. The generalized power law parameter $D_0$ and $D_1$ can be used to describe the glassy and transition behaviors (Veytskin et al. 2015, Park and Schapery 1999). Table 2 shows the generalized power law parameter values and goodness of the fit.

| Table 2. Creep Model Parameters of Bulk State of Binder and Phase State of Binder. |
|-----------------|-----------------|-----------------|-----------------|-----------------|
| PG Grade | Material | $D_0$ (kPa)$^{-1}$ | $D_1$ (kPa)$^{-1}$ | $n$ | $R^2$ |
|Binder 1 | Binder | 0.03621±0.00591 | 0.001404±0.000043 | 1.552±0.0015 | 0.9999 |
| | Binder Phase of AC | 0.0369±0.01191 | 0.00128±0.000343 | 1.46±0.0058 | 0.9979 |
|Binder 2 | Binder | 0.04391±0.00289 | 0.00234±0.000391 | 1.498±0.05 | 0.9999 |
| | Binder Phase of AC | 0.0442±0.00463 | 0.00251±0.000436 | 1.521±0.1 | 0.9979 |

Veytskin et al. (2015) found if the material loading stays within the linear viscoelastic (LVE) limit of the material, the creep compliance curves follow similar trends compared to any loading scheme. Table 1 also shows a similar pattern of creep response as both $D_0$ and $D_1$ parameter and $n$ value shows similar value. The similar pattern and trend also infer it is possible to identify the binder phase of asphalt concrete using nanoindentation test. To validate the process whether the analysis is analogs to other PG grade binders also, Binder2 samples were analyzed similarly in bulk and phase state and results are shown in Table1. It also renders similar GPL parameters.
Mastic Phase of Asphalt Concrete

A similar technique was used to identify the mastic phase of AC. Fig. 9 shows the comparison of creep curve from phase state of mastic of the AC as well as from the bulk state of mastic sample. Figure 12(a) and 12(b) show the extracted creep response of bulk state of mastic samples as well as the phase state of mastic AC.

![Fig. 9](image1)

![Fig. 12](image2)

(a) Creep Responses of Bulk Mastic Sample  (b) Creep Responses of Mastic Phase of AC

Figure 12. Comparison of Creep Response of Mastic Phase of Binder1 AC Samples.

It can be noticed that bulk state of mastic samples followed the almost similar trend for all indentations, however the due to heterogeneity of AC sample the mastic phase creep response showed small variability in their responses. However, during model fitting, only nine indentations creep responses were used instead of ten. The outlier tenth creep response was not used in the model prediction. Table 3 shows the GPL model parameters for the bulk state of mastic samples and Phase state of mastic AC samples. To evaluate the validity similar calculations were done for Binder 2 samples.

<table>
<thead>
<tr>
<th>PG Grade</th>
<th>Material</th>
<th>$D_0$ (kPa)$^{-1}$</th>
<th>$D_1$ (kPa)$^{-1}$</th>
<th>$n$</th>
<th>$R^2$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Binder 1</td>
<td>Mastic</td>
<td>0.02191±0.00591</td>
<td>0.001404±0.000043</td>
<td>1.055±0.005</td>
<td>0.999</td>
</tr>
<tr>
<td></td>
<td>Mastic Phase of AC</td>
<td>0.0185±0.00632</td>
<td>0.0012579±0.001</td>
<td>1.053±0.02</td>
<td>0.9982</td>
</tr>
<tr>
<td>Binder 2</td>
<td>Mastic</td>
<td>0.01558±0.00339</td>
<td>0.003099±0.00169</td>
<td>1.142±0.014</td>
<td>0.9999</td>
</tr>
<tr>
<td></td>
<td>Mastic Phase of AC</td>
<td>0.01314±0.00185</td>
<td>0.003384±0.00131</td>
<td>1.092±0.048</td>
<td>0.9861</td>
</tr>
</tbody>
</table>

Table 3. GPL Creep Analysis Parameters of Mastic
It can be noticed that independent mastic samples followed the almost similar trend for all indentations, however the due to heterogeneity of AC sample the mastic phase creep response showed small variability in their responses. However, during model fitting, only nine indentations creep responses were used instead of ten. The outlier tenth creep response was not used in the model prediction. Table 3 shows overlapped of GPL model parameters for individual PG grade sample, which also ensures it is possible to identify the mastic phase of AC. Therefore, it is found that the creep response parameters are similar and remains irrespective of the material state in the micro to nano scale, which also satisfies the LVE assumption of the study.

**Aggregate Phase**

Aggregate is strictly an elaso-plastic material and it is possible to identify and indent on the aggregate phase of AC. Elasto-plastic OP analysis has been used in the determination the modulus and hardness of aggregate samples. Previous studies of Tarefder and Faisal show, aging and moisture damage in asphalt concrete does not affect the material parameters of aggregate phase (Faisal et al. 2015). Therefore, current study only confined within the identification of the viscoelastic property change of binder and mastic phase of asphalt concrete.

**Determination of SDR Model Parameters**

Nanoindentation data is used to fit the above Eqs. (14), (17) and (20) and SDR model parameters elastic modulus, $E$; Hardness, $H$; and indentation viscosity, $\eta$ have been determined from the fitted data. Displacement data is fitted first to Eq. (17) to estimate indentation viscosity $\eta$. Next, the unloading data is fitted to Eq. (20), to estimate all the model parameters. The curve fitting of the displacement time curve was done in Matlab using nonlinear least square fitting, with the trust region algorithm.

**Comparing Bulk vs Phase Properties**

As discussed and showed in previous sections, the creep response of the bulk and phase state of asphalt can be used to identify the specific AC phases. However, it will be interesting to find out whether bulk state shows or extracts similar/different results compared to phase state of AC. Viscoelastic SDR model has been used to extract the modulus, hardness and indentation viscosity of the material. To compare bulk state vs phase state of AC, samples were aged using a draft oven for two days and five days. As discussed previously, the behavior of binder and mastic phase of AC is strictly viscoelastic, however, the behavior of aggregate phase is purely elastic (Faisal et al. 2014, 2015, Tarefder and Faisal 2013). SDR model has been adopted to extract the modulus, hardness and indentation modulus of the material.
Figure 13(a) shows bulk binder stiffness of Binder1 sample. Here all three replicated samples are prepared on a glass slide and then twenty nanoindentation tests were done on the sample. Each point on Figure 13(a) represents the average of 20 valid nanoindentations. In the plots, the standard error for each point for 20 indentations is shown. It can be noticed the as the aging time increases the variability among sample increases as well as the standard error among themselves. It might be due to the increase in asphaltene content of asphalt binder, which creates a heterogeneity among the same sample to create the higher amount of variability. Figure 13(b) and 13(c) compare the stiffness of bulk state and phase state of asphalt concrete. Both plots show aggregate samples have the highest stiffness of around 82 MPa and with aging, they did not show any change in the stiffness, which is also expected. The oxidative aging of only five days does not affect the stiffness of bulk as well as the phase state of aggregate samples. However, in both cases stiffness of the material increases, compared to the unaged stiffness of the binder, bulk state binder samples showed 3.81 and 8.27 times higher stiffness for two and five days oxidative aging respectively. Phase state of binder showed 2.38 and 5.71 times increase in stiffness for two and five days draft oven aging. Therefore, phase state of asphalt binder showed
less stiffening due to the oxidative aging of the sample. The reason might be related to void spaces present in asphalt concrete, when asphalt concrete samples are aged in the draft oven, some surface asphalt binder might have rearranged to expose the new binder on the surface. It is well known, AC is a combination of impermeable and permeable pores, test on impermeable pore trapped asphalt binder shows lower stiffness than the fully aged binder. Similarly, aged mastic stiffness is compared with unaged mastic stiffness, results show bulk state stiffness increased by 3.22 and 8.68 times for 2 days and 5 days draft oven respectively. However, the phase state mastic stiffness showed 2.31 and 6.15 times increase for the same amount of aging. In this case, also, phase state of the material showed lower stiffening in the material due to oxidative aging.
Figure 14. Hardness and Indentation Viscosity Comparison between Bulk Binder vs Phase Binder

Figure 14 compares the hardness and indentation viscosity of binder and mastic sample. Figure 14(a) and 14(b) show hardness comparison of bulk state and phase state of binder1 binder and mastic sample. The hardness of the material increases with the increase of aging of the material. Comparing with unaged bulk binder hardness of 60kPa, the aged binder hardness shows 2.71 and 4.3 times higher value for 2 days and 5 days of oxidative aging respectively. Similarly, mastic hardness increased by 1.71 and 4.15 time compared to bulk state mastic hardness. It should be noted, the aged hardening rate of mastic samples were higher than compared to binder sample, and it might be related to the binder absorption of aggregate present in mastic samples. A similar comparison of the hardness of phase state of binder shows an increase of 2.53 and 3.6 times compared to unaged condition for 2 days and 5 days draft oven aging. However, age hardening of phase state of mastic samples are 2.75 and 5.1 times compared unaged state.
Table 4. Bulk State vs Phase State for Different Aging Conditions

<table>
<thead>
<tr>
<th>Material</th>
<th>State</th>
<th>Stiffness Ratio = ( \frac{E_{\text{Bulk}}}{E_{\text{Phase}}} )</th>
<th>Hardness Ratio = ( \frac{H_{\text{Bulk}}}{H_{\text{Phase}}} )</th>
<th>Indentation Viscosity Ratio = ( \frac{\eta_{\text{Bulk}}}{\eta_{\text{Phase}}} )</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Unaged</td>
<td>2 Days</td>
<td>5 Days</td>
</tr>
<tr>
<td>Binder1</td>
<td>Binder</td>
<td>1.02</td>
<td>1.45</td>
<td>1.32</td>
</tr>
<tr>
<td></td>
<td>Mastic</td>
<td>0.94</td>
<td>1.28</td>
<td>1.29</td>
</tr>
<tr>
<td>Binder2</td>
<td>Binder</td>
<td>1.12</td>
<td>1.67</td>
<td>1.54</td>
</tr>
<tr>
<td></td>
<td>Mastic</td>
<td>1.03</td>
<td>1.57</td>
<td>1.64</td>
</tr>
</tbody>
</table>

Fig. 14(c) and 14(d) show the comparison of indentation viscosity. Indentation viscosity is an attractive parameter, as asphalt researchers show there is a strong correlation between asphalt aging and viscosity. However, nanoindentation gives indentation viscosity which also showed an increase with an increase in aging of the material, as shown in Figure 13(c). Indentation viscosity ranged from 30 Pa·s² to 800 Pa·s² for asphalt binder. Here also indentation viscosity is higher in bulk binder compared to binder phase material. In addition, the rate of viscosity change in mastic samples is higher than that of binder samples. Table 4 summarizes the stiffness, hardness, and indentation viscosity comparison between bulk state and phase state of asphalt. Comparing all the stiffness and hardness ratio, it can be observed that both aged bulk state stiffness and hardness show an average of 1.3 times higher than that of phase state stiffness of the material, whereas indentation viscosity ranges from 1.0 times to 1.25 times. To validate, whether bulk state material parameters are always higher that of phase state of AC, binder2 samples were tested comparison of material parameters show they are analogous to the findings of binder1 samples.

**Unaged vs. Aged Modulus and Hardness of Aggregate Phase**

Figure 12(b) shows that the average modulus of the unaged aggregate phase of AC is 105.3 GPa, whereas average modulus of 2 days aging and 5 days aging are 116.7 GPa and 87.43 GPa. From Figure 12(c) it can be seen the average hardness of unaged aggregate phase is 8.6 GPa, whereas that of level 1 aging and level 2 aging are 11.5 GPa and 8.65 MPa. Therefore, two levels of aging did not show any trend increasing/decreasing materials property for the aggregate phase of AC. Ideally, only 2 to 5 years of aging should not affect the modulus and hardness of aggregate. Therefore, statistical analysis has been done with respect to means of each group for an alpha level of 0.05, and the analysis shows a p-value of 0.153 which is greater than 0.05, therefore there is not enough evidence to say that the modulus and hardness of unaged and different level of aging are different in the aggregate phase of AC.
Conclusions

In the study, nanoindentation tests were conducted on the bulk state of asphaltic materials and AC phases. A mathematic modeling technique has been proposed and implemented to identify specific AC phases. Afterwards, $E$, $H$, and viscosity from SDR model have been extracted to make a comparative analysis of 3 different aging conditions. The following conclusions can be made:

1. Nanoindentation test can successfully identify binder, mastic and aggregate phases of AC. To identify the binder and mastic phases the creep parameters of the specific phase is compared with the creep response of bulk state of the respective material. However, results of AC indentations show only 10%-15% indentations are indented on the binder and mastic phases of asphalt concrete, though all the indentations were selected to indent the interface between two aggregates of AC. To identify specific phases GPL model with LVE limit analogy is successfully used in the study.

2. Currently, there is no widely accepted nanoindentation model for visco-elastoplastic materials such as AC. Therefore, this study shows that the SDR model gives satisfactory results on nanoindentation of asphalt concrete. However, more studies are required under different environmental conditions to validate the model for AC.

3. Comparison between different aging conditions shows, aging increases the nanoscale modulus, hardness and indentation viscosity of bulk binder and mastic material. A similar trend is found for binder and mastic phase when they are in phase state AC. However, results show on bulk material aging creates a higher amount of stiffening and hardness, in AC phases the effect of aging is comparatively lower. Nanoindentation was able to capture the high variability of stiffening in binder and mastic phases of aged concrete.

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