Construction and Building Materials 135 (2017) 411–419

Contents lists available at ScienceDirect

Construction and Building Materials

journal homepage: www.elsevier.com/locate/conbuildmat

Investigation of microscale aging behavior of asphalt binders using atomic force microscopy

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HIGHLIGHTS

- Aging affects the microstructures and mechanical behavior of asphalts.
- The percentage of bee structures and surfaceroughness were introduced to quantify microstructures of asphalts.
- The DMT modulus and adhesive force were measured to quantify the micro-mechanical properties of asphalts.
- A Parallel model was applied to determine asphalt composite phase mechanical response.

ARTICLE INFO

Article history:
Received 29 August 2016
Received in revised form 19 November 2016
Accepted 29 December 2016

Keywords:
Asphalt binder
Long term aging
AFM
Microstructures
Composite mechanical properties

ABSTRACT

Atomic force microscopy (AFM) has the capacity to distinguish among different phases at high resolution, and it is widely used in obtaining topography and mechanical property maps for asphalt binders. This study investigated the effects of long-term aging on both SBS-modified asphalt and base asphalt using AFM. In describing the study findings, this paper introduces two new indices used to quantify changes in the microstructures of asphalt, namely, the percentage of elliptical bee structures and surface roughness. In addition, the Derjaguin-Muller-Toporov modulus and adhesive force were measured to quantify the mechanical properties of the micro-phases. Finally, to evaluate the influence of aging on composite phase properties, a parallel model from the field of composite materials was introduced and applied. The results indicate that aging significantly affected the microstructures and mechanical behavior of micro-phases of asphalt binders. Aging also had a significant influence on the microstructures of the asphalt, especially in the bee structures. The percentage of bee structures increased or decreased after aging depending on the asphalt binder type, while surface roughness always decreased. Aging also increased the composite modulus of the micro-phases and decreased the composite adhesion of the micro-phases. These findings are in agreement with the macroscale aging behavior of asphalt binders.

1. Introduction

Asphalt aging during mixing, laying on the road, and exposure in the field represents an important cause of pavement distress, including moisture-induced damage, fatigue damage, and low-temperature cracking. A great deal of research has been conducted on asphalt aging at the macroscale, leading to a consensus that aging contributes to changes in components, chemical structures, and microstructures in asphalt [1,2,29,30]. Therefore, the aging behavior of asphalt binders can be explained by a microscale mechanism, and macroscale properties are determined by microscale structural properties. Furthermore, researchers have found that the thermal and rheological properties of asphalt binders are related to their microstructures [3–5]. These findings suggest that it would be beneficial to increase the study of the aging behavior of asphalt binders at the microscale.

In recent years, many advanced technologies have been developed for evaluating microstructure and microscale mechanical properties of micro-phases, including infrared spectroscopy, atomic force microscopy (AFM), and the nano-indentation test [6–9,26]. However, because of limited resolution, nano-indentation is unsuccessful in quantifying the response of the individual phase [10,11]. In contrast, AFM has been found to be a very suitable tool for use in exploring the microscale mechanical properties of phases in asphalt [16,17].

AFM has allowed researchers to characterize the microstructure of asphalt binders. Asphalt microstructures mainly consist of two phases at the microscale. Fig. 1 shows a typical structure, in which the elliptical bee-phase structure randomly distributes in the

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http://dx.doi.org/10.1016/j.conbuildmat.2016.12.180
0950-0618/

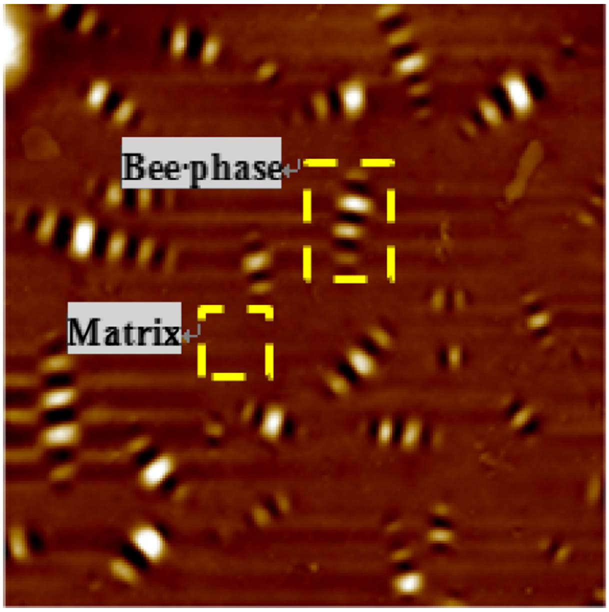

Fig. 1. Microstructures of asphalt binders showing bee phase and matrix phase.

matrix phase [12,13]. According to Hofko's studies, the occurrence of bee structures is connected with asphaltenes [29]. However, other studies have suggested that three or four material phases can be identified in the microstructure [14,15].

Quantitative Nano mechanical (QNM) AFM is a surface force mapping technique with high spatial resolution, which simultaneously measures topography and mechanical property maps of asphalt binders [16,17]. Fischer et al. [18] studied the mechanical properties of asphalt binders' micro-phases using QNM AFM, and the results indicated that the mechanical properties are dependent on temperature. Das et al. [13] investigated the mechanism of bitumen surface aging using the same method, finding that aging the product produced impurities in the matrix phase, which act as promoters in the crystallization process. In addition, the bee structure phase was proved to have a higher modulus and a lower adhesion than the smooth matrix phase [6]. Nahar et al. [18] determined that although the bitumen macroscopically shows similar penetration grades, it possess distinct morphological and nano-mechanical properties.

Aging of asphalt binders in an asphalt mixture is considered to be caused primarily by the oxidation of asphalt [13]. Long-term aging is the main process that occurs during the pavement's service life. Aging affects the microstructures of asphalt binders significantly, according to research by Allen et al. and Wu et al. [19,20,25] Rahman et al. [24] confirmed that aging causes changes in the viscoelastic characteristics of asphalt concrete and binders. Eberhardsteiner et al. proposed a micromechanical model that allows a prediction of the consequences of these microstructural changes on the mechanical behavior of bitumen [30]. Al-Khateeb et al. [21,22] demonstrated that aging affects unmodified asphalt pavements more significantly than modified asphalt pavements. Notably, little is known about the specific relationship between microscale aging behavior and the macroscale properties of asphalt binders.

This paper tested two Styrene Butadiene Styrene (SBS)-modified asphalt binders and two base asphalts in order to determine the effects of long-term aging on the microstructures and mechanical properties of micro-phases. First, to simulate long-term aging, the rolling thin-film oven (RTFO) test and the pressure aging vessel (PAV) tests were conducted. Two new indices were introduced and used to quantify the microstructures of the asphalt binders. The Derjaguin-Muller-Toporov (DMT) modulus and adhesion were measured to quantify the mechanical properties of micro-phases. Furthermore, a parallel model from the field of composite materials was introduced and applied to evaluate the composite properties based on the DMT modulus and adhesion of the micro-phase. Finally, the influences of PAV aging on the composite properties of micro-phases were determined for both SBS-modified bitumen and base asphalt binders.

The goal of this research is to determine the microscale aging behavior of asphalt binders using the QNM AFM technology. The specific objectives are as follows:

1. Quantify the microstructural characteristics of SBS-modified asphalt and base asphalt binders;
2. Investigate the evolution of microstructures and micro-mechanical properties of micro-phases of asphalt binders due to aging;
3. Determine the composite modulus and adhesion based on individual phase properties; and
4. Compare the capacity for aging resistance of SBS-modified asphalt and base asphalt binders in terms of microstructures and micro-mechanical properties of micro-phases.

2. Materials and experimental plan

2.1. Materials and aging method

Two base asphalt binders manufactured by different companies but with the same penetration grade of 60/80 were selected for use in this study. One asphalt binder was provided by Shell Company and is designated as 'base-A.' The other asphalt binder was provided by Zhonghai Company and is designated as 'base-C.'

In addition, SBS polymer was selected and added to these two base asphalts in order to get modified asphalts. The SBS polymer used in this research was supplied by Beijing Company and the basic properties of SBS polymer were presented in Table 1. Modified asphalt was prepared using a high-speed shearer at 4000 rpm for 60 min with 4% by weight of SBS polymer, in which process the blend was kept at 175 °C. The binders with the added polymer are designated as 'Modified-B' and 'Modified-D.'

In order to investigate the effects of long-term aging on the microstructures and mechanical properties of the micro-phases, the RTFO test was combined with the PAV test in the laboratory. During the RTFO procedure, the asphalt was aged at 163 °C for 85 min. Then PAV aging was conducted. During the PAV aging procedure, the asphalt was heated at 100 °C for 20 h (ASTM D6521). The main characteristics of the original and PAV asphalt binders are presented in Table 2.

2.2. Sample preparation

In order to meet the requirements for AFM observation and obtain homogenous samples with proper thickness and no surface contamination, this study developed a molding method, which is recommended for future use [27].

Table 1
The basic properties of SBS polymer.

Basic properties	SBS polymer
Molecular structure	Linear
S/B ratio	30/70
Specific gravity	0.93
Physical form	White granular
Process temperature	175 °C