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Characterization of asphalt materials' moisture susceptibility using multiple methods

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HIGHLIGHTS

- AFM can qualitatively and quantitatively characterize the moisture damage of asphalt.
- AFM and surface energy methods correlate well in evaluation of adhesion of asphalt.
- AFM and nanoindentation can determine the types and degrees of moisture damage.
- A systematic evaluation of asphalt materials' moisture susceptibility was achieved.

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ABSTRACT

Moisture damage seriously deteriorates asphalt pavement's performance and durability and is therefore one of the major problems of asphalt pavements. Currently, researchers pay most attention to macro investigation of moisture damage in asphalt materials. However, moisture damage in asphalt materials can be attributed to the complex interactions between water, asphalt and aggregates occurring at the micro level so that macro tests cannot provide precise insight into this process and its mechanisms. To this end, two promising micromechanical tests, atomic force microscopy (AFM) and nanoindentation, were used in this paper to capture the micromechanical properties of asphalt binder and mixture samples before and after moisture damage. For data comparison, surface energy method and freeze-thaw splitting test were also used. Results show that AFM and nanoindentation are promising methods in investigation of asphalt materials' moisture damage because they can quantitatively determine the loss of asphalt samples' strength and also determine their damage types (adhesion or cohesion failure). Results of surface energy method and freeze-thaw splitting test correlate well with AFM and nanoindentation results in characterization of moisture susceptibility of asphalt binder and mixture samples respectively. It is concluded that these four testing methods can achieve a systematic and accurate evaluation of asphalt materials' moisture resistance.

1. Introduction

Moisture damage of asphalt pavements is a phenomenon that moisture penetrates into the asphalt mixtures and then degrades its mechanical properties, leading to the decline of pavement performance. Moisture damage can induce serious pavement diseases such as cracking and pumping. Hence, it has become a prevalent problem for most transportation departments in the world and generates wide attention in both research and engineering fields.

Moisture damage of asphalt mixtures can be divided into two types, namely adhesive and cohesive failure [1]. Adhesive failure occurs when water deteriorates the adhesion between asphalts and aggregates, leading to a loose state in their interface. Another type of moisture damage is the loss of cohesive strength within asphalt binders. In general, moisture damage of asphalt materials can be attributed to the complex interactions between moisture, asphalt and aggregates which occurring at the micro level. However, due to the lack of proper micro techniques, existing research can seldom provide a comprehensive interpretation of moisture

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damage in asphalt mixtures. Current laboratory testing methods of moisture damage remain at the macro and empirical level. For instance, freeze-thaw splitting test and Immersion Marshall Test are two widely used methods in evaluation of asphalt mixtures' moisture susceptibility in China [2]. The tensile strength ratio of asphalt mixture samples after freeze-thaw treatment is used to evaluate the moisture resistance in freeze-thaw splitting test which is similar with AASHTO T283 test while Immersion Marshall Test uses Marshall stability as the index. These macro tests focus merely on the overall strength of a specimen while showing no promise in revealing the moisture damage mechanism within asphalt mixtures. Hence, it is pretty critical to develop a practical and precise technique to investigate the moisture damage of asphalt materials from a new angle.

In recent years, the emergence of atomic force microscopy (AFM) and nanoindentation in asphalt research filed has provided many senseful achievements. These two micro methods can give precise insight into asphalt materials and consequently have a great potential in research on asphalt materials' moisture damage.

AFM can scan and capture the micro topography images of sample surface which contains the micromorphology information of the selected area [3]. These images are reported to have the potential in qualitative characterization of asphalt binders' micro properties [3–5]. Tarefder found that microstructures on asphalt surface were eroded under wet condition which indicating the adverse effect of moisture [6]. Moreover, micromechanical properties of asphalt binders such as modulus and adhesion force can be calculated through the AFM force-displacement curves of selected sites on asphalt surface [6–11]. Tarefder used AFM with chemically functionalized tips to measure the adhesion force between the surface of modified asphalt binders and the functionalized AFM tips. Results showed that the loss of adhesion force due to moisture was about 20nN for the lime-modified samples and about 50nN for the Kling Beta modified samples [6]. Arifuzzaman utilized AFM to capture the adhesion forces of base and SBS-modified asphalt binders. It was reported that base asphalt binders were more susceptible to moisture damage than SBS-modified asphalt [12]. Hence, AFM can achieve both qualitative and quantitative characterization of asphalt binders' moisture susceptibility.

Nanoindentation is capable to measure the micromechanical properties of multiphase mixtures. Generally, a silicon carbide probe with a radius of several nanometers is used to indent into the sample and the load-displacement data of the tip during loading and unloading process are recorded. Oliver-Pharr method is applied to analyze the load-displacement curves to calculate the hardness and modulus of the sample [13]. With the microscope in nanoindentation instrument, the tip can indent into different phases of samples, for example, aggregate and mastic (a mixture of fines and asphalt binders) phase in asphalt mixtures and then determine their mechanical properties respectively [14,15]. Nanoindentation is quantitative and precise and is therefore promising in investigation of micromechanical behavior and its mechanism of asphalt mixtures. Tarefder applied nanoindentation to capture the mechanical properties of aggregate and mastic phase. Results showed that hardness of aggregate increased after wet conditioning while that of mastic phase decreased [16]. Hossain conducted nanoindentation on dry and wet mastic materials to determine their contact creep compliance to investigate the moisture damage in mastic materials. It was shown that wet materials exhibited less viscoelastic behavior but high stiffness than dry materials due to the loss of asphalt binder covering on the aggregate [17]. Existing research indicate that nanoindentation is a promising quantitative testing method in investigation of asphalt mixtures' moisture damage.

Recently, surface energy method has also gain more and more applications in research of moisture damage of asphalt materials. Surface energy method can theoretically calculate the adhesion work between asphalt and aggregate surfaces without complicated experimental procedures. Generally, the greater the adhesion work is, the better the adhesion between asphalts and aggregates [18]. Researchers use Wilhelmy plate and Universal Sorption Device method to determine the surface energy components of asphalt binders and then calculate its adhesion work with specific kind of aggregate [1,18–20]. The adhesion work data under hydrous and anhydrous conditions are subsequently used in evaluation of asphalt's resistance to moisture damage.

In summary, this article aims to use AFM and nanoindentation to investigate the moisture damage of asphalt materials from a micro perspective. To achieve a systematic characterization, surface energy method and freeze-thaw splitting test are also used. All the data were cross compared to provide a comprehensive understanding of this issue.

1.1. Research contents

One purpose of this study is to investigate the moisture damage of asphalt materials through AFM and nonoindentation test to make up for the faultiness of research at the micro level. The second is to verify the feasibility and accuracy of AFM and nanoindentation in investigation of moisture damage of asphalt materials. The main contents of this paper are as follow:

1. Use AFM to capture the topography images and micromechanical properties (adhesion force and modulus) of Pen30 and Pen70 asphalt binders at different temperatures before and after immersion in water (20 °C, 72 h);
2. Use Sessile Drop Method to determine the surface energy components of Pen30 and Pen70 asphalt and then calculate their adhesion work with basalt aggregate to achieve theoretical characterization of asphalt binders' moisture susceptibility;
3. Measure the freeze-thaw splitting tensile strength of Pen30 and Pen70 asphalt mixture samples to evaluate their moisture resistance at the macro level.
4. Determine the modulus data of aggregate, mastic and interface phases in asphalt mixture samples before and after freeze-thaw treatment by nanoindentation.
5. Analyze and compare the data of these four tests to achieve a systematic evaluation.

2. Background

2.1. AFM

One major application of AFM is to capture the surface micromorphology images of the sample. The micro topography images of asphalt samples captured by AFM have many unique microstructures. These unique structures which were first observed by Loeber et al. have elongated regions with a series of aligned protrusions and depressions [21,22], just like little bees. In early 2000s, the formation of bee structures was attributed to asphaltene aggregation because some researchers reported seeing a corresponding increase in the density of bee-shaped microstructures when investigating different contents of asphaltene-doped asphalt samples [4,23]. However, more and more research indicated that bee structures may be related to the presence of wax and its crystallization [5,24]. Pauli made a hypothesis that the interactions between waxes and the remaining asphalt fractions is responsible for much of the structuring. Because they found bee-shaped structures in the 'pure' maltene phase which contained no asphaltenes, while the de-waxed asphalt fractions did not show any microstructures [25,26]. Generally, though no agreement has been reached, it is certain that the presence of wax and its complex