

Study on the phase behavior of asphalt mastic



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HIGHLIGHTS

- We studied phase behavior of asphalt mastic upon DMA, DSC and Brookfield viscometer.
- The addition of fillers can increase T_g and glass transition activation energy.
- The addition of fillers can improve the temperature sensitivity of asphalt mastic.
- The addition of fillers can promote phase separation occur in modified asphalt.
- Fillers have little impact on the discrete distribution of wax in asphalt.

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ABSTRACT

In order to improve the road performance of asphalt mixture, the phase behavior of asphalt mastic which is the most important component of asphalt mixture was studied. The glass transition temperature (T_g) and glass transition activation energy of asphalt mastic were obtained by the dynamic mechanics analysis (DMA) method, and the effect of fillers on the distribution of wax in asphalt was studied by the differential scanning calorimetry (DSC). The effect of fillers on temperature sensitivity of asphalt mastic was studied through Brookfield viscometer. The phase separation of asphalt mastic and the applicability of time temperature superposition principle (TTSP) on asphalt mastic were studied by van Gurp–Palmen (vGP) plot. The results show that the faster the load frequency is, the higher T_g is, and the addition of fillers can increase T_g and glass transition activation energy. The distribution of wax in asphalt is discrete, and fillers have little impact on it. The addition of fillers can improve the temperature sensitivity of asphalt mastic, and the effect order is as follows: granite > andesite > limestone. The TTSP is effective for virgin asphalt at any temperature because of its homogeneity, but it is not effective for modified asphalt when the temperature is higher than 45 °C because of its phase separation, and the addition of fillers can promote phase separation occur in modified asphalt.

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1. Introduction

Asphalt mixture is used widely in the pavement construction, and the asphalt mastic is the most important one in its three levels distributed system, which has significant impact on the road performance and life of the asphalt mixture. Asphalt filler mastic consists of asphalt and mineral powder, usually the modulus of the powder is much greater than the asphalt, so the mineral powder can be regarded as the solid filler [1,2]. “Phase” means a physically distinctive form of a substance, such as the solid, liquid, and gaseous states of ordinary matter—also referred to as a “macroscopic state”. The adding of the filler can change the phase structure of asphalt, especially for the modified asphalt, the complex interaction among the asphalt, modifier and fillers make the phase structures more complex. The phase behavior of asphalt mastic is mainly

divided into phase transition and phase separation. “Phase transition” is the transformation of a thermodynamic system from one phase to another. “Phase separation” means different components in material show remarkable difference.

Asphalt mastic can exhibit various features including glass brittle solid, elastic rubber or viscous fluid with different measurement temperatures and time scales (Fig. 1), which is directly related to road performance of asphalt mixture. In the glassy state region, heat energy is not sufficient to overcome the barrier of the polymer molecules rotational and translational movement, and the both ends of polymer molecular are basically frozen in the fixed position. In the glass–rubber transition region, polymer started the short-range diffusion movement, and chain segments can jump from a certain location to another location, and the polymer changes from brittle glass into a rubber with rebound deformation. In the rubber platform area, though the glass transition results from short-range diffusion movement of polymer chain segment is caused, the entire molecular translational motion and the

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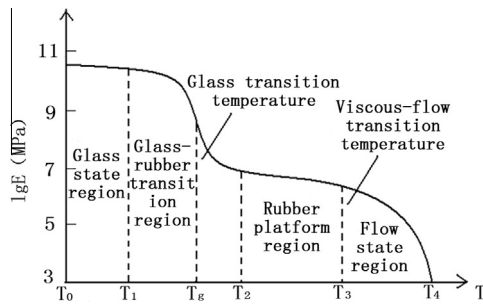


Fig. 1. The relationship between modulus and temperature for asphalt mastic.

remote cooperative movement of the molecular chain are still limited. In the flow area, when the temperature continues to rise, the polymer will become viscous liquids if no chemical change takes place [3]. The phase changes of asphalt mastic studied in this paper including the glass transition, viscous flow state transition and the influence of wax crystallization.

The methods describing the phase separation of blend polymer mainly include morphology, solid physical property and thermodynamics. Morphology is mainly used to evaluate the compatibility of the blending system, whose test methods mainly include microscopy (optical microscopy, phase-contrast microscope, electron microscope) and scattering (light scattering, neutron scattering, X-ray scattering and laser scattering). We can see the presence of two phases and the connectivity in the blending system directly by the microscopy method. Optical microscope and phase-contrast microscope method requires there is a difference between the two phases as an index, and the different preferably come from transparency or color. Electron microscopy mainly includes transmission electron microscopy (TEM) and scanning electron microscopy (SEM), which also take the differences as indexes. The TEM is only can be used to study the morphology of blends, and we need other test methods to study the phase structure and phase dynamics [4–6]. The scattering method is very sensitive to the density and concentration fluctuations in polymer matrix in a microscopic scale [7].

Solid physical property is mainly used to study the compatibility of blends, whose test methods mainly include differential scanning calorimetry (DSC), the dilatometer method, dynamic mechanical analysis (DMA) and rheological method. And the glass transition temperature (T_g) of the blends is commonly taken as a compatibility index. There is a single T_g for miscible polymer blends, which is between the two blending components, and its change trends is similar with the blending component. In the critical solubility condition, the change will be widened. If it is a limited blend, two spaced transformations will appear. The method using T_g to determine compatibility of the polymer blends is applicable only to the situation that the T_g difference of the two components is less than 20 K [8,9].

Kriz et al. studied the glass transition and phase stability of asphalt binders by modulated differential scanning calorimetry and also via dynamic mechanical analysis. A certain analogy between the glass transition of amorphous polymers and asphalts is suggested. They found that the overall transition is very broad on the temperature scale. It was suggested that phase incompatibility may exist in asphalts; however, the phase separation is observable after long-term isothermal conditioning at a temperature within the glass transition range [10]. Wu et al. studied the flammability and rheological behavior of asphalt modified by mixed flame retardants by using differential scanning calorimetry and dynamic shear rheometer, respectively. They found that the inclusion of 6 wt% mixed flame retardants improves the flame retardancy significantly by consuming the released heat of asphalt in a

temperature range of 300–500 °C. The close values of the activation energy obtained by fitting Arrhenius model demonstrate flame retardant modified asphalt exhibits similar thermal susceptibility to base asphalt in the temperature range of 30–100 °C [11]. Michon et al. studied the amorphous and crystalline phases in asphalt by using low-temperature solid-state carbon-13 cross polarization with magic-angle spinning (CP/MAS) nuclear magnetic resonance (NMR) and DSC techniques. They found that the NMR mass percent of the crystalline methylene carbons correlates linearly with the mass percent of crystalline wax in asphalts measured using DSC. And 1% or less of aliphatic carbons in the crystalline phase has little effect on the fracture temperature. For these asphalts, the fracture temperature depends mainly on the initial amount of mobile aliphatic carbons in the amorphous phase at 23 °C. For asphalts containing 1% or more of crystalline aliphatic carbons, the fracture temperature increases with increasing crystalline methylene carbon content [12]. Guern et al. found that the modification by polyphosphoric acid (PPA) leads to an increase in asphaltene content and a more dispersed asphaltene structure than that found in pure bitumen by using size exclusion chromatography under “high-speed” conditions (HS-SEC) combined with IATROSCAN chromatography (coupling between a thin-layer liquid chromatography on a silica gel and a flame ionization detector). And the asphaltene association does not systematically depend on quantity alone, as its chemical type also enters into play [13]. Elseifi et al. demonstrated that an inverse correlation exists between binder ductility at intermediate temperatures and failure strain at low temperatures using high pressure gel permeation chromatography (HP-GPC), DSC and DMA. DSC measurements showed that an increase in crystalline fractions may have a negative impact on the binder stiffness at low temperatures [14]. Kriz successfully identified binders that are sensitive to time hardening at low temperatures by using modulated differential scanning calorimetry (MDSC). In such binders, several thermodynamic and morphology parameters are strongly time-dependent, suggesting that the viscoelastic properties change with time [15]. García-Morales et al. deals with the viscous properties of recycled-polymer modified bitumens (PMBs) in a wide range of temperatures. They conclude that the viscous properties of bitumen, at high temperature, are improved by adding recycled ethylene–vinyl acetate (EVA) copolymer in amounts that depend on bitumen penetration grade. Moreover, significant microstructural changes, related to the development of a polymer-rich phase, tend to occur in the bitumen as polymer concentration increased. These changes in microstructure have a significant influence on the flow behavior of the binder and on its in-service performance [16].

As can be seen, the test methods studying asphalt phase structure are usually DSC and DMA, in addition, NMR, Fourier transform infrared spectroscopy (FTIR) and optical microscopy is sometimes used to explain related phenomena from different angles. There is much research on the polymer modified asphalt or the influence of environmental factors (such as aging etc.) on the asphalt, but little on the effect of inorganic filler on asphalt phase structure.

2. Materials and test methods

2.1. Experimental materials

The raw materials used in this paper mainly include asphalt with the penetration grade 90, asphalt modified by styrene butadiene styrene (SBS) block copolymer and three kinds of mineral fillers, whose technical data are shown in Table 1. Fifteen kinds of virgin asphalt mastics were obtained by blending the virgin asphalt and fillers, and the volume fractions of fillers in each kind of filler mastic are 10%, 20%, 30%, 40%, 50%. Three kinds of modified asphalt mastics were obtained by blending the modified asphalt and fillers, and the volume fractions are 50% of mastic. The stirring time is 15 min at 1000 rpm speed.

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