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Study on the Rheological Properties of Fly Ash Modified Asphalt Mastics

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Abstract: Fly ash is one of the industrial waste residues with significant emissions in China, and its rational utilization has important economic significance and environmental value. Due to the similarity in properties between fly ash and limestone mineral powder, it is possible to replace the mineral powder filler in asphalt concrete with fly ash. This article explored the feasibility of replacing the mineral powder with fly ash in an asphalt mixture through the study of fly-ash-modified asphalt mastic. Firstly, the microstructures and elemental compositions of fly ash and mineral powder were studied using scanning electron microscopy (SEM) and X-ray diffraction (XRD) tests. Then, the rheological properties of asphalt mastics with different fillers were studied using dynamic shear rheological (DSR) tests. The results show that when the stress level was 3.2 kPa, the change in the J_{nr} value was different from that at 0.1 kPa, indicating that after increasing the stress level, the J_{nr} of fly ash asphalt mastic was smaller, and fly ash can improve the high-temperature creep performance of asphalt mastic. Replacing mineral powder with fly ash can improve the high-temperature rheological properties of asphalt mastic, but this damages the elastic and crack resistance properties of the asphalt mastic. In practical applications, partial substitution of mineral powder can be considered for the preparation of an asphalt mixture.

Keywords: fly ash; mineral powder; asphalt mastic; rheological properties

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1. Introduction

Fly ash is the main solid waste discharged from coal-fired power plants. In China, the annual emission of fly ash is more than 200 million tons. How to comprehensively utilize fly ash to turn waste into a resource and turn harm into benefit has become an important technical and economic policy in China's economic construction. At present, limestone powder is mostly used as an asphalt mixture filler. In the context of the high price of limestone powder, it is of great significance to select high-quality limestone powder substitutes to alleviate the pressure on the raw material supply and improve the quality of highway engineering construction. Fly ash has similar physical properties to limestone mineral powder. If fly ash can partially or completely replace limestone powder, this can produce huge economic benefits.

Fly ash is a product of coal combustion. During the combustion process, coal particles become spherical particles under the influence of surface tension, and the carbon component in them generates CO_2 and escapes, forming loose and porous spherical particles [1–3]. In aquatic environments, fly ash can react with alkalis to form materials with cementitious

properties, so it was widely used [4]. Some scholars have found that the volcanic ash effect of fly ash is related to the amorphous phase of glass in the component. The geometric characteristics and pore structure of fly ash have made it widely used in road construction materials, wastewater treatment, and soil improvement [5–9].

Mistry et al. through experiments found that an asphalt mixture using fly ash as the filler had higher Marshall stability values and a lower optimal asphalt content [10]. Xie et al. found that the surface of fly ash used as a filler can improve the indirect tensile strength and tensile strength ratio of asphalt mixtures and increase the fatigue life of asphalt pavement [11–13]. Suched et al. found that fly ash can improve the strength, stiffness, and anti-stripping properties of asphalt mixtures [14]. Atakan et al. found that fly ash showed great potential as a filler for self-repairing asphalt concrete, as it can improve the self-repairing and mechanical properties of asphalt concrete [15]. Compared with asphalt mixtures prepared with mineral powder, within a reasonable dosage range, the maximum densities and bulk densities of asphalt mixtures gradually decrease with an increase in the fly ash dosage. The porosity and frost resistance of these mixtures meet the requirements [16,17]. Raja Mistry et al. found that an asphalt mixture mixed with fly ash exhibited superior Marshall stability and volume characteristics at a relatively low optimal asphalt dosage [18]. In addition, treated fly ash can be used to synthesize zeolite, which can then be used in warm-mix asphalt mixtures [19]. Fly ash can replace the mineral powder filler in porous asphalt mixtures, improve the durability of porous asphalt mixtures in drainage roads, and reduce asphalt failure and damage [2].

Some scholars have extensively studied the impact of fly ash on the macroscopic properties of asphalt mixtures, while others have measured the conventional indicators of modified asphalt mastic. This project studied the rheological properties of fly ash asphalt mastic and explored the feasibility of replacing mineral powder in an asphalt mixture with fly ash. Firstly, the microstructures and elemental compositions of fly ash and mineral powder were studied using scanning electron microscopy (SEM) and X-ray diffraction (XRD) tests. Then, the high-temperature rheological properties of asphalt mastics with different fillers were studied using dynamic shear rheological (DSR) testing.

2. Materials and Methods

2.1. Raw Materials

2.1.1. Base Asphalt

Donghai brand 70# A-grade road petroleum asphalt produced by Sinopec was used in this study. The technical indicators are shown in Table 1.

Table 1. Technical indicators of asphalt binder.

Test Item	Requirement	Test Result	Test Method
Penetration (25 °C; 5 s; 100 g), 0.1 mm	60–80	67	T 0604-2011
Softening point (R&B), °C	≥46	48	T 0606-2011
60 °C dynamic viscosity, Pa. s	≥180	231	T 0620-2000
Ductility (10 °C), cm	≥15	24	T 0605-2011
After TFOT	**Requirement**	**Test Result**	**Test Method**
Mass change, %	±0.8	−0.059	T 0609-2011
Residual penetration ratio, %	≥61	64.5	T 0604-2011
Residual ductility (10 °C), cm	≥6	6.4	T 0605-2011

2.1.2. Fly Ash

Two types of fly ash were selected for this study. Fly ash 1# was a first-level fly ash produced by Shanxi Shuozhou Shentou (Shuozhou, China) No. 2 Power Plant, and fly ash 2# was a second-level fly ash provided by Yuncheng Zhengyang Fly Ash Compony (Yuncheng, China). The mineral powder selected was produced by Yuanzhen Building

Materials Compony (Loufan, China). The appearances of the two types of fly ash are shown in Figure 1.

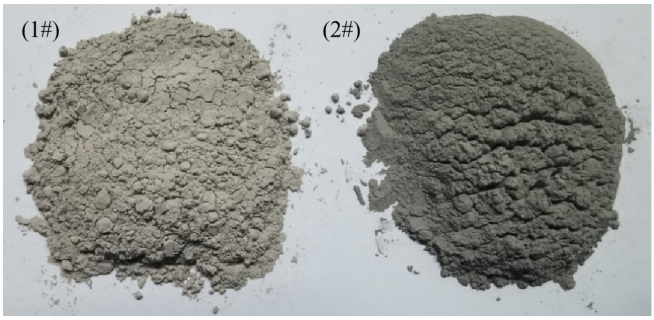

Figure 1. Appearances of two types of fly ash (fly ash 1#; fly ash 2#).

2.1.3. Mineral Powder

The mineral powder was limestone mineral powder, and the test results are shown in Table 2.

Table 2. Test results of mineral powder and fly ash.

Test Item	Mineral Powder	Fly Ash 1#	Fly Ash 2#
Apparent density (g/cm^3)	2.785	2.373	2.416
Hydrophilicity coefficient	0.687	0.612	0.633
Stability	No discoloration	No discoloration	No discoloration

In Table 2, it can be seen that the density of fly ash is lower than that of the selected mineral powder. Compared with mineral powder, as a lightweight filler, fly ash was not easy to settle and segregate in the process of asphalt mastic mixing and asphalt mixture construction and maintained the uniformity of the mastic. The hydrophilicity coefficients of fly ash 1# and fly ash 2# are relatively small and may contain more glass bodies, while the selected limestone mineral powder has a slightly higher hydrophilicity coefficient.

2.2. Composition and Preparation of Asphalt Mastic

The effect of the filler–bitumen ratio on asphalt mastic is very significant. In this project, it was 1.2. The reason for this was that the filler–bitumen ratio for an ordinary asphalt mixture is usually in the range of 0.8–1.6, and the typical median value was selected to be representative. The filler was produced by different manufacturers, and its water content was different due to transportation and environmental conditions. In order to remove the water content and reduce the impact on the asphalt mastic, the filler was dried in an oven. The drying temperature was set to 105 °C, and the duration was 2–4 h. Because this was a laboratory project, a small mixing appliance with an adjustable speed device was adopted. During the mixing process, an oil bath was used to heat the asphalt to 160 °C. In order to prevent the agglomeration of the filler, it was added several times. During the mixing process, the rotation speed was adjusted according to the actual situation to prevent the asphalt mastic from splashing until it was uniform.

2.3. Test Methods

2.3.1. Scanning Electron Microscope (SEM)

The microscopic appearance of a material can provide researchers with an intuitive impression, which greatly helps the analysis of the material. In this project, an SEM device model was used. In order to obtain better imaging results, a variety of fillers were sprayed with gold. During the sample preparation process, a conductive double-sided adhesive was first bonded to the sample dish, and a small amount of filler was bonded to the double-sided

adhesive. Then, the filler particles on the surface were blown off with a blowing earball. Finally, the sample dish was placed into a special appliance to complete the gold spraying. The prepared samples were tested with SEM equipment using different magnifications, and the pictures presented in this paper have a 500 times magnification.

2.3.2. X-ray Diffraction Test (XRD)

X-ray diffraction analysis (XRD) is the most commonly used method for studying the internal structures of materials and can obtain data on material composition, structure, and morphology.

2.3.3. Dynamic Shear Rheology (DSR) Test

According to the relevant requirements of asphalt rheological properties testing (T0628-2011, JTGE20-2011), rheological properties tests were conducted on various asphalt mastics, including temperature scanning (TS), frequency scanning (FS), and multi-stress creep recovery tests (MSCR).

3. Results and Discussion

3.1. SEM

China is rich in coal resources, and the structure and physicochemical properties of fly ash vary from place to place. The current fly ash standards are mainly formulated based on the needs of cement concrete and may not be applicable to the requirements for fillers in asphalt mixtures. Therefore, it is necessary to compare the physical properties of fillers and pay attention to the impacts of the mineral properties and pore structures of fillers on the performance of mastics. For this purpose, relevant micro-inspection tests were conducted to clarify the differences in the performances of fly ash and mineral powder. The SEM results for fly ash and mineral powder are shown in Figure 2.

The shape, texture structure, and angularity of the filler have significant effects on the performance of the asphalt mastic. In Figure 2, it can be seen that at the same magnification of 500×, the mineral powder presents more cubic shapes with more obvious surface angularity, while the particles of fly ash are more spherical, whether it is first-level or second-level fly ash. The particles of mineral powder are larger, the surfaces of the particles are relatively smooth and dense, and there are no obvious pores. Compared with the mineral powder particles, the fly ash particles are relatively small, the surfaces are fluffy, and some particles have pores. Fly ash with its fluffy surface adsorbs asphalt more easily than the smooth and dense surface of mineral powder. Different surface characteristics can affect the performance of asphalt mastic. Through the microscopic experiments and performance tests on the above fillers, it can be seen that compared with mineral powder, fly ash has the characteristics of lightweight, a fine particle size, a large specific surface area, and strong activity, which is conducive to the high-temperature stability of an asphalt mastic system.

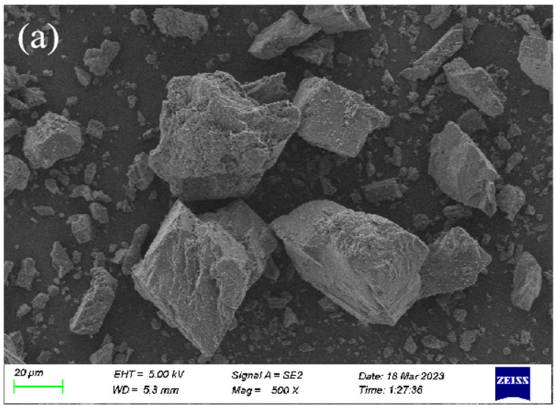

Figure 2. *Cont.*

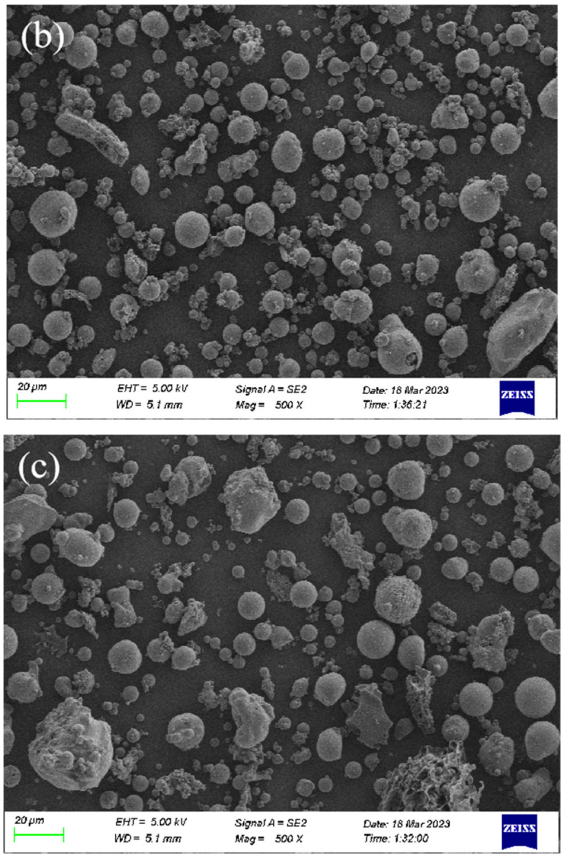

Figure 2. SEM images of various fillers (500×). (**a**) Mineral powder; (**b**) fly ash 1#; (**c**) fly ash 2#.

3.2. XRD

As a dispersion system, asphalt mastic may exhibit a volcanic ash effect similar to that of fly ash in cement concrete. The stable structure of the crystals in fly ash is not conducive to interaction between the filler and the interface, while the strong activity of the glass bodies is conducive to interface adhesion. Therefore, different fillers such as fly ash and mineral powder were tested for their crystal structures and chemical compositions, and the effects of these factors on the performance of asphalt mastics were analyzed. The results of the X-ray diffraction (XRD) tests are shown in Figure 3.

In Figure 3a–c, it can be seen that the XRD test curves of mineral powder and fly ash have significant differences. Mineral powder is made of a limestone material mainly composed of calcium carbonate. Calcite is the main crystal form of calcium carbonate, and the diffraction angles of its standard spectrum are 23.08°, 29.46°, 35.96°, 39.42°, 43.16°, 47.64°, and 48.58°. Quartz and mullite are the main crystalline phases in fly ash, and the components are SiO_2 and silicon aluminate. Wide diffraction peaks appear in the 16–35° region, indicating the presence of glass bodies in fly ash. As amorphous phases with lattice defects, glass bodies have strong chemical activity. The crystal phase of mineral powder is calcite, its composition is $CaCO_3$, and there is no active functional group on its surface, which makes fly ash have higher activity than mineral powder.

3.3. Rheological Performance Results of Asphalt Mastic

3.3.1. Temperature Sweep Test (TS)

In Figure 4, it can be seen that the complex shear moduli (G*s) of the two types of fly ash asphalt mastic are relatively close, and both are higher than that of the mineral powder asphalt mastic. The use of fly ash as a filler enhanced the high-temperature stability of the asphalt mastic. As the temperature increases, the G*s of various asphalt mastics show a downward trend, indicating that the main properties of the asphalt mastics are the same as those of asphalt and have temperature sensitivity.

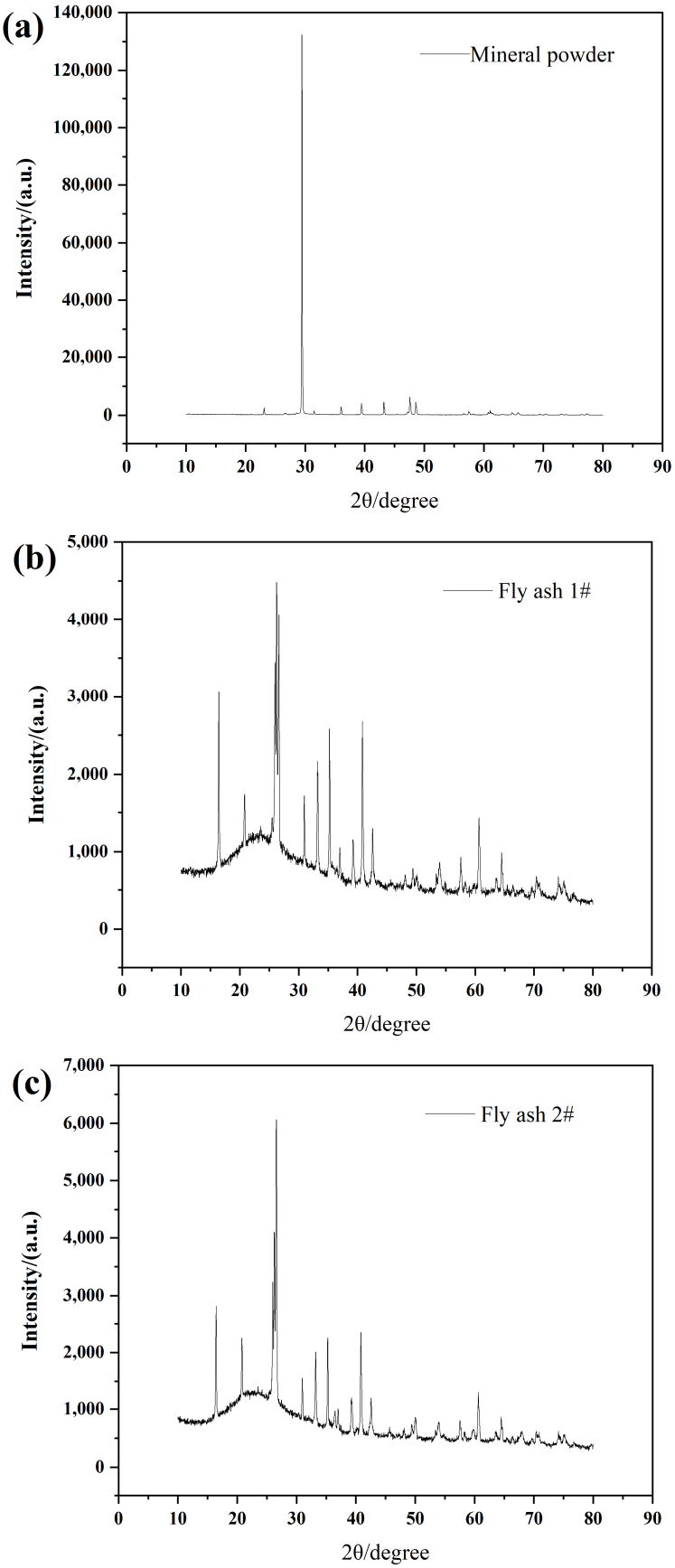

Figure 3. X-ray diffraction test results of fly ash and mineral powder. (**a**) Mineral powder; (**b**) fly ash 1#; (**c**) fly ash 2#.

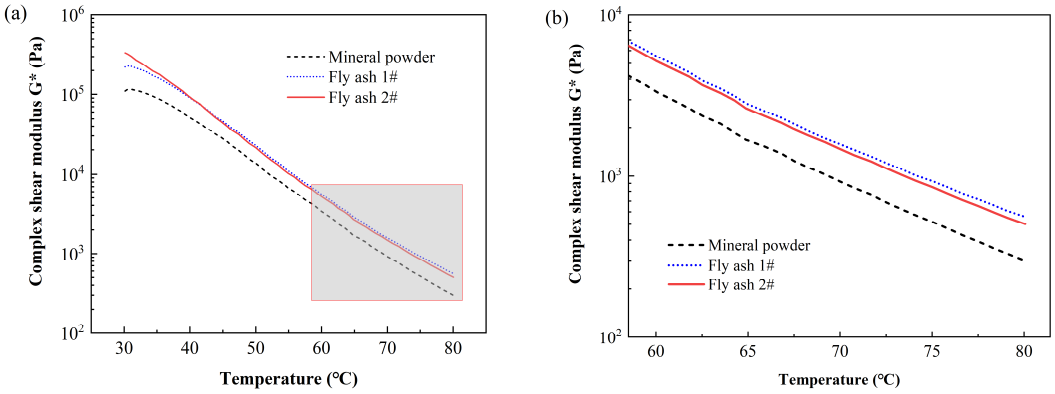

Figure 4. Complex shear moduli changes of various asphalt mastics: (**a**) full image; (**b**) partial enlarged view.

In DSR experiments, the phase angle is displayed as the time lag of strain relative to stress generation in the controlled stress mode. The larger the phase angle, the higher the viscous component of the asphalt, and the longer the time interval between deformations after being subjected to external forces. As shown in Figure 5, within the test temperature range of 30–80 °C, as the temperature increases, the phase angles of the various asphalt mastics show an increasing trend, with similar curves and values.

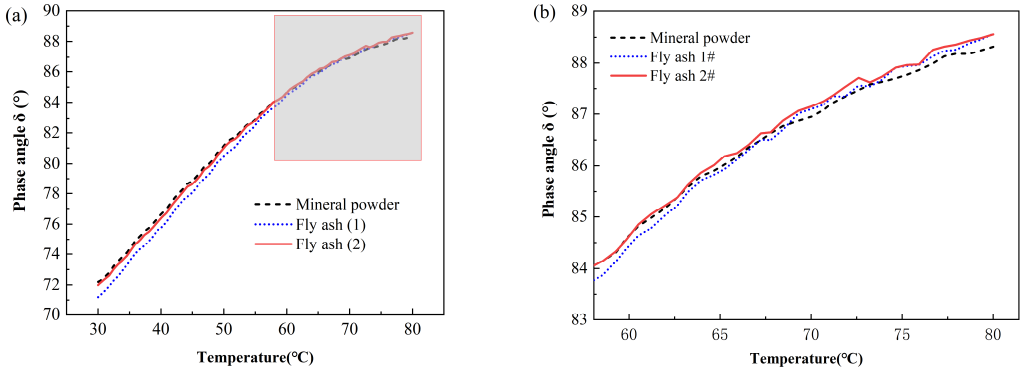

Figure 5. Phase angle changes of various asphalt mastics: (**a**) full image; (**b**) partial enlarged view.

3.3.2. Multi-Stress Creep Recovery (MSCR) Test

In order to evaluate and compare the stress recovery performances of different reinforced modified asphalts, this study conducted MSCR tests under stress conditions of 0.1 and 3.2 kPa at a test temperature of 58 °C. The experimental results are shown in Figure 6 and Table 3. In Figure 6, it can be observed that when the stress was at a high stress level of 3.2 kPa, the strain value was generally 1–2 orders of magnitude higher than that under the 0.1 kPa condition, indicating that asphalt materials are more prone to permanent deformation and irreversible creep shear failure under larger loads.

In Table 3, the deformation recovery rate (R) and irrecoverable compliance (J_{nr}) values of various asphalts are given. Asphalt is a temperature-sensitive material that exhibits more viscosity under high-temperature conditions, gradually losing its elastic properties, and decreasing its creep recovery ability. In Table 3, it can be seen that the creep properties of the various modified asphalt mastics are basically similar. By calculating the R and J_{nr} of each asphalt mastic sample, it was found that all types of asphalt mastics had a certain recovery ability. When the stress level was 0.1 kPa, the R and J_{nr} of fly ash 1# and fly ash 2# asphalt mastics were basically similar, while the R of the mineral powder asphalt mastic was higher, indicating an increase in the deformation recovery rate. The J_{nr} values of the various asphalt mastics varied little. The results indicate that at low stress levels, the deformation recovery rate of the mineral powder asphalt mastic was better, and the J_{nr} was

the smallest. When the stress level was 3.2 kPa, the change in the R value was basically consistent with the change obtained at 0.1 kPa. The change in the J_{nr} value was different from that at 0.1 kPa, indicating that after increasing the stress level, the J_{nr} of the fly ash asphalt mastic was smaller, indicating that fly ash can improve the high-temperature creep performance of asphalt.

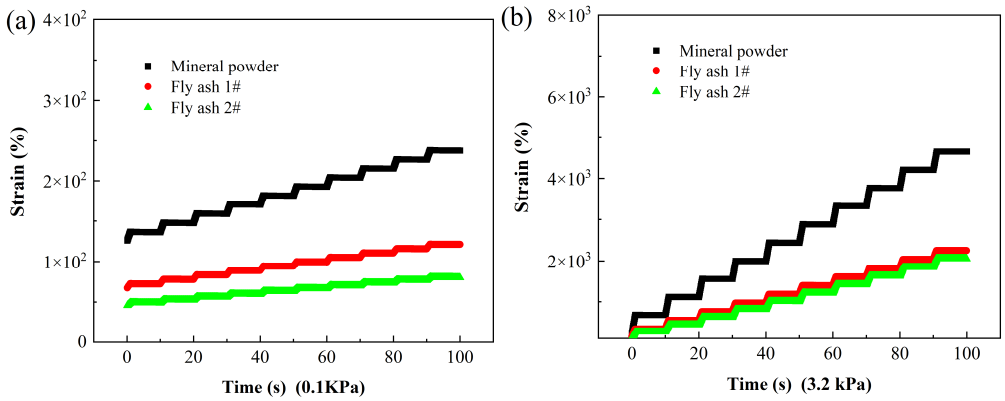

Figure 6. MSCR curves of various asphalt mastics: (**a**) 0.1 Kpa (**b**) 3.2 Kpa.

Table 3. Calculation results of deformation recovery rate (R) and irrecoverable compliance (J_{nr}).

Filler Type	0.1 kPa		3.2 kPa	
	R/%	J_{nr}/kPa^{-1}	R/%	J_{nr}/kPa^{-1}
Mineral powder	11.66	0.63	0.67	1.21
Fly ash 1#	5.12	0.79	0.50	0.99
Fly ash 2#	4.85	0.70	0.49	0.89

3.3.3. Frequency Scan (FS)

The complex shear modulus G* determines the permanent deformation resistance of a material, while the high-temperature deformation and resilience of an asphalt binder are determined with the phase angle. In Figure 7, it can be seen that the G*s of the various asphalt mastics increase from low frequencies to high frequencies, and the basic trend of change is similar, showing a gradually increasing trend. Moreover, the overall numerical differences are not significant, and there is no change in magnitude. The G*s of the fly ash asphalt mastics are slightly higher than that of the mineral powder asphalt mastic, and the high-temperature performance is improved.

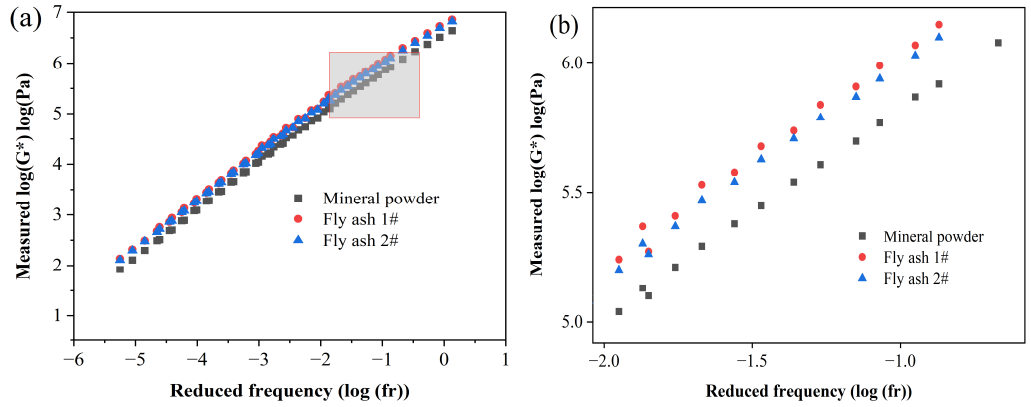

Figure 7. (**a**) Complex shear moduli main curves; (**b**) partial enlarged view.

In terms of phase angles, it can be seen in Figure 8 that the phase angles show a trend of first slightly increasing and then decreasing, and the initial phase angles of the

various asphalt mastics vary greatly. This may be due to some unevenness in the samples during the initial stage of the test. The frequencies decrease from low frequencies to high frequencies, and the phase angles show a decreasing trend, reflecting a decrease in the viscosities and an increase in the elasticities of the various types of asphalt. The phase angle of the mineral powder asphalt mastic is higher than those of the fly ash asphalt mastics, indicating that mineral powder asphalt mastic has good elasticity and low-temperature crack resistance.

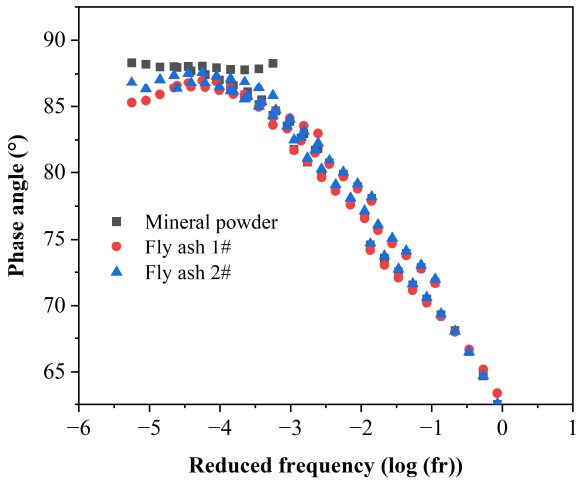

Figure 8. Main curves of phase angles.

3.4. The Comparison of Asphalt Mastics' Characteristics

In Table 4, the properties of the asphalt mastics prepared with different fillers are compared.

Table 4. The comparison of asphalt mastics' characteristics.

Filler Type	Microscopic Characteristics		Rheological Properties		
	SEM	XRD	TS	MSCR	FS
Mineral powder	Cube-shaped, smooth, and dense surface	Main crystal form: calcite	The values of G* of mineral powder were slightly lower	At 0.1 kPa, the R value was higher, indicating better deformation recovery rate	The phase angle was higher, indicating good elasticity and low-temperature crack resistance
Fly ash 1#	Spherical and fluffy surface	Main crystal forms: quartz and mullite Presence of glass bodies	The values of G* and phase angles were similar	At 3.2 kPa, the J_{nr} values were lower, indicating better high-temperature creep performance	The G* values were slightly higher, indicating better high-temperature performance
Fly ash 2#					

4. Conclusions

(1) Mineral powder particles presented more cubic shapes with more obvious surface angularity, while fly ash particles were close to spherical. The particle size of mineral powder was larger, and its surface was smooth and dense. Compared with mineral powder, the particle size of fly ash was relatively small, and its surface was fluffy. Some particles had pores. The fluffy surface of fly ash adsorbed asphalt more easily than the smooth and dense surface of mineral powder, forming an interlocking interface structure with the asphalt, thereby forming a stronger interaction ability with the asphalt.

(2) The XRD test results of fly ash show that a wide diffraction peak appeared in the range of 16–35°, indicating the presence of glass bodies in fly ash. As an amorphous phase with lattice defects, the glass bodies had strong chemical activity, which made fly ash more active than mineral powder.
(3) Compared with mineral powder, fly ash as a filler improved the high-temperature stability of asphalt mastic, but the improvement was not significant. The phase angle of the mineral powder asphalt mastic was higher than that of the fly ash asphalt mastic, indicating that fly ash had a negative impact on the elastic properties and low-temperature crack resistance of the asphalt mastic.

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