Additional Graduation Work

Effect of fiber length and dosage on the performance of reed fiber-modified bitumen and mortar

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Content

Chapter 1: Introduction

Bitumen is the material most used for road pavement through all over the world. The increase in traffic due to economic development has resulted in the performance of original bitumen being unable to meet the requirement of daily damages such as fatigue cracks. When the virgin bitumen cannot meet the requirements of climate, traffic, and pavement structure, modification is considered one of the most effective means to improve the performance of asphalt[1]. In addition to the well-known method of polymers, as interest in eco-friendly construction has increased over time, more recently, due to improved pavement performance and reduced material costs, it is becoming more and more common to use natural or regenerated fibers as stabilizing additives or reinforcing phases in asphalt binders and mixtures[1-3].

1.1 Fiber as modifier

Fiber has been used in road construction since the 1900s when the Warren Brothers Company of Boston, MA, used asbestos fibers in sheet asphalt[4]. At that time asphalt construction continued in the cold-laid method and asbestos fibers were used to prevent aggregate segregation during construction[4]. In the 1950s, the use of fibers in hot mix asphalt (HMA) was evaluated for the first time[4]. And since then more and more types of fibers are proven to improve asphalt performance[5-7].

Previous research has shown that the presence of glass and metal fibers in bitumen allows to reduce the thermal susceptibility of bitumen and can increase the rutting resistance of the mixture[7, 8]. Su, Kai and Yoshitaka Hachiya add a cellulose fiber to recycled asphalt concrete as a supplement and the results show that the addition of fibers improves the rutting resistance, moisture resistance, crack resistance, and durability of RAC[9]. In addition, because of its excellent mechanical properties and inherent compatibility with asphalt, adding carbon fibers to asphalt can increase the strength of the material and improve its ductility of the material[10]. Putman and Amirkhanian[11]compared the performance of SMA mixtures containing waste tire fibers and carpet fibers and showed that these materials significantly improved the toughness of the blends, while not helping with permanent deformation.

To minimize the environmental influence, sustainable bio-based materials continue to receive global attention. Considered the most common, prominent, and renewable source of raw materials and ubiquitous, a large number of plant materials are being extensively studied[12-14]. And organic fiber, like various stalk fibers, motivates scholars to investigate the effect and application because of its sustainable, and biodegradable, and superior specific properties. Zhexia Li's group used cotton stalk powder to improve the tenacity, temperature sensitivity, and rheological properties of asphalt[15]. Chen mixed corn stalk fiber with asphalt binder to verify the effect on the performance of asphalt. The results showed that the corn stover had better mixing uniformity and the addition of corn fiber increased the deformation resistance and recovery performance of asphalt[16]. Further, Chen also analyzed the possibility of the application of corn stalk fibers in SMA mixtures, which embodies the value and potential of such materials in pavement[17].

1.2 Reed fibers

Reed is one of the most widely distributed plants worldwide. It can be found all over the world except in Antarctica[18]. As a wetland plant genus, reed grows mainly on lakes and river banks, and peat lands. Reed stands can also be found on the edge of the desert. In addition to fresh water, the reed can also grow in salt water [19]. With the strengthening of environmental protection awareness, wetlands have received legislative protection in many countries [20]. And when these protections are implemented, the source of economic income for local communities needs to be reconsidered. The economic value of reed is increasingly attractive because of its high yield and the fact that it is less likely to compete for land used for food production[21]. In 2008, it is reported that 6–7 million bundles (The length of a standard bundle is 1.2–1.7 m, its circumference is (60–)62–64 cm, its diameter is 20 cm, and it weighs 4.5–6 kg) of reed were harvested in The Netherlands[22]. The abundant output makes the application of reed in engineering possible.

Generally, agricultural stalk fibers are the main fibers used to make composite materials and have been used by many researchers. Wheat straw fibers are mixed with different types of binders to make particleboard[23]. Nourbakhsh and Ashori evaluated the application of agro-waste materials like corn stalks and oilseed stalks and concluded that these materials have superior mechanical properties[24].

Compared with these agricultural by-products, reed, one of the most widely distributed and highly productive wetland plant genera in the world, are readily and abundantly available at low cost due to its fast-growing and the ability to re-grow after frequent cuts without the requirement of fertilizer, it more competitive in raw material production costs[25]. And the rational management and logging of reeds can not only increase the yield but also enhance its function of purifying wetland water quality, making reeds more valuable when economic growth leads to accidental or unavoidable eutrophication of lakes and wetlands[21]. In addition, the unique growth environment of reeds also brings another large advantage. When peatlands are drained, large amounts of greenhouse gases are released due to the decomposition of peat[20]. Because reeds can grow on peat-formed silts, the commercial value brought by reeds can reduce the likelihood of these peatlands being drained, thereby limiting greenhouse gas emissions[21].

Similarly, the reed stalk has a similar microstructure to the above fiber with lignin content, high elastic modulus, and hardness[26]. Some researchers have evaluated the wide availability of reed fiber. Reichel mixed chopped reed with glue and clay to make granulate panels, which can be used as insulation or plaster base[27]. Composites of high-density polyethylene and common reed natural fibers showed a substantial enhancement in ductility[28]. Reed fibers are incorporated into concrete in the construction industry to reduce unit weight and improve thermal performance[29]. Ismail et al. reported the possibility to use reed ash and air-dried fibers to replace sand in concrete mixes[30]. Additionally, the effects of reed by-product fly ashes on the asphalt binders or mixtures also were investigated by researchers[31].

1.3 Project Objectives and Research ideas

As previously stated, the use of reed fiber is becoming justified in various construction technology. Many materials with a similar structure to reed fiber are used in road construction and have been verified to have excellent performance[16, 32, 33]. However, it is inhibited by a lack of research on the role of reed fibers in the pavement. Considering this background, to investigate more applications of reed fiber-modified bitumen (RFMB), the properties of RFMB with different fiber lengths and dosages were examined. The applicability of reed fiber in bitumen and the optimal combination of length and dosage were determined by testing nine groups of samples with different lengths and dosages of reed fiber in terms of high temperature, low-temperature performance, and fatigue properties.

The first chapter of this report gives a detailed description of fiber reinforcement and reed fibers. The second chapter introduces the materials and related experimental methods used in this study. The Fourier transform infrared spectroscopy (FT-IR) test is presented in Chapter 3, followed by the viscosity test in Chapter 4. Chapter 5 illustrates rheological measurements performed by the dynamic shear rheometer (DSR), including the Frequency sweep test, Multiple stress creep recovery (MSCR) test, Linear amplitude sweep (LAS) test and Relaxation test. Chapter 6 contains the tensile monotonic tensile test for mortar. The ranking of each specimen's performance based on the above experiments is presented in Chapter 7. A conclusion is presented in Chapter 8.

Chapter 2: Material and experimental methods

2.1 Material

2.1.1 Base bituminous

70/100 bituminous binder from Total Nederland N.V. was used in the experiments. Table 1 shows the physical properties and chemical components of this virgin bitumen.

Table 1 Physical properties and chemical components of virgin bitumen

Properties		Value	Test standard
25°C Penetration (1/10mm)		91	ASTM D5
Softening point (°C)		48	ASTM D36
135°C Dynamic viscosity (Pa·s)		0.8	AASHTO T316
25°C Density		1.017	EN 15326
60°C Density		0.996	
Chemical fractions (wt %)	Saturate, S	3.6	ASTM D4124
	Aromatic, A	53.3	
	Resin, R	30.3	
	Asphaltene,	12.8	
	AsColloidal Index CI		
Element compositions	Carbon, C	84.06	ASTM D7343
	Hydrogen, H	10.91	
	Oxygen, O	0.62	
	Sulphur, S	3.52	
	Nitrogen, N	0.9	
Complex shear modulus at 1.6 Hz& 60°C (kPa)		2.4	AASHTO M320
Phase angle at 1.6 Hz (°)		84.5	

2.1.2 Reed fiber

The different lengths of the reed fibers were produced by ESEM, Eindhoven, Netherlands. The color of the reed fiber was golden and the reed fiber was dry. The surface of this fiber was not modified. Obviously, if the fibers are too long, they may mix together, making it difficult to blend well with the bitumen, creating the so-called "balling" problem. Fibers that are too short do not provide enough reinforcement, making fiber additions a useless and expensive filler[2]. In this research, to study the effect of fiber length on the bituminous binder, considering the mixability, four lengths of reed fiber (<0.4mm, 0.4-0.6mm, 0.6-0.8mm, 0.8mm) were selected, as shown in Fig 1. And the reed fiber was added to the bitumen at a certain ratio based on the mass of the bitumen. The dosage of reed fiber in this study is determined concerning the three ratios of high, medium, and low in the experimental dosage of corn stalk fiber with similar properties to reed fiber. i.e., 2%, 6%, 10%.

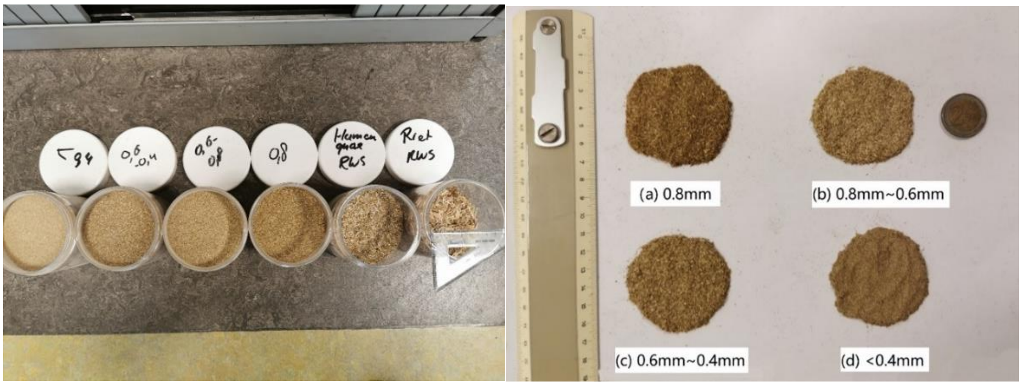


Fig. 1. Reed fiber s' appearance (0.8 mm, 0.8 mm~0.6 mm, 0.6 mm~0.4 mm, and <0.4 mm).

According to some previous experimental experiments, the difference between 0.6-0.8mm and 0.8mm on performance is very small. So the 0.6-0.8mm group is canceled in subsequent experiments.

2.1.3 Preparation of the modified bitumen

To avoid the potential effects of high-speed shearing on fiber length, a low-speed agitation method was used. Referring to the preparation of other similarly structured fiber-modified bitumen, the preparation of the RFMB is as follows[16, 34]:

(1) All these percentages of fibers are based on the weight of the bitumen. Weigh the reed fibers based on different content.

(2) The bitumen and fibers were placed in an oven and heated to 150℃.

(3) The above-mentioned were gradually added to the asphalt and used at low speed to blend for 30min

to disperse the fibers evenly.

The preparation process of the RFMB is shown in Fig.2.

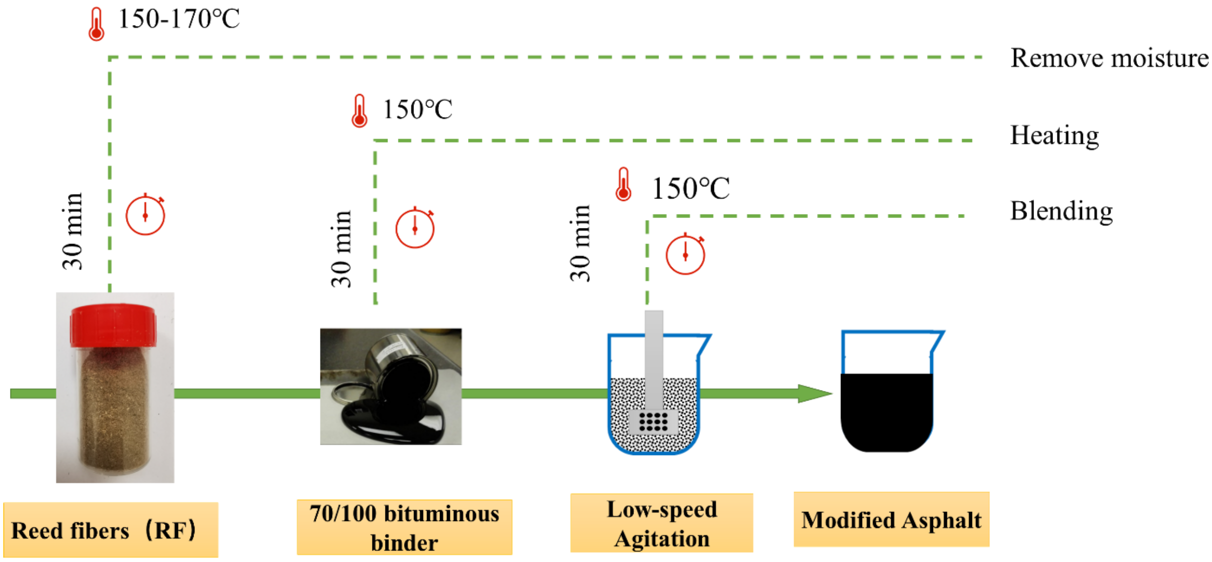


Fig. 2. Preparation process of reed fiber-modified bituminous binder

2.2 Experiment methods

Therefore, ten different groups of specimens including virgin bitumen are designed to investigate the optimal amount of reed fiber. Fourier transform infrared spectroscopy was measured to determine whether the addition of fiber affected the chemical structure of the bitumen. Subsequently, various rheological parameters such as complex shear modulus, phase angle, and viscosity were measured to characterize the rheological properties of the modified bitumen at various temperature ranges[35, 36]. Finally, a monotonic tensile test was performed on the mortar made with modified bitumen to determine whether the modification improved the tensile strength. The framework for this experimental program is illustrated in Fig 3.

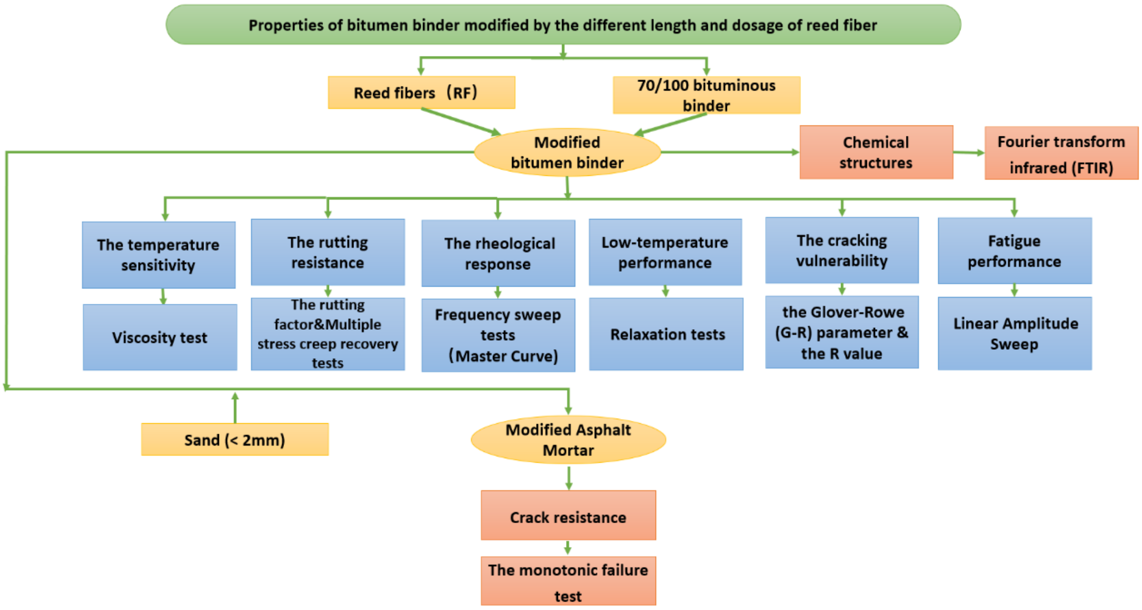


Fig. 3. The framework of the experimental program

Chapter 3 FT-IR

The effects of the reed fibers were detected using Fourier transform infrared spectroscopy (FT-IR) for the reed fiber-modified binder. The Reed fiber will be tested by different weights (2%，6%，10%). Fig. 4 (a) and (b) presents the FTIR spectra of the virgin bitumen, and modified bitumen with different amounts of reed fiber and pure reed fiber.

Based on the FT-IR spectra of reed fiber, the following wavenumber positions have more pronounced peaks, which represent the main functional groups: the C-O stretch (1053cm^{-1}) and C-C stretch (1028 cm^{-1}), appearing as cellulose deposition during the synthesis of the secondary cell wall of the fiber; NH_2 (1537 cm^{-1}), as a protein or amino acids; Adsorbed H_2O (1633 cm^{-1}), possibly due to moisture absorbed during open storage of the fibers, a hypothesis that will be verified by re-testing of the dried fiber; -CH_2 asymmetric stretching (2918 cm^{-1}) and originates from waxes and intermolecular hydrogen bonds (3334 cm^{-1}) on the cuticle of the fiber surface [37-39]. It is obvious from Fig. 4 (b) that the intensities of the absorption peaks of modified bitumen and virgin bitumen are almost the same, which indicates that the addition of reed fiber does not cause significant changes to the chemical components of bitumen, which suggests that the modification of the bitumen by the reed fiber was based on physical mixing without any obvious chemical reaction. Furthermore, a comparison of the absorbance values of the bitumen and fiber components shows that the absorbance of the fiber component is almost negligible. Considering again that the maximum addition of fiber is only 10%, the FTIR spectrum of bitumen is not greatly affected by the physical superposition effect of absorbance.

What is noteworthy is the high intensity of the reed fiber absorption peak at 1028 cm-1 and the increasing trend in peak area for 2%, 6%, and 10% RFMB at this location. Such a finding could be considered as a criterion for testing the homogeneity of the fiber and bitumen blend when promoting reed fiber in the future. However, according to the iS50 FTIR Spectrometer operating manual, the infrared light under the attenuated total reflection (ATR) test only interacts with the surface of the material in contact with the ATR crystal (Penetration depth-values of 0.25 - 2.5 μm in the 4000-400 cm^{-1} interval[40]). The lower fiber content set in the experiment and the limitation of the fiber length may result in no or few reed fibers on the side of the sample under test that is in contact with the crystal. Therefore, this application may need to be verified using transmission FTIR measurements.

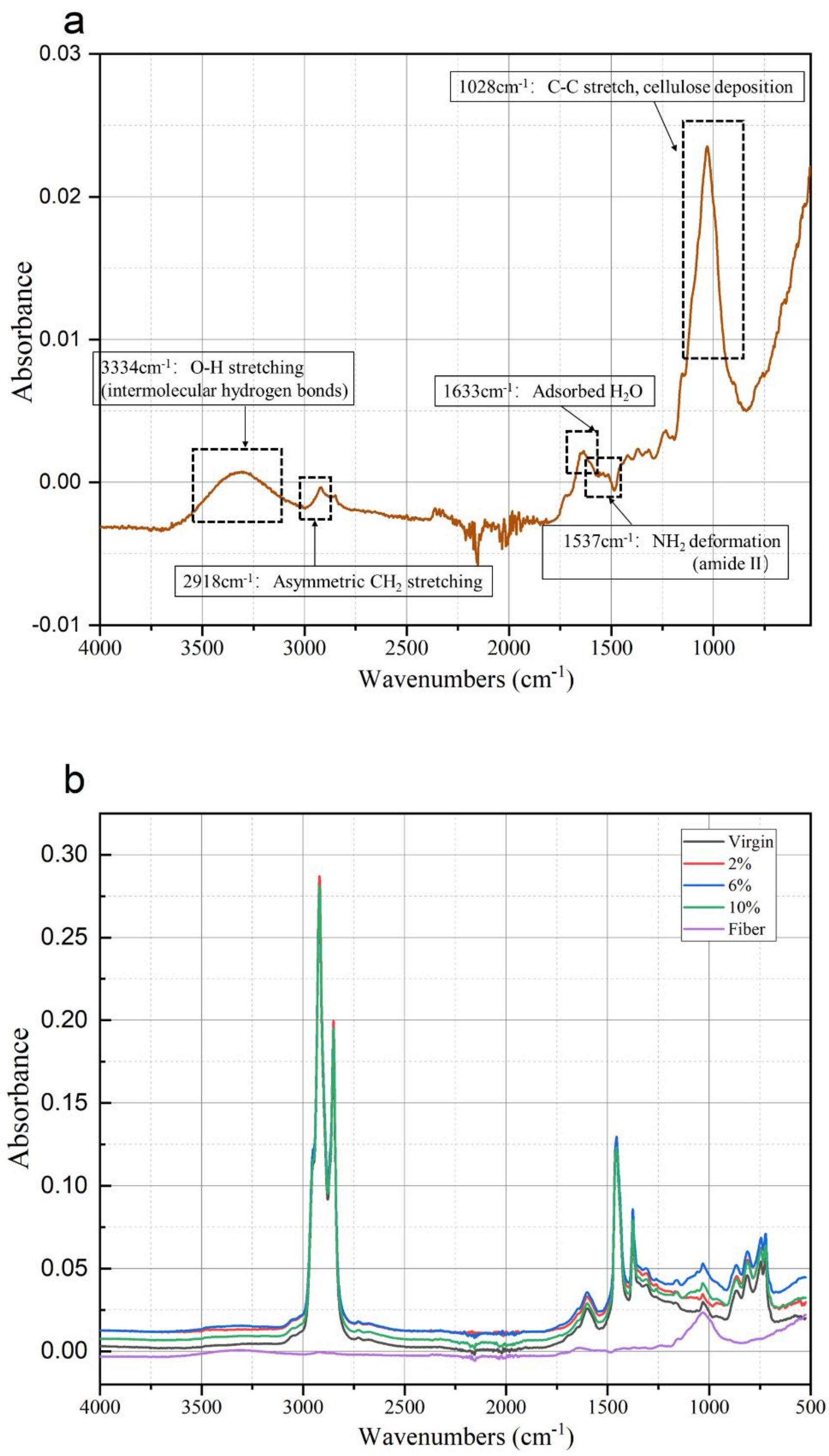


Fig.4 The FTIR spectra of (a)pure reed fiber and (b)virgin bitumen, RFMB

Chapter 4 Viscosity

The viscosity of modified bitumen with three different lengths and contents of Reed fiber was tested. The viscosity of modified bitumen reflects its flow characteristic and construction workability. According to the standard rotational viscosity test procedure, the viscosity of the bituminous binder at different temperatures was measured using a Brookfield viscometer and the viscosity-temperature curve was plotted. In this study, eight test temperatures, including 90°C, 100°C, 110°C, 120°C, 135°C, 150°C, 160°C, and 170°C were used. The viscosity at each temperature was measured and plotted to evaluate the temperature sensitivity of fiber-modified bitumen. Fig. 5 illustrates that the viscosity of RFMB decreased with increasing temperature and increased with increasing fiber length and content.

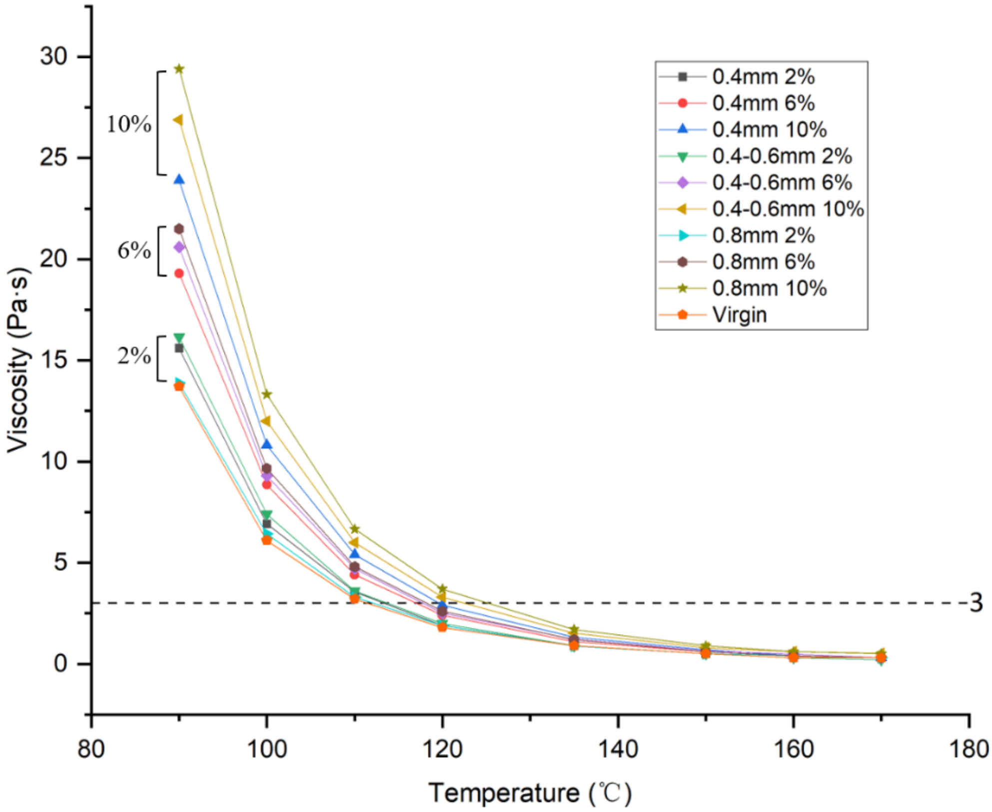


Fig. 5. Viscosity as a function of the temperature of each group

Furthermore, as shown in Fig 5, the viscosity of RFMB increases significantly with increasing content and forms three gradients according to fiber content. Although the length of the fiber also affects the viscosity of RFMB, the viscosity of the longest fiber at lower content is still smaller than that of the minimum length fiber at a higher content. When the fiber content is low, the reed fiber is only used as a dispersing material in the bitumen, and the viscosity increase is limited. But when the fiber content increases, the reed fiber begins to form a network structure, and the viscosity increases by 1-2 times compared with virgin bitumen. And it can be seen that the viscosity of all RFMB is higher than that of virgin bitumen.

From the construction temperature perspective, the RFMB temperature needs to be raised appropriately during storage, blending, and pumping. After mixing, transportation, and pumping, the mixture is generally compacted at a temperature of 135℃[41-43]. The AASHTO specification requires the viscosity of the modified bitumen to be no greater than 3 Pa·s at 135°C[43]. Therefore, it is considered that asphalt with a viscosity of 3 Pa·s at temperatures below 135℃ is acceptable, and the lower the temperature the better. Alternatively, bitumen with a viscosity below 3 Pa·s at 135℃ is considered acceptable, and the lower the viscosity the better.

The viscosity-temperature curve was used to determine the temperature at which the viscosity of each sample was 3 Pa·s at 20 rpm, to determine the temperature at which each sample could be well compacted in practical engineering, shown in Fig. 6.

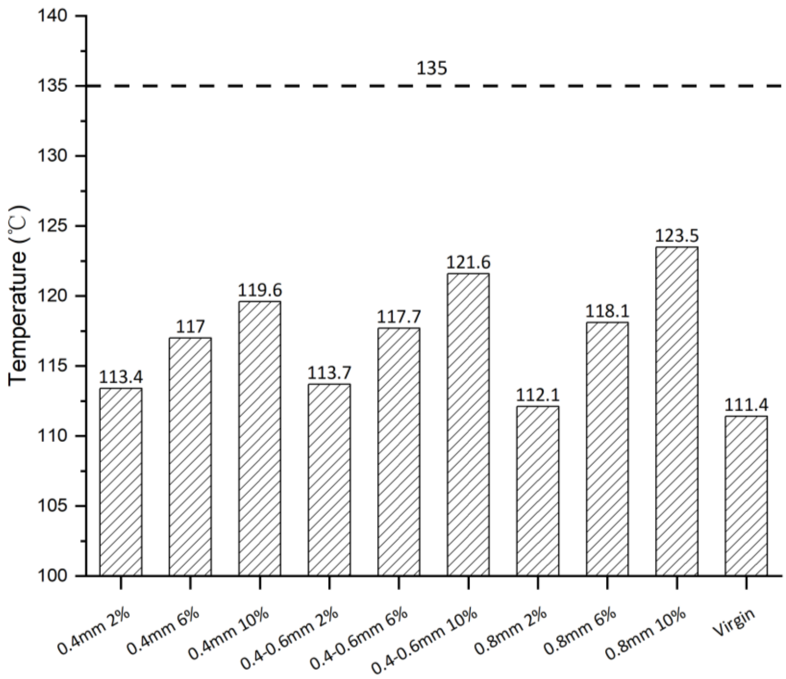


Fig. 6. Temperature with a viscosity of 3 Pa·s per sample at 20 rpm

It can be seen from Fig. 6. that the addition of reed fiber increases the compaction temperature, and the trend is the same as the analysis of the above viscosity. It is worth noting that the temperature of the 0.8 mm 2% sample is lower than that of the same content of longer fiber length, which is contrary to the previous conclusion. It may be because 0.8mm is difficult to blend evenly due to its long length, and the actual content of fiber in the test sample is small.

Besides, the viscosity at this 135℃ of each sample is shown in Fig.7. The viscosity of the modified bitumen with low fiber content hardly changed, and the viscosity of the modified bitumen with high fiber content increased by 50%-80%, but still met the viscosity requirement of 3 Pa·s. From these results of each sample, the viscosity is lower than this limitation, indicating that the addition of fiber will not cause the bitumen to be unusable in actual engineering.

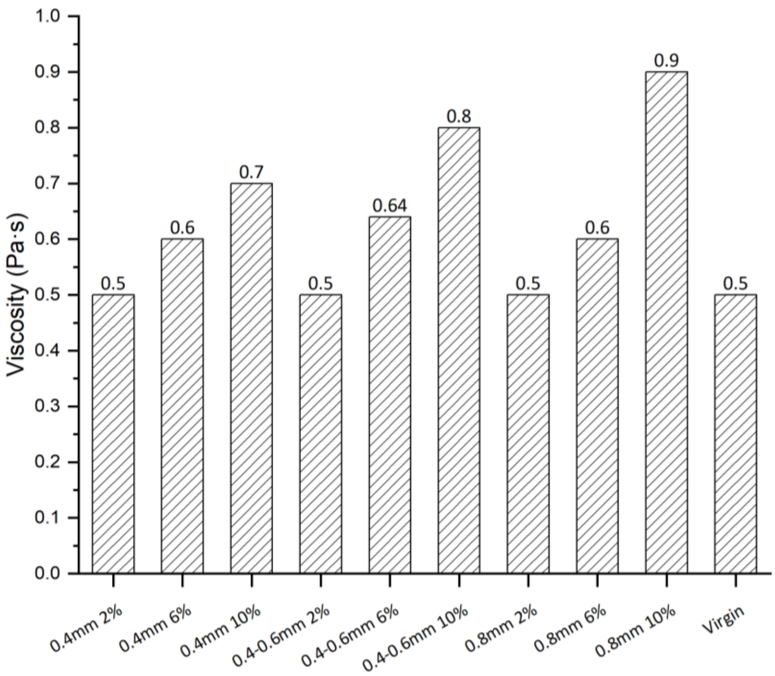


Fig.7. Viscosity at 135℃

Chapter 5 Rheological Measurements

The dynamic shear rheometer (DSR) was selected to run the Frequency sweep test, Multiple stress creeps recovery (MSCR) test, Linear amplitude sweep (LAS) test, and Relaxation test to assess the rheological performance of the reed fiber-modified bitumen[44].

5.1 Frequency Sweep

The complex modulus and phase angle of the bituminous binder modified by reed fibers will be measured at different temperatures (0°C, 15°C, 30°C, 40 °C, 60°Cand 80°C) and frequencies (0.1–10 Hz). Then the master curves for complex modulus and phase angle had been presented.

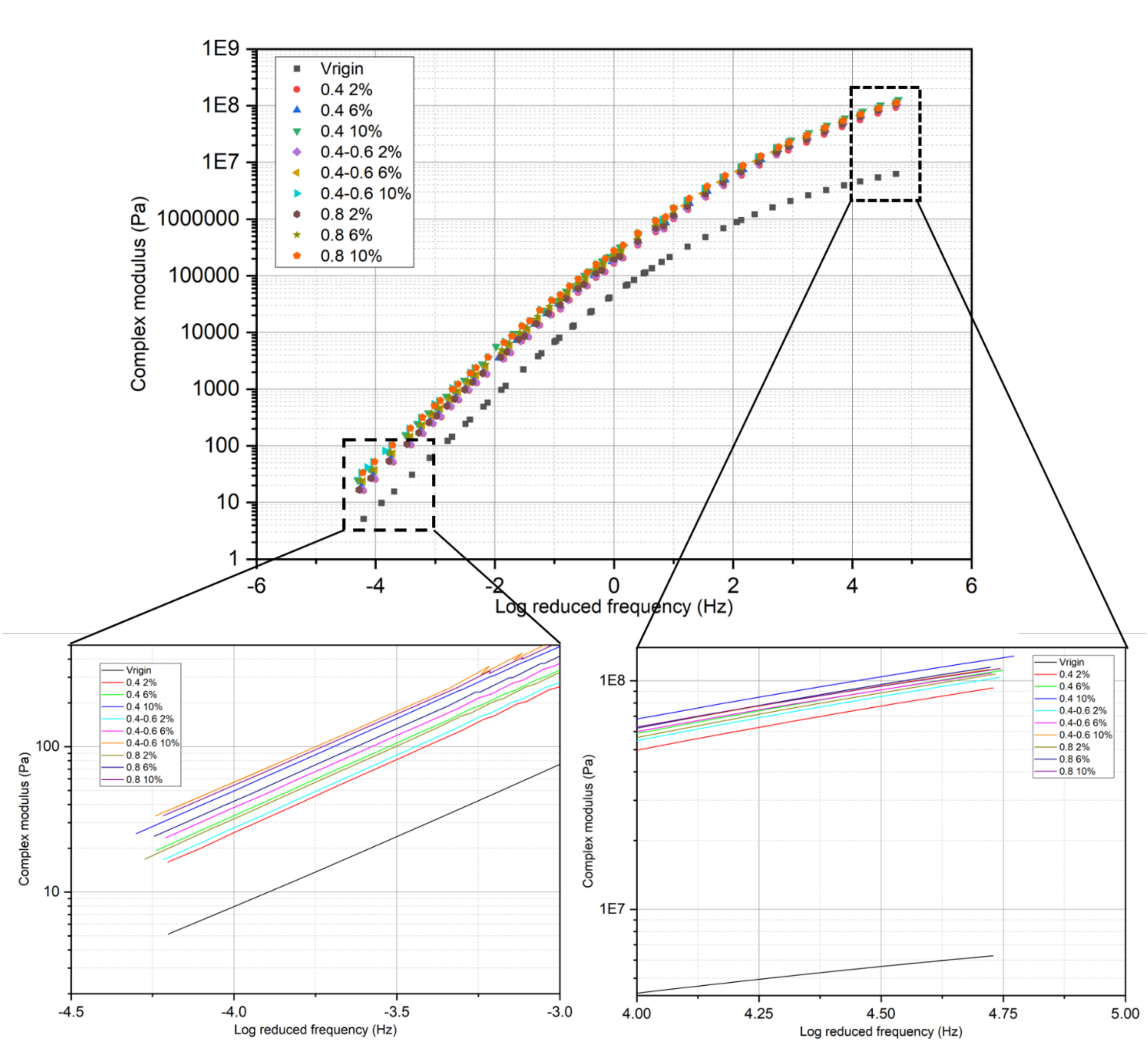
Based on the time-temperature superposition principle, the sigmoidal model is utilized to establish the master curve of the reed fiber-modified bitumen, which is expressed below[45].

$$lg|G^*| = \delta + \frac{\alpha}{1 + e^{\beta+\gamma\times\lg(f_r)}} \quad (1)$$

Where G^* is complex modulus, δ is value of the lower asymptote of complex shear modulus $|G^*|$, α is value of the upper asymptote of complex shear modulus $|G^*|$, β and γ are shape factors.

The temperature of 30°C is chosen as the reference temperature. Then determine the fitting parameters of the complex modulus. Fig.8 shows that the complex modulus of the RFMB increased as the loading frequency increased. And with the increase of the reed fiber content in the bitumen, the complex modulus of the fiber-modified bitumen increase. Similarly, the increase of the fiber length will also increase the master curve of the complex modulus, but the effect is smaller than that of the increase of the dosage, which is the same as the conclusion obtained in the previous viscosity experiments. In addition, the low-frequency region corresponds to the performance of bitumen at high temperatures, and the high-frequency region corresponds to the low-temperature performance. From the partial zoom frequency part of Fig. 8, it can be seen that with the addition of fibers compared with virgin bitumen, the modulus of all temperatures is greatly increased, and in the higher frequency region, the curve distance between RFMB group and virgin is larger, which means that the addition of reed fiber has a more obvious effect in this area. When comparing within RFMB groups, it can be found that the modulus increases by about 100% at high temperatures, while the difference is about 10% at low temperatures, which is relatively insignificant. The higher the G* value, the harder the bitumen.

Fig. 8. Complex modulus master curve at 30℃



It can be seen that there is a significant increase in the complex modulus of the RFMB in the high-frequency region, implying that the RFMB is stiffer at low temperatures, which may lead to crack resistance problems. At this point, the black space diagram is quoted to assess the crack resistance. Black space diagram is a graph of complex shear modulus, G^*, versus the phase angle, δ without the requirement to perform the Time-temperature superposition principle. It is convenient to use it to provide 'fingerprints' for different types of bitumen. Anderson and his team [46] developed a method to fit the master curve of G^* and phase angle with an important parameter R-value, shown in the following equation.

$$R = \frac{(log2) \times log\frac{G^*(\omega)}{G_g}}{\log{(1-\frac{\delta(\omega)}{90}}} \qquad (2)$$

Where $G^*(\omega)$ is complex modulus at frequency ω, G_g is glassy modulus and assumed to be 1E+09 Pa, $\delta(\omega)$ is the phase angle at frequency ω. With the aid of the regions divided by the R=1, R=2, and R=3 curves, it is interesting to view the results of modification on a Black space diagram about potential damage, as shown in Fig. 9.

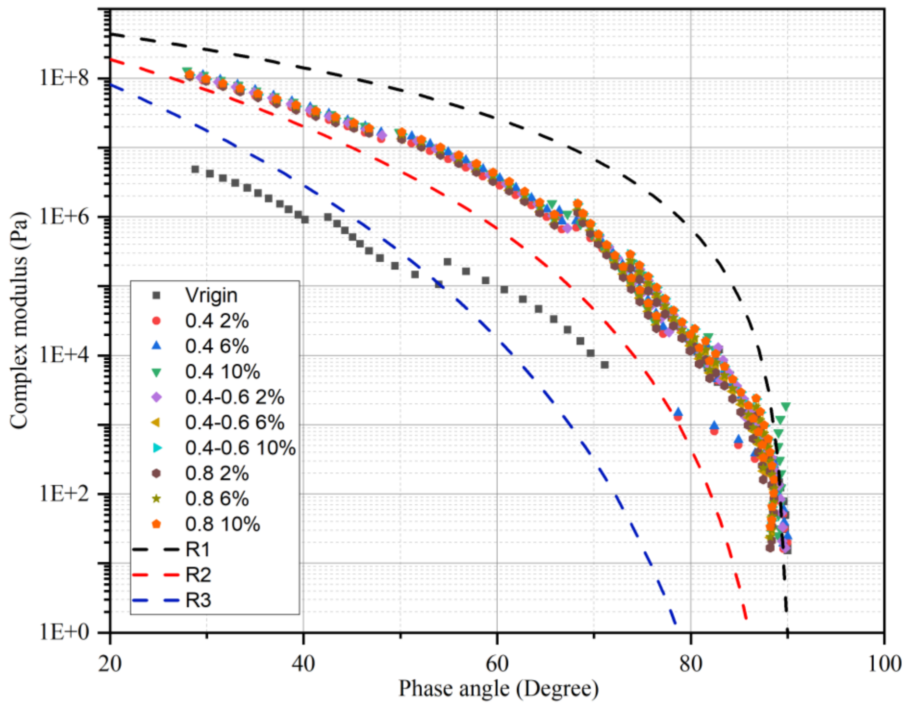


Fig. 9. Black space diagram of rheological data for each sample

Based upon ranges in R-value, Higher R-values mean that cracking is more likely to occur, i.e. the area in the lower-left corner of the graph represents the possibility of block cracking and the area in the upper right corner represents no block cracking. As can be seen in Figure 9, the overall shift in the curve after the addition of the reed fiber means that RFMB is closer to the curve for lower R values than virgin bitumen and is consequently less vulnerable to cracking. Furthermore, this difference is more pronounced in the lower phase angle region, i.e. in the low-temperature section, indicating that the addition of fiber considerably improves the low-temperature cracking resistance of the bitumen. In contrast, for the groups where fibers were added, all curves almost overlapped, indicating that the difference in the effect of length and content on their properties was not significantly noticeable.

5.2 Multiple stress creep recovery (MSCR)

The presence of elastic response in bitumen and the change in elastic response at two different stress levels were identified by the multiple stress creep recovery (MSCR) method following ASTM D 7405-15[43]. Analyzing the effectiveness of the addition of reed fibers on rutting resistance by determining the percentage recovery and the non-recoverable creep flexibility of bitumen. According to AASHTO[43], $J_{nr\,0.1}$ and $J_{nr\,3.2}$ indicate the average nonrecoverable creep compliance at 0.1 kPa and 3.2 kPa respectively. This suggests that the higher the J_{nr} value, the lower the rutting resistance of the bitumen. And J_{nrdiff} means the percent difference in nonrecoverable creep compliance between 0.1 kPa and 3.2 kPa. This value characterizes the stress sensitivity of the material. The results of MSCR under 70℃ and 76℃ are shown in Fig. 10 and Fig. 11.

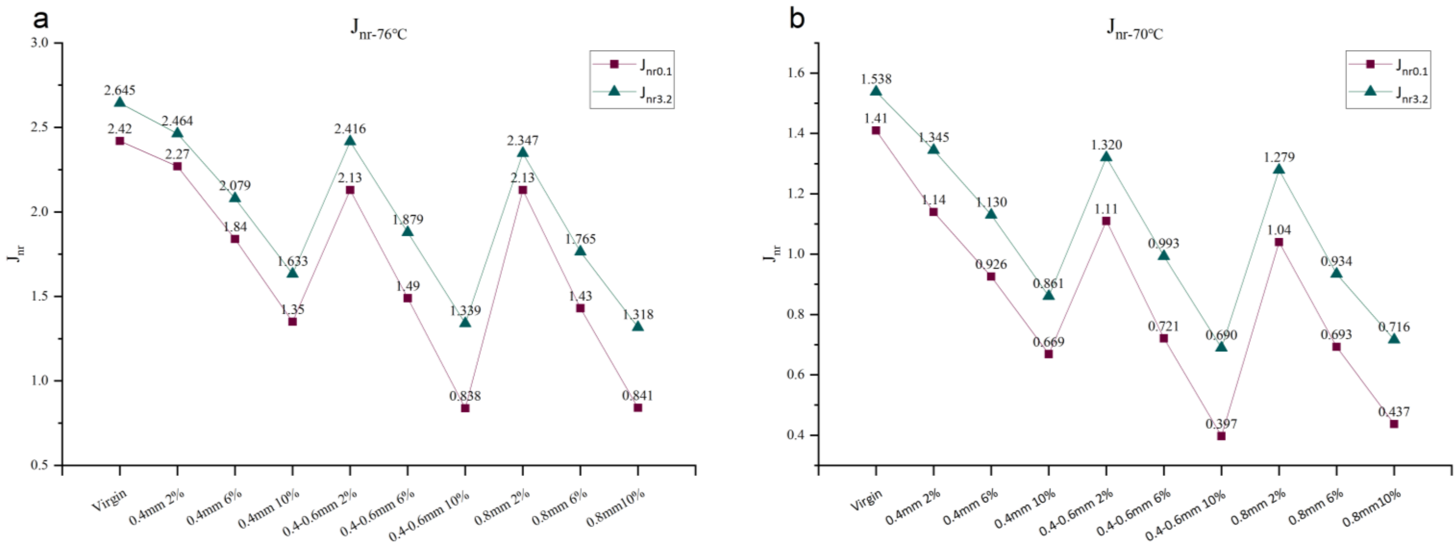


Fig. 10. J_{nr} under (a)76℃ and (b)70℃

As can be observed, the J_{nr} of all RFMBs is less than virgin bitumen at two different temperatures and stress levels. In terms of the three different contents, the J_{nr} decreases with increasing reed fiber content ,and the tendency to decrease is in an approximately linear relationship. As for the effect of fiber length, J_{nr} slightly decreases with increasing fiber length, but the influence is not noticeable. The modified bitumen with 10% 0.4-0.6mm reed fiber showed better rutting resistance at different temperatures and stress levels.

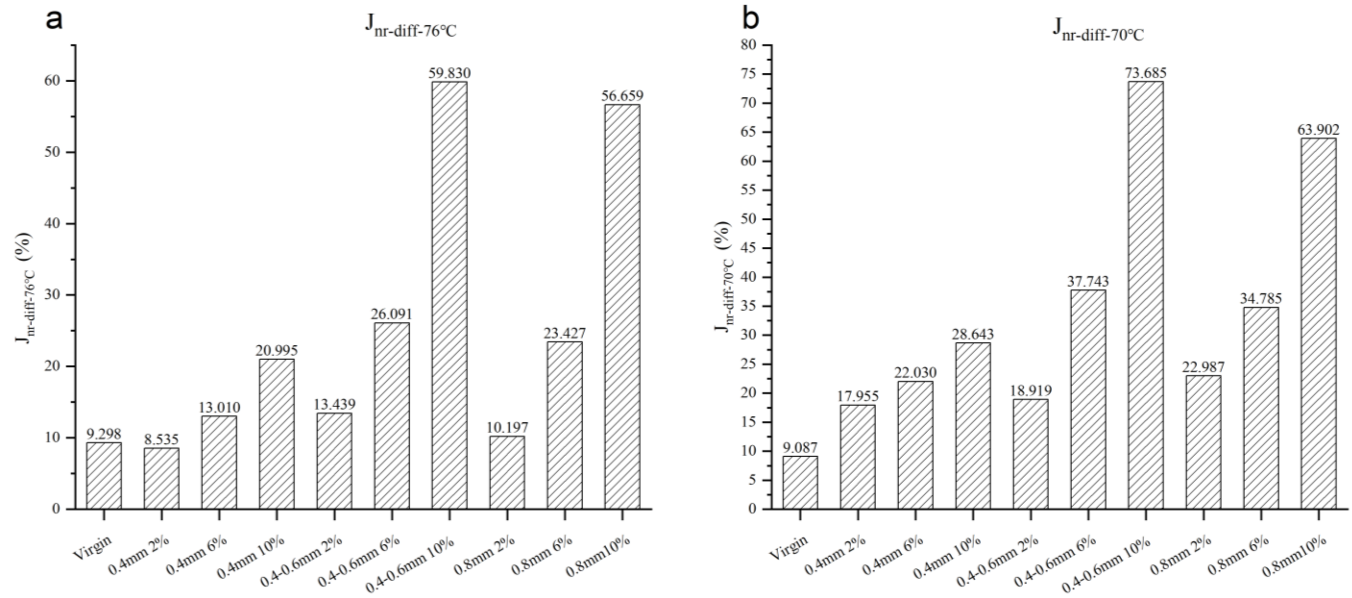


Fig. 11. J_{nrdiff} under (a)76℃ and (b)70℃

However, regarding J_{nrdiff}, the addition of fiber led to an increase in stress sensitivity and the result was the exact opposite of the improvement in rutting resistance, i.e. the stress sensitivity of the asphalt increased significantly with increasing fiber content and the 10% 0.4-0.6 mm reed fiber group with the best improvement in rutting resistance had the worst stress sensitivity. A possible reason for this is the agglomeration effect of the longer fibers at high content, which causes the fibers to become merely useless fillers in the bitumen. This situation is less pronounced in the 0.4 mm fibers.

Based on these data, it is clear that none of the bitumen binders exceeded the maximum 75% J_{nrdiff} limit specified in AASHTO M332. Given the variation in the samples and the blending conditions in the actual engineering, the shortcomings in the stress sensitivity of the 10% 0.4-0.6mm reed fiber group may make this modification unusable. Combining the results of rutting resistance and stress sensitivity, the 0.4-0.6mm 6% as well as the 0.8mm 6% modified bitumen showed superior rutting resistance as well as relatively lower stress sensitivity.

5.3 Relaxation test

Using an 8 mm diameter plate with a 2-mm gap and testing at 0 °C, bitumen samples were examined by DSR. The relaxation tests began with 1% shear strain (in 0.1 s) and were followed by 100 s of relaxation period. The data collecting frequency was set to 100 Hz. As an example, the stress time curve for the reed fiber 0.4 mm 6% group is shown in Fig.12. At low temperatures, the relaxation test can be used to assess stress production and relaxation abilities. The maximum shear stress of each sample under the same shear strain is shown in Fig.13. By normalizing the initial maximum shear stress to 100%, the time for the stress to return to 50% T_{50} and 10% T_{10} for each sample is shown in Fig. 14. One can easily notice that shear stress increases substantially with the addition of reed fibers, indicating that the RFMB is stiffer, meaning that the modified bitumen is stiffer at low temperatures, but also leading to more brittleness.

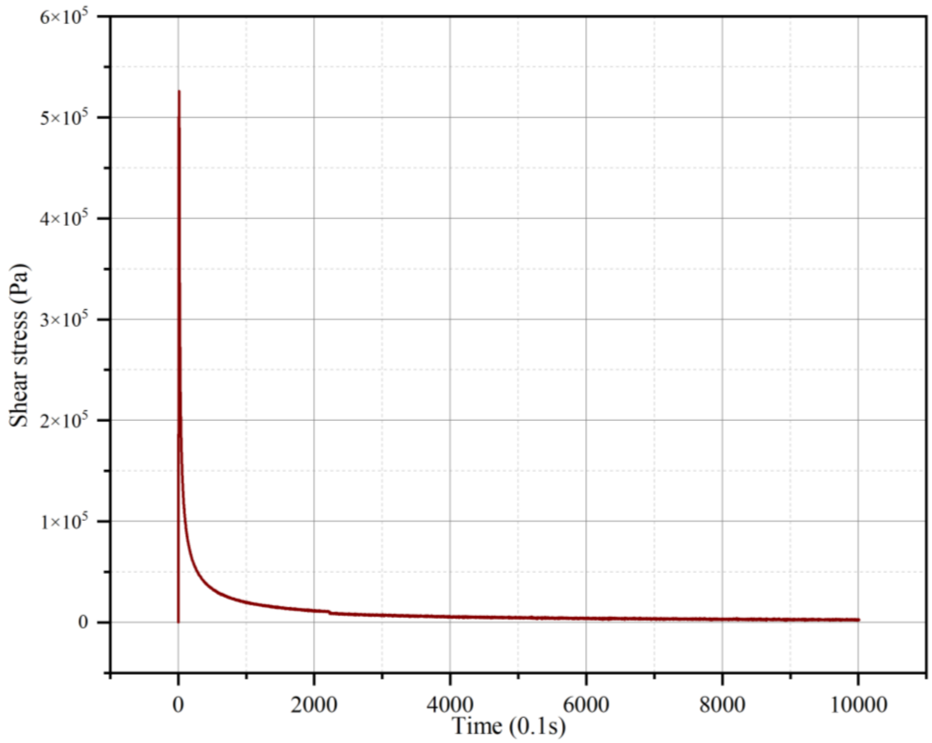


Fig. 12. Relaxation test stress-time curve of 0.4mm 6% reed fiber

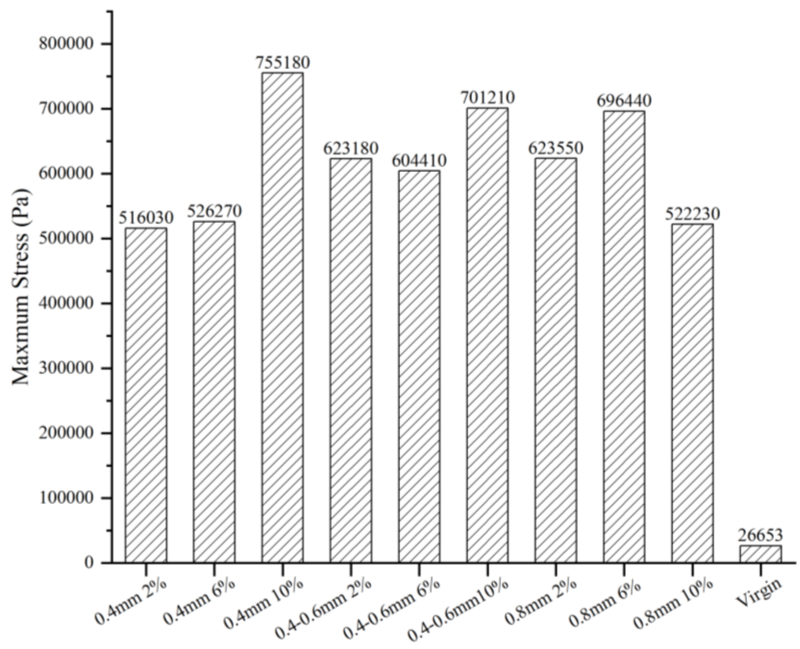


Fig. 13. Maximum shear stress at relaxation test

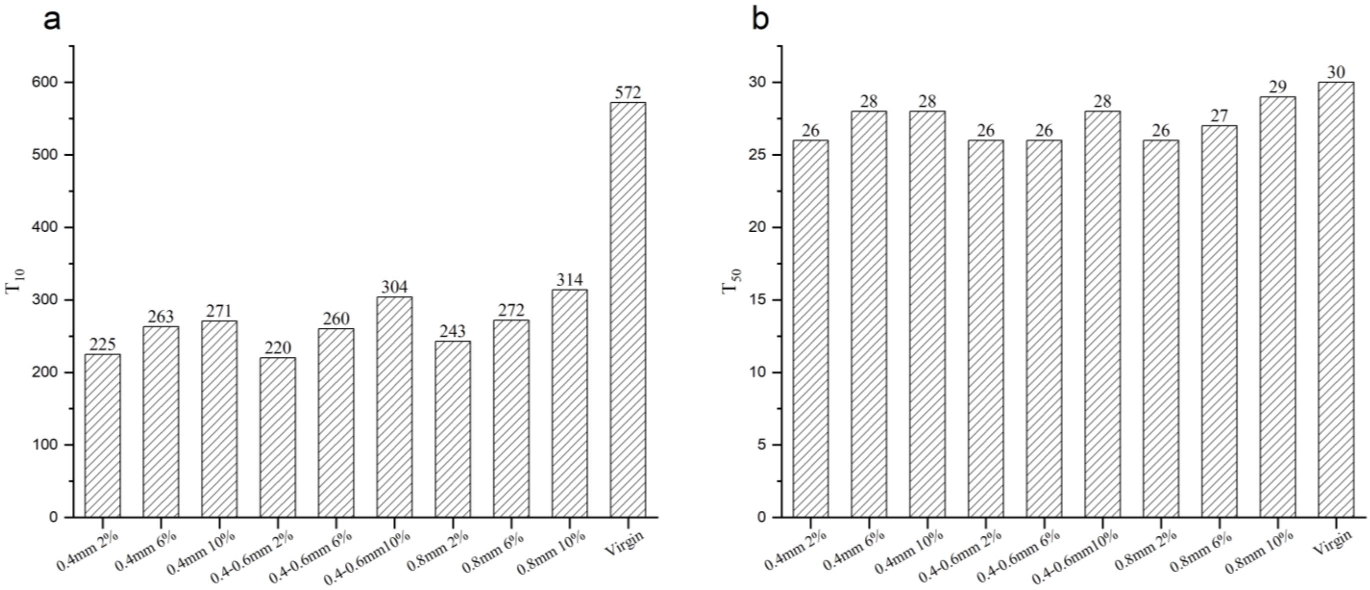


Fig. 14. The time for the stress to return to (a) 10% and (b) 50%

As can be observed from Fig. 14, the RFMB remains virtually unchanged in the time required for the stress to fall back to 50%. However, for the time it takes for the stress to fall back to 10%, the time for RFMB is approximately equal to half that of virgin bitumen, the addition of the fibers enhances the stress relaxation and reduces the residual stress. The reduction in relaxation time can be explained by the fact that it is related to the properties of viscoelastic materials, that is, when the external force disappears, the elastic part of the material quickly releases part of the force, and then the viscous part releases the remaining stress after a subsequent period. The incorporation of reed fibers makes the bitumen more viscous, which in relative terms also leads to an easier release of the remaining stresses.

Another possibility is that the strain applied to the sample in DSR is created by twisting the plate that bonds the bitumen, and this method relies on the adhesion between the plate and the bitumen. The addition of fiber may have caused the presence of fibers in this contact surface, resulting in a reduction in the adhesion, making it impossible for the bitumen to maintain this strain after the strain has been applied. The relaxation of the stress may therefore be due to a change in strain.

5.5 Linear Amplitude Sweep

The linear amplitude sweep (LAS) test was performed to understand the fatigue criteria. The sample is tested in shear using a frequency sweep to determine rheological properties and then tested using a series of oscillatory load cycles at linearly increasing amplitudes at a constant frequency to cause accelerated fatigue damage. The frequency sweep test employed an applied load of 0.1 percent strain over a range of frequencies from 0.2-30Hz. Test data is used to determine the damage analysis "alpha" parameter. Amplitude sweep is run at 10Hz with linearly increased strain from zero to 30% over the course of 3100 cycles. Peak shear strain and peak shear stress are recorded every 10 load cycles, along with phase angle and dynamic shear modulus.

The results of linearly increasing amplitudes present in Fig. 15. The area enclosed by the LAS curve is shown in Fig. 16, and this area represents the energy required to destroy the sample It can be found that as the shear strain increases, the shear stress response for all RFMBs shows a clear peak, after which the stress drops dramatically, indicating that damage has occurred in the samples. Furthermore, for the modified bitumen with the addition of 0.4mm reed fibers, as shown in Fig. 15(a), the peak stress elevated with increasing fiber content and the peak appeared at a higher strain value, indicating that the modified bitumen with the addition of fibers remained undamaged at higher strains and became increasingly effective as the fiber content increased. However, other fiber length groups did not show the same regularity concerning fiber content as the 0.4mm group, but all were further back than the peak position of virgin bitumen. Similarly, as shown in Fig. 16, the area enclosed by the LAS curve showed a similar regularity with the increasing fiber content in the 0.4mm and 0.8mm groups, but the impact of the content variation on the results was not clear in the 0.4-0.6mm group, so the effect of increasing fiber content on the improved strain tolerance of the bitumen requires more subsequent investigation. For the present analysis of the data, however, the addition of 10% 0.4mm reed fiber produced the best improvement in the tensile strength of the bitumen.

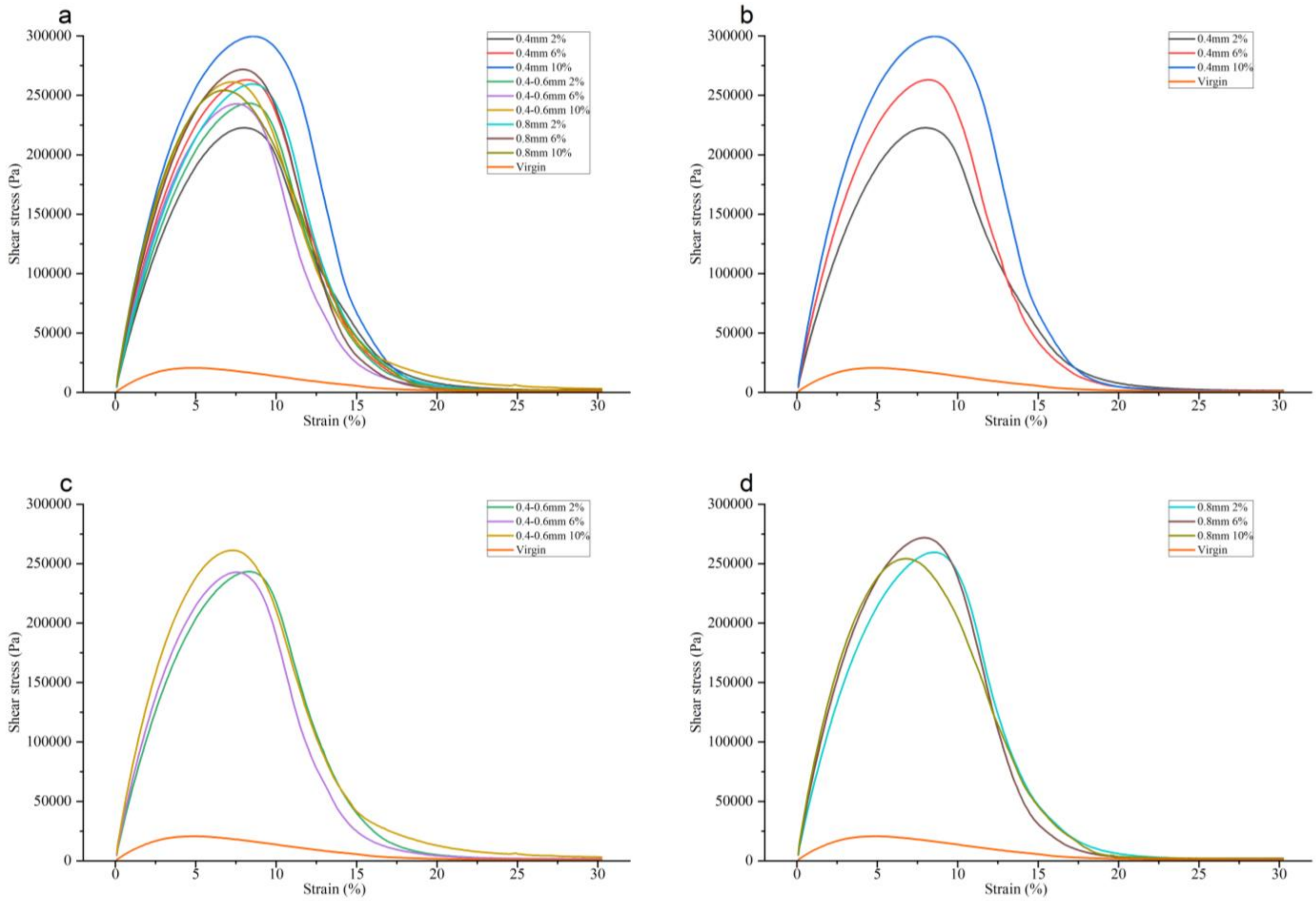


Fig. 15. Stress-strain curves form the LAS test at different reed fiber length:(a) all (b)0.4mm; (c) 0.4-0.6mm; (d) 0.8mm.

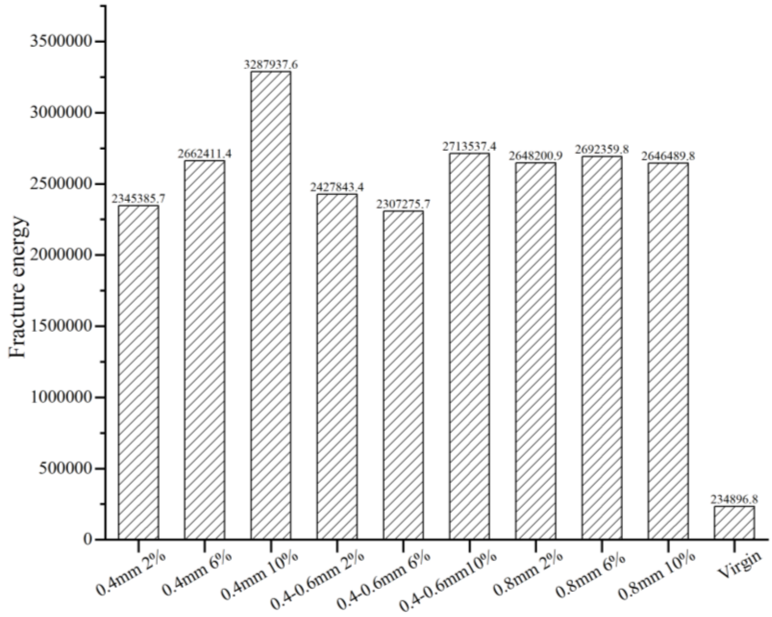


Fig. 16 The area enclosed by the LAS curve

According to AASHTO TP 101-14, the bitumen fatigue performance parameter N_f can be calculated based on the above data. Figure 17 demonstrates the predicted N_f of the bitumen by showing it at two strain levels, i.e. 2.5% and 5%. Obviously, RFMB exhibits excellent fatigue resistance at both of these given strain levels. Undoubtedly, the 2% fiber addition had the best effect on improving fatigue resistance at all lengths and strain levels. At the same fiber length, fatigue life showed a clear linear decrease with increasing fiber addition content. At a strain level of 5% and a fiber content of 2%, the fatigue performance of reed fibers with 0.4mm, 0.4-0.6mm, and 0.8mm additions was 16.8, 17.2, and 17.4 times that of virgin bitumen respectively. Whereas when the content was increased to 10%, the fatigue performance decreased to 11.3, 7.7, and 6.5 times that of virgin bitumen, respectively. While at strain levels of 2.5%, the values for 2% fiber dosage are 13.0, 13.1, and 13.6 times higher and 9.0, 6.5, and 5.4 times higher at 10% dosage. By combining the analysis of the above statistics, one can easily identify that the effect of fiber content is even stronger for the group with longer fiber lengths. In general, the reed fibers have a significant effect on the improvement of fatigue resistance. Of all the strain amplitudes applied, RFMB with a reed fiber length of 0.8 mm and a 2% addition had the most noticeable fatigue life.

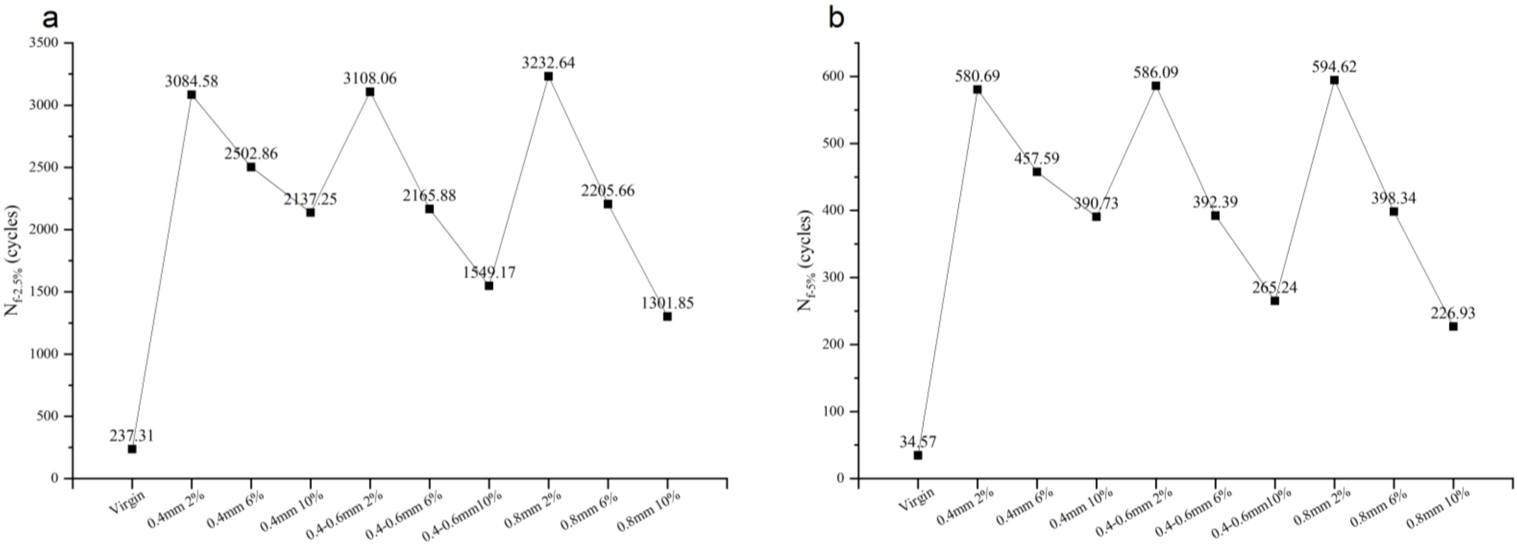


Fig. 17 Predicted fatigue lives at (a) 2.5% and (b)5% strain amplitudes

Recently, the LAS test has been utilized to characterize the relative damage tolerance of asphalt binders based on trends in the macrocrack growth rate [47]. The crack length (a_f) calculated using the LAS test represents the maximum allowable crack length of the sample before the damage, as shown in Fig.19. After this position (a_f) the growth rate of the crack length of the sample increases abruptly, indicating that the sample has been damaged. Larger crack lengths at breakage are preferable, as this indicates that the binder can tolerate larger crack lengths before complete breakdown[48].

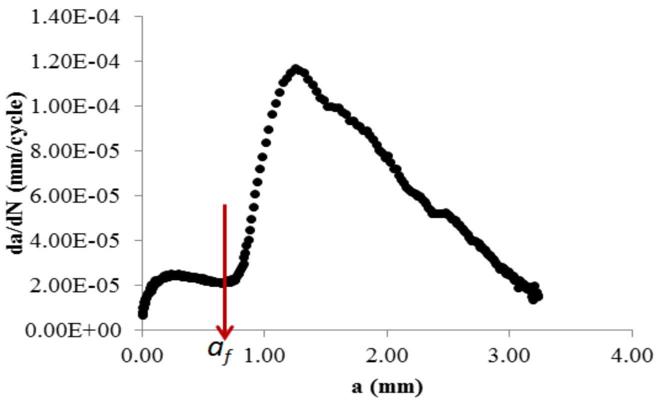


Fig.18 Cracking length demonstration

The results of cracking length at the failure of each specimen are shown in Fig.19. It reveals that the addition of fiber causes a dramatic reduction in crack length and then a slight recovery as the fiber content increases. This implies that the fibers have a strong negative effect on the relative damage tolerance of the bitumen and that increasing the fiber content slightly mitigates this reduction.

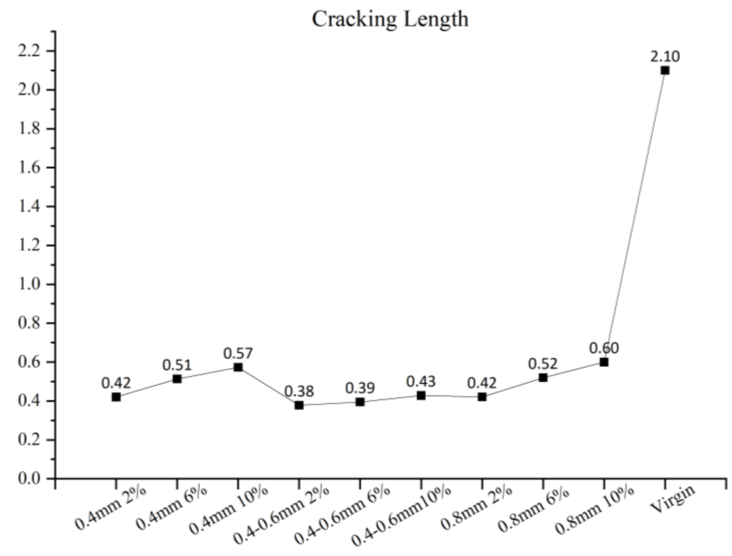


Fig. 19 Crack length at failure

Chapter 6 Monotonic tensile test for reed fiber reinforced mortar

6.1 Introduction and Experiment method

In light of its relationship to fatigue and crack resistance, a bituminous mixture's tension resistance is a crucial property. Uniaxial direct tensile testing of ordinary cylindrical samples would inevitably encounter the problem of uncertainty of the failure location, which may even happen near the end caps of the specimens[49]. To enable tensile damage to occur at the central location of the specimen, the specimen was modified into a complex geometry similar to a dog bone[50]. The aim of this study was to investigate the effect of reed fibers on the mechanical properties of asphalt mortar specimens using samples with a parabolic shape with a straight section in the middle.

The test specimens were manufactured by the following procedure. First, bitumen, sand, and fiber were preheated to the proper temperature and then mixed according to the dosage in Table 2 to complete the mortar mixture. The mixture was then heated back in the oven to maintain its rheology and make it easier to manipulate, and the silicone mould was also heated in the oven together. Lastly, the mortar was poured into the mould, as shown in Fig. 20(a). After pouring, the mould is set for a while to allow the mortar to flow freely to fill the pores. And then placed in the refrigerator until the samples reached a relatively low temperature and a reasonable stiffness so that they can be easily demoulded and keep their shape. Six specimens can be made per mould at a time, and the specimens are stored horizontally in a sand-filled storage container when they are removed from the mould, as shown in Fig. 20 (b), and checked for large pores or other defects.

Table 2 Material dosage for every three specimens prepared

Sand	Bitumen	Filler	Reed Fiber		
			2%	6%	10%
100g	50g	50g	1g	3g	5g

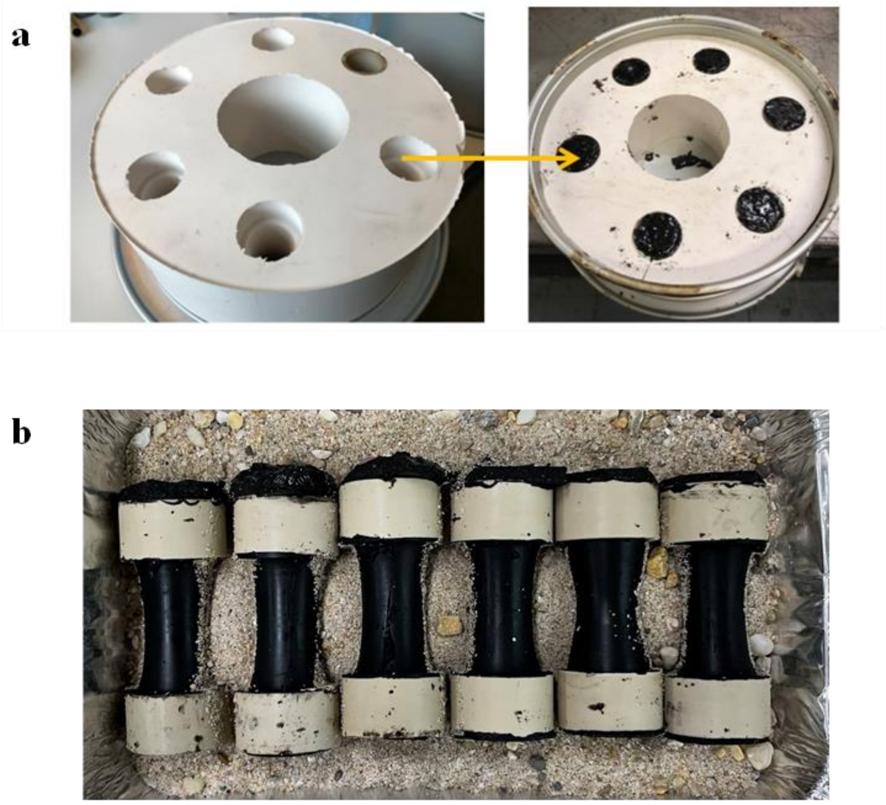


Fig. 20 (a) The silicone mould with mortar and (b) mortar sample after demoulding

The now-ready specimens are subjected to a monotonic tensile test. The mortar sample was loaded in tension with a displacement-controlled speed of 0.1 mm/s under 5℃ and 20℃ by Universal Testing Machine（UTM）. The fixation of the specimen during loading is shown in Fig. 21(a). The result of the monotonic tensile test is a series of force-displacement data. Finally, the peak stress and the total amount of dissipated energy of the test can be determined, as shown in Fig.21 (b).

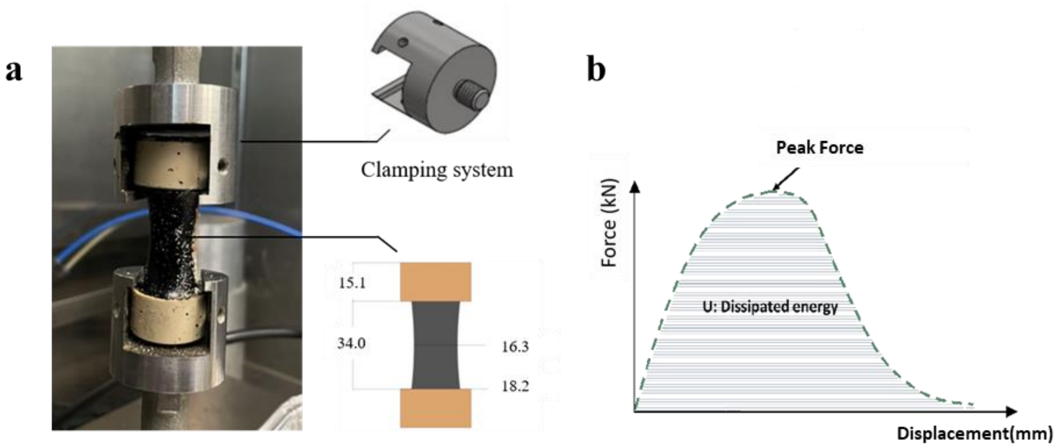


Fig. 21 (a) Tensile test setup and (b) monotonic tensile Force-displacement curve

6.3 Results and discussion

The Force-displacement data from the monotonic tensile test and the maximum tensile force of each specimen are shown in Fig. 22. Typically, bituminous mortars increase in tensile strength with the addition of fibers [51]. It is interesting to note that mortar contains 6% reed fiber at either 5 or 20℃, the strength was the lowest in the group with the same fiber length, and even the mortar with 6% 0.8 mm added had a lower strength at 5℃ than the mortar with no fiber added. According to the results of the error bar, the effect of the addition of fiber on the tensile strength is not yet clear, but at 20℃ in the 0.4-0.6 mm and 0.8 mm groups with a 2% addition showed an impressive increase of 3.09 and 2.59 times respectively. This phenomenon can be explained by the fact that the stiffness strengthening of the fiber to the matrix is related to the fiber reinforcement effect and that an increase in temperature promotes the degree of incorporation. Although an increase in the amount of fiber in the specimen increases the ability to transfer forces between fibers, the weakest link within the specimen is the bond at the fiber-mastic interface [52]. At higher temperatures, the fiber-mastic adhesion is stronger than the adhesion strength of the mortar itself, so this weakness can be mitigated by a higher fiber proportion, which explains why the strength decreases with the addition of 6% reed fiber at 20°C and increases slightly with the addition of 10%. The lack of a clear pattern in the results for the groups at 5°C and the lack of significant differences in values between the groups is since the effect of load transfer between fibers is not significant at low temperatures. Nevertheless, the phenomenon of an increase in tensile strength of the mortar with the addition of reed fibers is still ambiguous and requires more research.

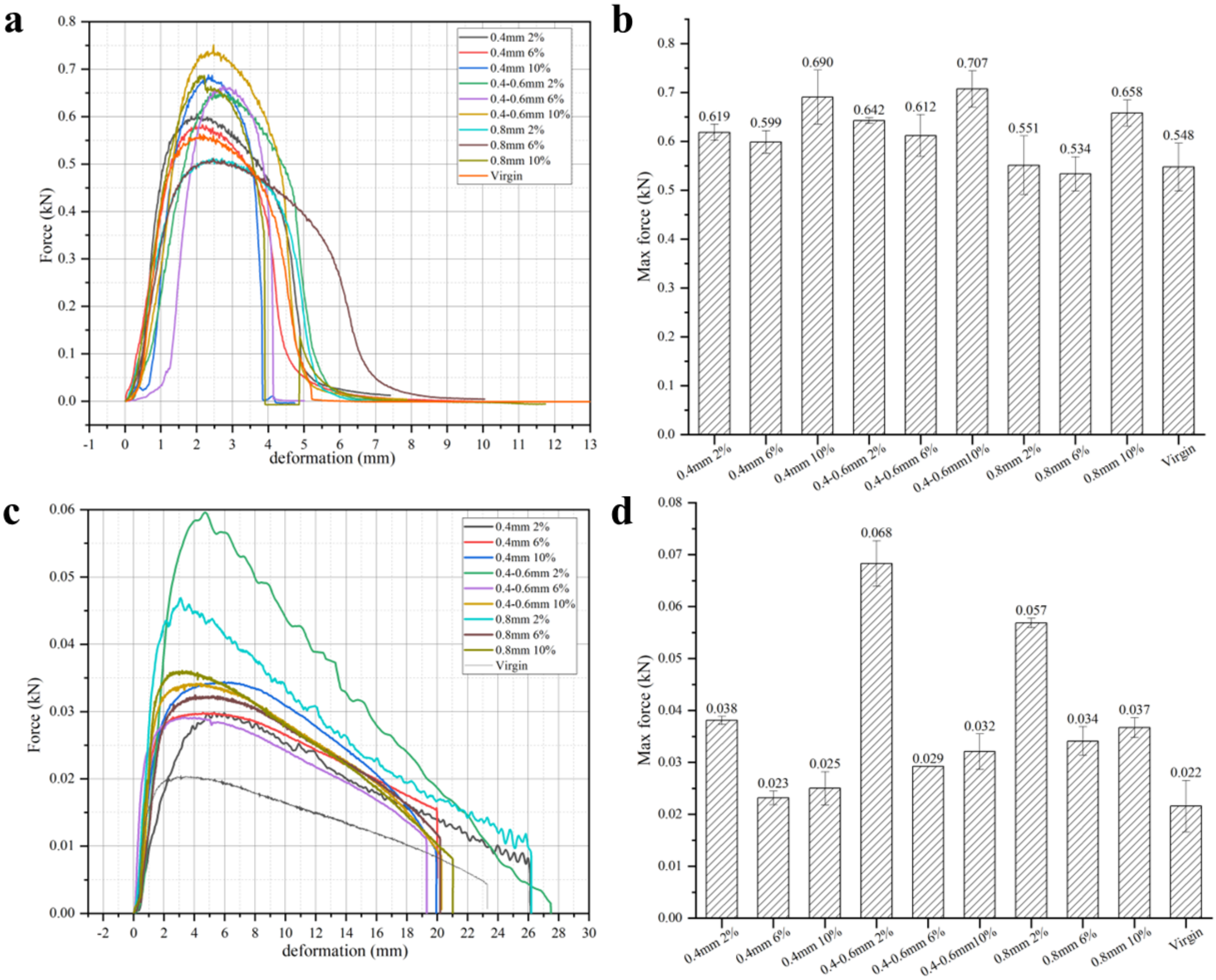


Fig. 22. Force-deformation curve at (a) 5℃, (c) 20℃ and maximum tensile force at (b) 5℃, (d) 20℃

Higher fracture energy represents the capacity of the material to absorb energy before failure. Fig. 23 shows that under monotonic tensile loading, the fibers at 5°C do not clearly show a boost to the energy required to fracture the specimen, and the effect of length and content appears to be irregular. This may be due to the fact that the interface zone between the fibers and the mastic has become a potential weak point in low temperature testing, a situation which could then lead to faster damage to the specimen and thus a reduction in the total energy.

However, the enhancement of fracture energy by the fibers at a temperature of 20 °C was shown to be effective, but only at a reed fiber content of 2%, which was not significant for the other two tested fiber contents. This is similar to the previous pattern for strength analysis. It is worth noting that although the 0.4mm fiber has a considerable increase in performance, the effect is slightly less than that of the longer fiber, probably due to the shorter lengths of fiber not being as integral to mastic which limits the ability to transmit forces.

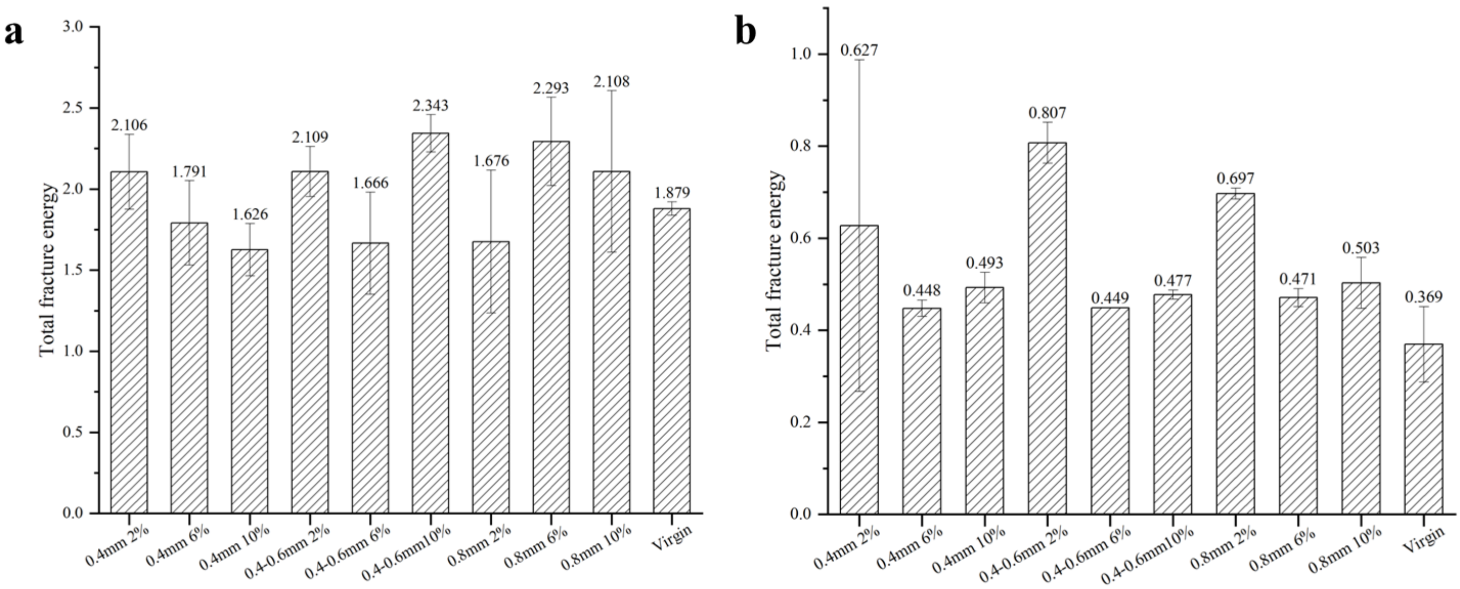


Fig. 23. Fracture energy at (a)5℃ ,(b)20℃

In conclusion, the monotonic tensile test was able to test the improvement in tensile strength brought about by the reed fibers. Analyzed in terms of peak forces, the effect at 5°C was less pronounced than at 20°C, probably because fiber-mastic adhesion is the weakest point in the specimen and the fibers are primarily responsible for the strength of the mixture at high temperatures. In this case, the adhesion is stronger than the cohesion of the matrix itself. The total amount of fracture energy is related to the stress and elongation of the sample under tension, implying the total energy required to break the sample. The combination of peak force, as well as fracture energy, suggests that the addition of 2% of 0.4-0.6 mm reed fibers is most effective in improving the tensile properties of the mortar.

Chapter 7 Ranking

Based on the tests discussed earlier, a simple ranking method is introduced to select the most applicable fiber amount and length to be added. First, the materials studied were ranked for each test. The highest-ranked material receives a ranking value (RV) of 10 and the last material is ranked at 1 RV. The total ranking value (TRV) for this material is then given by summing the RV for each binder. The results of RV and TRV of the RFMB and virgin bitumen are shown in Table 3.

Table 3 Ranking values of different RFMB and virgin bitumen

Parameters	**0.4mm 2%**	**0.4mm 6%**	**0.4mm 10%**	**0.4-0.6mm 2%**	**0.4-0.6mm 6%**	**0.4-0.6mm 10%**	**0.8mm 2%**	**0.8mm 6%**	**0.8mm 10%**	**Virgin**
Workability										
Viscosity	8	6	3	7	5	2	9	4	1	10
High Temperature Properties										
$J_{nr\ 0.1}$	2	5	8	3	6	10	4	7	9	1
$J_{nr\ 3.2}$	2	5	8	3	6	10	4	7	9	1
$J_{nr\ diff}$	9	7	5	8	3	1	6	4	2	10
Medium Temperature Properties										
LAS-Fracture energy	3	7	10	4	2	9	6	8	5	1
Fatigue life-5% strain	8	7	4	9	5	3	10	6	2	1
Fatigue life-2.5% strain	8	7	4	9	5	3	10	6	2	1
Cracking length at failure	3	6	8	1	2	5	4	7	9	10
Low temperature Properties										
Maximum Stress	9	7	1	5	6	2	4	8	3	10
Relaxation (T10)	9	6	4	10	7	3	8	5	2	1
Tensile strength of mortar										
Tensile strength of mortar-5℃	6	4	9	7	5	10	3	1	8	2
Tensile strength of mortar-20℃	8	2	3	10	4	5	9	6	7	1
Fracture energy-5℃	6	4	1	8	2	10	3	9	7	5
Fracture energy-20℃	8	2	6	10	3	5	9	4	7	1
Total ranking value	**89**	**75**	**74**	**94**	**61**	**78**	**89**	**82**	**73**	**55**

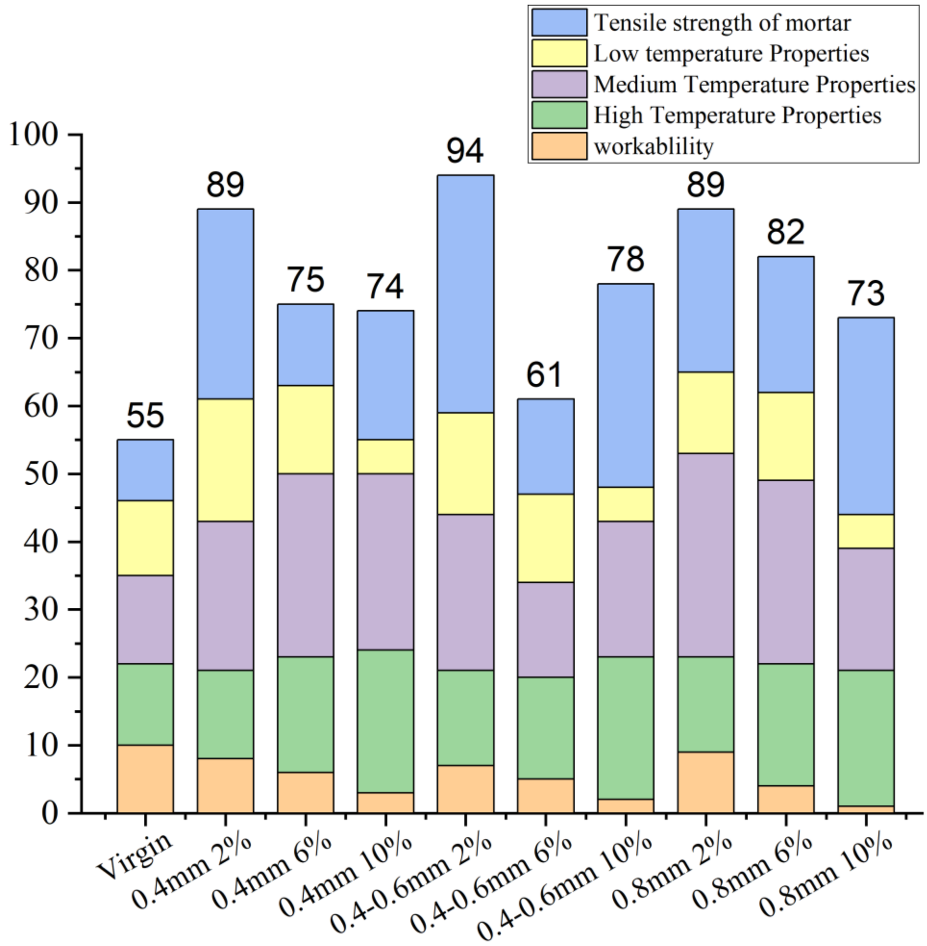


Fig. 24 Rank value of different RFMB and virgin bitumen

The various properties were categorized as low temperature, medium temperature, high temperature, workability, and tensile strength of mortar. According to Fig. 24, bitumen with 0.4-0.6 mm 2% reed fiber is optimal for overall performance. Besides, it can be seen that the reed-modified bitumen with the addition of reed fiber scores higher than virgin bitumen, indicating that reed fiber has an overall improvement in the performance of the bitumen. However, the modified bitumen with a 10% addition did not perform well at low temperatures, indicating that this amount of addition is not suitable for use in low-temperature areas.

Chapter 8 Conclusion

In this paper, reed fibers were used to modify bitumen and the effect of the fibers on the properties of the bitumen was investigated at different lengths and contents. The chemical characterization of fiber modification on bitumen was investigated, followed by rheological performance tests on modified bitumen with different fiber contents and fiber lengths and tensile strength tests on mortar made using modified bitumen to evaluate the performance of RFMB, and finally, the optimum combination of fiber length and content was selected. The main conclusions are as follows.

(1) The FTIR results show that the addition of fiber does not affect the chemical structure of the bitumen, but only the physical mixing. The large quantity of C-C stretch contained in the reed fiber affects the increase in peak area at this location in the RFMB as the fiber content increases and can be used as a basis for determining the fiber content.

(2) The viscosity of RFMB will increase with the increment of fiber content. Under the same content, the viscosity of RFMB will be larger with the use of longer fibers, but the influence is comparatively slight, which can reflect the influence of fiber length.

(3) Rheological tests show that the addition of reed fiber increases the complex shear modulus of the bitumen and improves its high-temperature performance, whilst having a negative effect on the low-temperature performance. This negative effect became more pronounced as the fiber content increased. In addition, RFMB also showed excellent fatigue and rutting resistance, but poor stress sensitivity.

(4) Monotonic tensile tests of the fiber-modified mortar showed that the addition of fibers had an inconsequential effect on its regularity, with only 0.4-0.6mm and 0.8mm showing noticeable improvement. Due to the short length of the 0.4mm reed fibers, the reinforcing effect on the bitumen is less visible. In addition, the resting step in the sample preparation process may have led to uneven deposition of sand and fiber in the mortar. More rational preparation steps and improvements to the sample model need to be investigated in subsequent experiments.

Further work

The influence of fibers on the adhesion of bitumen needs further discussion and verification of whether the relaxation time is related to the detachment of the bitumen sample from the plate. And the further experiment is required to investigate the effect of reed fiber on aged bitumen and asphalt mixtures.

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