

1 **Effects of Waterborne Epoxy Resin on the Fatigue Performance of Bitumen**  
2 **Emulsion**

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# Effects of Waterborne Epoxy Resin on the Fatigue Performance of Bitumen Emulsion

**Abstract:** Bitumen emulsion has been widely used in various paving applications, such as tack coat, surface treating, cold recycling, and cold mixes. However, bitumen emulsion faces the major concern of low mechanical strength, especially at high service temperatures. To improve the mechanical performance of bitumen emulsion, waterborne epoxy resin was used as a modifier in this study. The objective of this study was to evaluate the effects of waterborne epoxy resin on the fatigue performance of the bitumen emulsion residues. The waterborne epoxy resin modified bitumen emulsion residues were first subjected to pressure aging vessel (PAV) test. The fatigue performances were then measured using the stress-controlled time sweep test and linear amplitude (LAS) test. The results indicated that the fatigue life of the emulsion residues increased with the incorporation of waterborne epoxy resin from the time sweep test conducted at the stress levels from 300 kPa to 500 kPa. On the other hand, the LAS test suggested that the predicated fatigue life of the emulsion residues was strain dependent. The emulsion residues containing more waterborne epoxy resin demonstrated larger fatigue life at lower shear strains (<5%), while the reverse was true when the strain exceeded 5%.

**Keywords:** Bitumen emulsion; Waterborne epoxy resin; Rheological properties; Time sweep; Linear amplitude sweep

## 1. Introduction

Bitumen emulsion has been widely used in various paving applications, such as tack coat, pavement surface treatment, cold recycling, and cold mixes (Gingras et al. 2005). The viscosity of bitumen emulsion is low enough to achieve good workability at ambient temperature (Yuliestyan et al. 2017), reducing energy consumption and greenhouse gas emissions during manufacturing and construction. Therefore, bitumen emulsion is considered a more environment-friendly bonding material than hot-mix asphalt binder. However, bitumen emulsion faces the major concern of low mechanical strength, especially at high temperatures (Al Nageim et al.

2012; Gómez-Meijide and Pérez 2014). To improve the mechanical performance of bitumen emulsion, polymer latexes are commonly used as modifiers. Polymer latexes can be mixed with bitumen emulsion easily due to their liquid form. In addition, the latex modification generally leads to improved performance of bitumen emulsions, such as higher resistance to load deformation and reduced thermal cracking (Ruggles 2005). Polymer latexes, such as styrene-butadiene-styrene (SBR) latex (Khadivar and Kavussi 2013) and natural rubber latex (Jiang et al. 2020), are commonly used. More recently, waterborne epoxy resin has emerged as a novel waterborne modifier for bitumen emulsion, which showed significant effects in improving the performance of the bitumen emulsion (Zhang, He, and Ao 2007; Hu et al. 2019; Li et al. 2019). The rheological properties, microstructure, interface bonding strength with aggregate, and resistance to moisture and long-term aging of waterborne epoxy resin modified bitumen emulsion have been extensively studied in previous studies (Li et al. 2019; Li, Leng, Wang, et al. 2021; Li, Leng, Partl, et al. 2021). However, the effect of waterborne epoxy resin on the fatigue performance of bitumen emulsion residues has not been well understood.

Fatigue damage represents one of the major distresses that appear in asphalt pavements, due to repeated vehicle loading and weathering. Because of the large difference in stiffness between binder and aggregate in asphalt mixture, most of the bulk strain is concentrated in the binder domain (Bahia et al. 1999). Thus, the fatigue behavior of the asphalt mixture is primarily determined by the binders (Bahia et al. 2001). Various testing methods have been developed to evaluate the fatigue performance of bituminous materials. Bahia et al. (2001) proposed the repeated cyclic time sweep test using the dynamic shear rheometer (DSR), with stress-controlled or strain-controlled mode, to characterize the fatigue behavior of the binders. It is found that the damage accumulates faster in the stress-controlled time sweep than that of the strain-controlled

time sweep. There is apparent failure at the end of the stress-controlled test, which is not observed for the strain-controlled mode.

The criteria for determining the fatigue life using the time sweep test are somewhat arbitrary. The so-called dissipated energy concept was then proposed to evaluate the fatigue life as an alternative (Van Dijk 1975; Van Dijk and W 1977). Dissipated energy is the energy lost in cyclic shear test, due to mechanical work, heat generation, and damage done to the material (Ghuzlan and Carpenter 2006). Previous studies assumed that all the dissipated energy contributes to the fatigue damage of the material, which is not exactly correct, as only part of the dissipated energy is responsible for the damage done to the material (Ghuzlan and Carpenter 2000; Shen et al. 2006). Ghuzlan and Carpenter (2000) found that only the increment of the dissipated energy of one loading cycle compared with the previous loading cycle would produce further damage. Thus, the ratio of dissipated energy change (RDEC), defined as the relative dissipated energy of two consecutive loading cycles, was adopted as a parameter to represent fatigue damage (Ghuzlan and Carpenter 2000). Moreover, RDEC is independent of the loading mode, i.e., stress-controlled or strain-controlled, which provides a more fundamental understanding of the fatigue damage. The failure is defined as the number of loading cycles at which the value of RDEC starts to rise rapidly. It is believed that significant damage has been done to the material at this point.

The dissipated energy  $W_i$  is calculated from the following equation,

$$W_i = \pi \sigma_i \varepsilon_i \sin \delta_i \quad (1)$$

where  $W_i$  is the dissipated energy at load cycle  $i$ ,  $\sigma_i$  is the stress amplitude at load cycle  $i$ ,  $\varepsilon_i$  is the strain amplitude at load cycle  $i$ , and  $\delta_i$  is the phase angle at load cycle  $i$ .

RDEC is obtained from Eq. (2).

$$RDEC = \frac{DE_n - DE_m}{DE_m(n - m)} \quad (2)$$

where  $DE_m$  and  $DE_n$  represent the dissipated energy at load cycle  $m$  and  $n$ , respectively.

The time sweep test is an effective method to evaluate the fatigue life of bituminous materials. However, this method is very time-consuming. In contrast, the linear amplitude sweep (LAS) test, developed by Bahia et al. (Johnson and Bahia 2010), represents an accelerated fatigue test based on the viscoelastic continuum damage (VECD) theory. It is found that the LAS test correlated fairly well with the pavement long-term fatigue performance (Hintz et al. 2011). The LAS test has been adopted to study the fatigue performance of bituminous materials by many researchers in recent years (Hintz et al. 2011; Sabouri, Mirzaiyan, and Moniri 2018; Cao and Wang 2018; Notani et al. 2019).

This study set out to evaluate the fatigue performances of waterborne epoxy bitumen emulsion residues (WEBERs). The rheological behavior of bitumen emulsion residues containing different amounts of waterborne epoxy resin was first measured using the dynamic shear rheometer (DSR). Pressure ageing vessel (PAV) test on all the WEBERs was then performed. Both the stress-controlled time sweep test and the LAS test were applied to characterize the fatigue performance of the WEBERs.

## **2. Materials and methods**

### ***2.1 Materials preparation***

A base bitumen with a penetration grade of 60/70 (Pen 60/70), commonly used in Hong Kong,

was used in this study. **Table 1** demonstrates the basic properties of the bitumen. The cationic slow setting emulsifier was kindly provided by Ingevity under the trade name of INDULIN® W-5. Bitumen emulsion with a solid content of 60% was first prepared with a laboratory colloid mill. Waterborne epoxy resin was then prepared following the procedure detailed in our previous studies (Li et al. 2019; Li et al. 2022). The waterborne epoxy resin was subsequently mixed with bitumen emulsion at three mass percentages, 1wt%, 3wt%, and 5wt%. At last, the waterborne epoxy resin modified bitumen emulsions were conditioned at ambient room temperature for three days, followed by 24 h of oven conditioning at 60 °C. This procedure ensures the fully curing of the waterborne epoxy resin, and the cured WEBERs were then obtained, denoted as WEBER-1, WEBER-3, and WEBER-5. The bitumen emulsion residue without waterborne epoxy resin was used as control and denoted as WEBER-0.

**Table 1.** Properties of base bitumen

Property		Value	Specification
Penetration (25 °C, 0.1 mm)		64.5	ASTM D5
Softening point (°C)		48.5	ASTM D36
Viscosity at 135 °C (mPa·s)		477.5	ASTM D4402
Ductility at 25 °C (cm)		78.5	ASTM D113
SARA fraction	Saturate	17.58 wt %	ASTM D4124
	Aromatic	46.85 wt %	
	Resin	26.20 wt %	
	Asphaltene	9.37 wt %	

## 2.2 Experiments

### *PAV aging of WEBERs*

Since bitumen emulsions are generally applied at ambient temperature without heating, no short-

term aging is experienced. Thus, the PAV aging was performed directly on the WEBERs according to ASTM D6521 to simulate the long-term in-service aging.

#### *Frequency sweep test*

Bulk rheological properties of WEBERs were measured employing a dynamic shear rheometer (DSR, MCR 702, Anton Paar). Frequency sweeps over the range of 0.1-30 Hz from -10 °C to 60 °C at an interval of 10 °C were conducted within the linear viscoelastic domain on all the specimens. The black diagrams and Cole-Cole plots were then constructed to evaluate the rheological properties of WEBERs. One advantage of the black diagrams and Cole-Cole plots is that they are independent of temperature or frequency, which enables one to analyze the rheological properties of bituminous materials without the application of the Time-Temperature Superposition (TTS) principle (Airey 2002).

#### *Time sweep fatigue test*

The fatigue tests were conducted using the DSR with an 8 mm diameter parallel plates and a 2 mm gap. All the fatigue tests were performed with the PAV-aged WEBERs at 20 °C. For the time sweep tests, three stress levels were evaluated, i.e., 300 kPa, 400 kPa, and 500 kPa, at a frequency of 10 Hz. The time sweep tests were continued until complete failure of the materials.

#### *Linear amplitude sweep test*

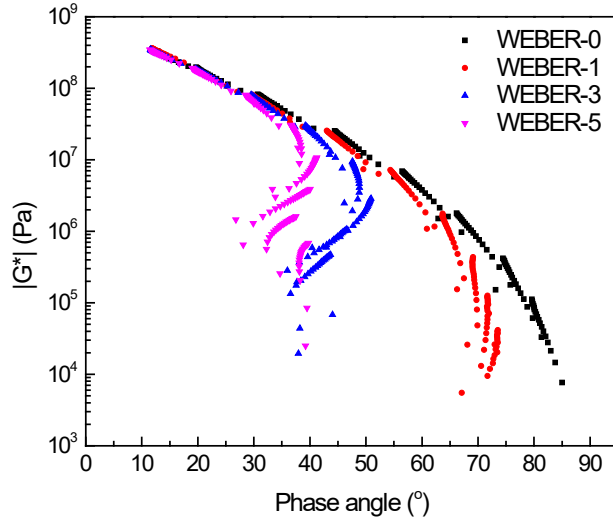
The LAS test was conducted at 20 °C following AASHTO TP-101. During the LAS test, the frequency sweep from 0.2 Hz to 30 Hz was first applied at a constant shear strain of 0.1% to capture the material's response in the linear viscoelastic domain, followed by the amplitude sweep from 0.1% to 30% at the frequency of 10 Hz.

### 3. Results and Discussion

#### 3.1 Rheological properties

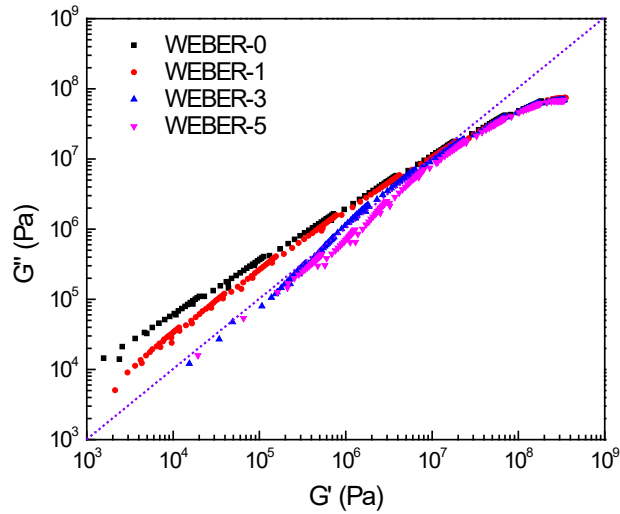
**Figure 1** shows the black diagrams of different WEBERs. The control emulsion residue, WEBER-0, showed a smooth transition from viscoelastic solid to viscoelastic fluid. It is noted that the phase angle of WEBER-0 approached close to  $90^\circ$  at  $60^\circ\text{C}$ , demonstrating an essentially Newtonian fluid response. On the other hand, with the increase of the waterborne epoxy resin contents, the phase angle decreases compared with WEBER-0, indicating a more elastic component for the cured emulsion residues containing waterborne epoxy resin. What stands out in the figure is that the phase angles of WEBER-3 and WEBER-5 first increased to approximately  $50^\circ$  and  $40^\circ$ , respectively, and then decreased. The appearance of the plateau is usually associated with the formation of interconnected polymer structures in bituminous materials (Polacco et al. 2006; Xia et al. 2016). At very low temperatures, the bitumen is very stiff, and the overall performance of the WEBERs is dominated by bitumen. Upon increasing the temperature, both the bitumen and the polymer structure softened to some extent, thus the phase angle increased. With further increasing of the temperature, the bitumen transfers to a fluid state, and the overall performance of the mixture is dominated by the epoxy resin networks. Therefore, the phase angle decreased at higher temperatures. This results clearly indicate that the addition of waterborne epoxy resin significantly improved the elasticity of the emulsion residues.





**Figure 1.** Black diagram

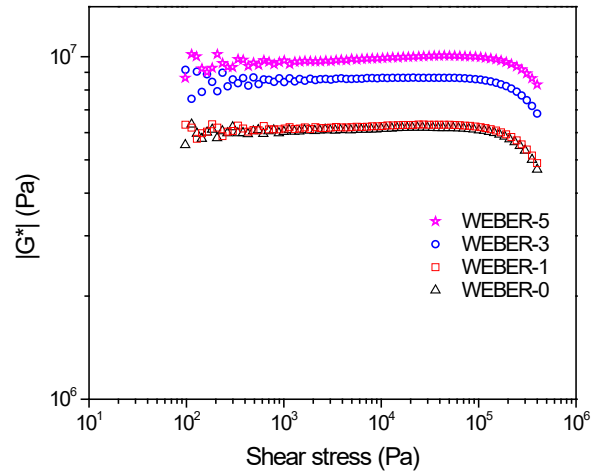
The Cole-Cole plot shows the contributions of elastic and viscous components to the overall stiffness of bituminous materials. A line at 45° angle in Cole-Cole plot is drawn to compare the storage modulus  $G'$  and loss modulus  $G''$ . The materials above the line indicate viscous-dominant behavior, while those below this line demonstrate elastic-dominant behavior (Loderer, Partl, and Poulikakos 2018). **Figure 2** depicts that different WEBERs display different viscoelastic behaviors. The WEBERs containing more waterborne epoxy resin show a greater elastic component than the control emulsion residue. Specifically, the elastic modulus of WEBER-3 and WEBER-5 is larger or equal to the viscous modulus at all the studied temperatures, indicating that these two emulsion residues demonstrate elastic-dominant behavior at all the temperatures in consideration. The increase of elasticity can be attributed to the interconnected epoxy resin structure in the cured WEBERs (Li, Leng, Wang, et al. 2021).



**Figure 2.** Cole-Cole plot

### ***3.2 Time sweep fatigue test***

The stress sweep tests were first conducted on the four emulsion residues at 20 °C, and the results are presented in **Figure 3**. The complex modulus  $|G^*|$  was unstable at the initial stage because of the very small stress, which then remained almost unchanged until the shear stress reached around 100 kPa. With further increase of the shear stress,  $|G^*|$  decreased rapidly, indicating that the response of the materials has become non-linear. The time sweep fatigue test was then conducted at stress levels larger than 100 kPa. It was found that the test would last for too long if the stress was smaller than 200 kPa. Thus, the stress levels were determined to be 300 kPa, 400 kPa, and 500 kPa.



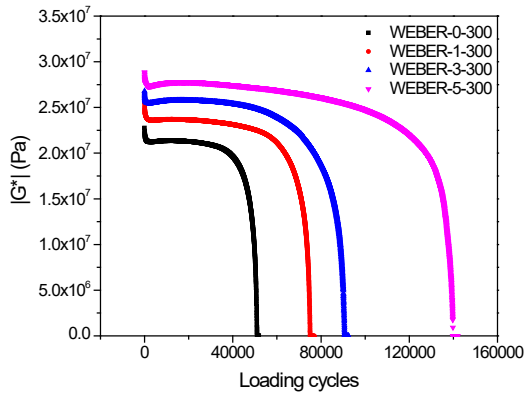
**Figure 3.** Stress sweep at 20 °C

**Figure 4** depicts the time sweep test results at 300 kPa, 400 kPa, and 500 kPa, respectively. The initial shear strain values (the average strain of the first 100 loading cycles) are presented in **Table 2** for comparison. It is clear that the initial strain decreased with larger dosages of waterborne epoxy resin at each stress level, because of the increase of the modulus. A typical three-stage fatigue damage is observed in **Figure 4**. At the initial stage, the modulus decreased to some degree because of instability. In the second stage, the modulus declined at a rate that is almost constant, and the damage accumulated gradually at this stage. The last stage shows that the modulus dropped sharply, indicating a complete fracture of the materials. It is observed that the loading cycles increased apparently with increasing waterborne epoxy resin dosages at all three stress levels.

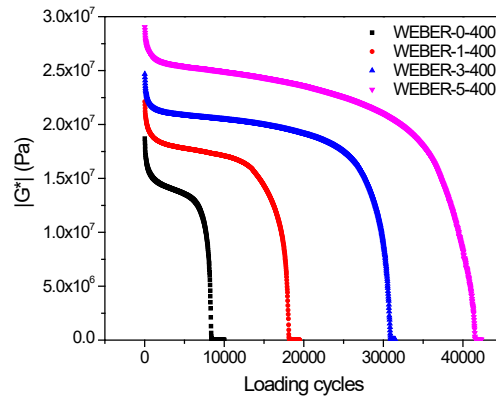
**Table 2.** Initial shear strain for the time sweep fatigue tests

Sample ID	Initial strain (%)		
	300 kPa	400 kPa	500 kPa
WEBER-0	1.55	2.48	3.04
WEBER-1	1.19	2.22	3.01

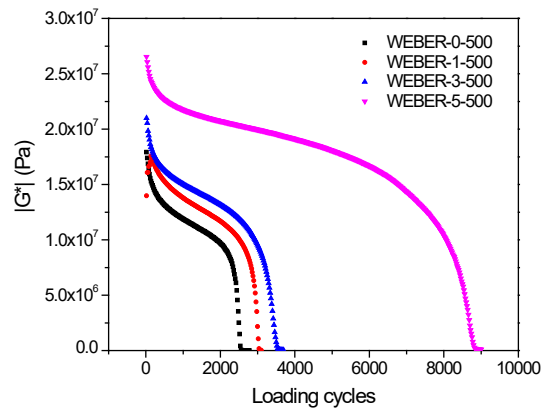
WEBER-3	1.15	2.20	2.53
WEBER-5	1.05	1.79	2.00



(a)



(b)

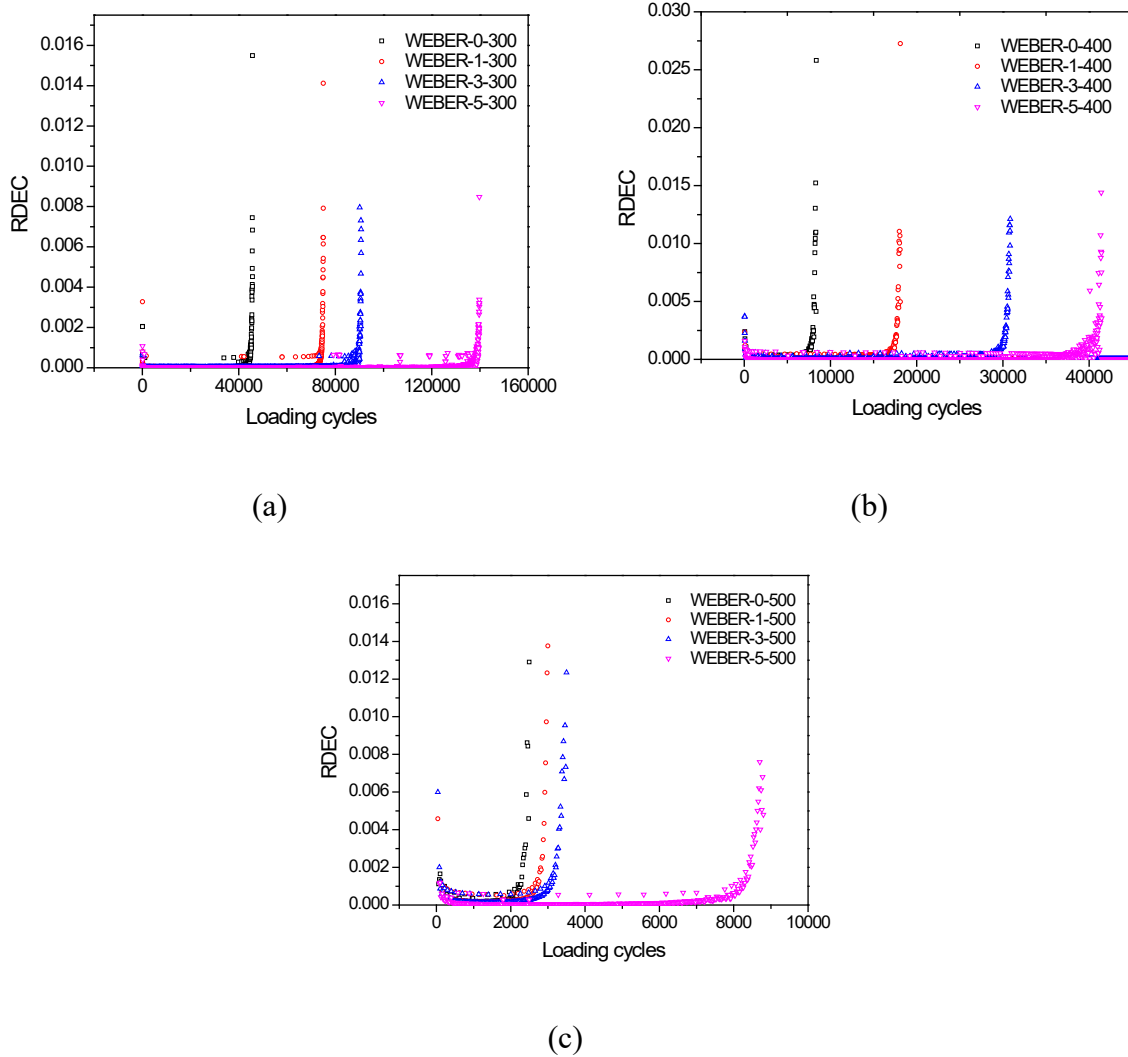


(c)

**Figure 4.** Stress-controlled time sweep tests at the stress level of (a) 300 kPa, (b) 400 kPa, and (c) 500 kPa

**Figure 5** presents the RDEC plotted against the loading cycles. It can be seen that the RDEC first decreased, and then kept constant in the second stage, which increased rapidly in the final stage. It clearly shows that a larger percentage of the dissipated energy was converted into

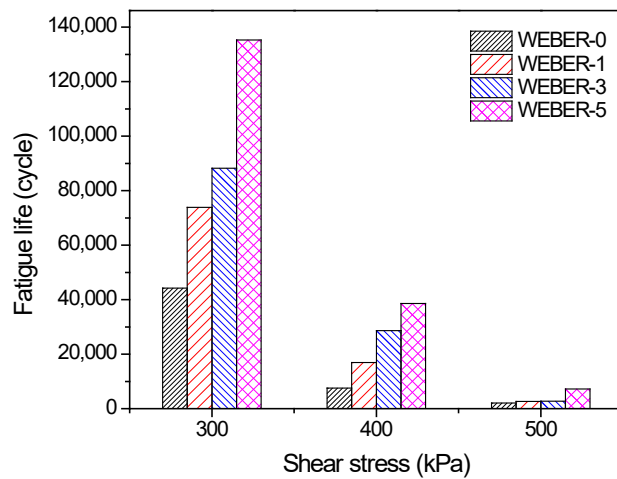
damage to the material at the early stage. The damage done each cycle then decreased to a lower ratio and stabilized in the second stage. At the last stage, the damage rate increased sharply, as the material was completely fractured.



**Figure 5.** Rate of dissipated energy change (RDEC) at the stress level of (a) 300 kPa, (b) 400 kPa, and (c) 500 kPa

The fatigue loading cycles of all the residues at the three stress levels are summarized in **Figure 6**. It is clear that the fatigue life of WEBERs decreased dramatically with increasing shear stress. On the other hand, it is noticeable that the fatigue life become larger as the increase

of the waterborne epoxy resin contents at all the three tested stress levels, indicating the epoxy resin networks increased the emulsion residues resistance to fatigue damage. It is worth mentioning that the fatigue life of WEBERs at 500 kPa did not show a significant increase by waterborne epoxy resin due to the large stress magnitude. The incorporation of waterborne epoxy resin formed the inter-connected polymer structure, and the bitumen phase is encapsulated by the epoxy resin film (Li, Leng, Wang, et al. 2021), which increased the material's resistance to damage. Thus, the fatigue life of the emulsion residues containing waterborne epoxy resin increased compared with the pure bitumen emulsion residue in the tested stress levels.

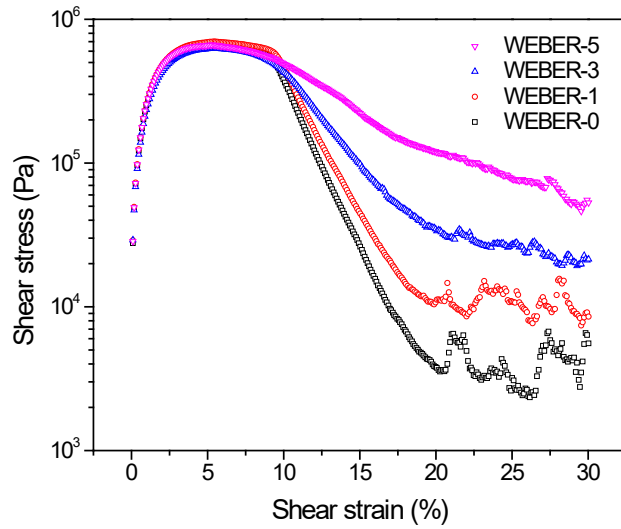


**Figure 6.** Fatigue life at different stress levels

### ***3.3 Linear amplitude sweep test***

Despite many studies have employed the RDEC approach to evaluate the fatigue behavior of bituminous binders and mixes (Shen et al. 2006; Pitawala et al. 2019). It is still limited regarding the time-consuming of the test. The LAS test was then used to evaluate the fatigue life of the emulsion residues. Based on R.A.Schapery's work on crack growth of viscoelastic materials (Schapery 1984), the viscoelastic continuum damage (VECD) concept was adopted to evaluate the fatigue performance of WEBERs.

**Figure 7** shows the relationships between the shear stress and strain for different WEBERs. It can be seen that the shear stresses first increased rapidly at the lower shear strain before reaching the peak, and then declined progressively with further increase of the strain amplitude. What stands out in the figure is the significant difference regarding the stress drop rate in the large strain region (>10 %) for different emulsion residues. It is clear that larger dosages of waterborne epoxy resin led to lower drop rate of the stress, indicating that the emulsion residues become more robust to damage with the formation of the epoxy resin polymer networks.



**Figure 7.** Stress-strain curves of different WEBERs (20 °C)

The fatigue life  $N_f$  of WEBERs can then be calculated based on the VECD theory, which is summarized in the form in Eq. (3).

$$N_f = A (\gamma_{max})^{-B} \quad (3)$$

where  $A$  and  $B$  are fitting parameters obtained from the VECD model, and  $\gamma_{max}$  is the shear strain. The calculating procedure is detailed in the standard AASHTO TP-101.

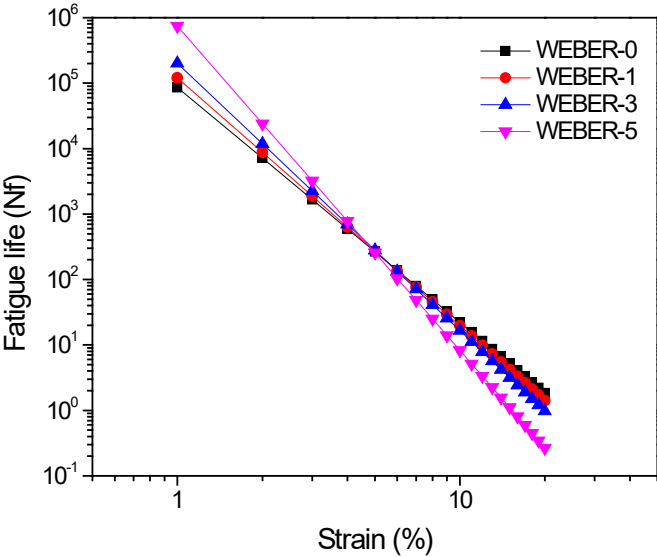
**Table 3** presents the predicted fatigue lives ( $N_f$ ) at the strain levels of 2.5% and 5% based on the VECD model. It can be seen that the increase of fatigue life for WEBERs with larger concentration of waterborne epoxy resin was more significant at the lower strain level (2.5%). The predicated fatigue life of WEBER-5 is more than double that of the WEBER-0 at 2.5% of strain. However, it should be pointed out that at the higher strain of 5%, the predicated fatigue life for the different WEBERs become very close. The predicted fatigue life versus shear strain (1%~20%) is depicted in **Figure 8**. Based on the VECD model, the emulsion residues containing waterborne epoxy resin had higher fatigue life compared with the pure bitumen emulsion residue at lower shear strain (<5%). On the other hand, the trend reversed when the shear strain became larger than 5%. The reason may be that the emulsion residues with waterborne epoxy resin forms a honeycomb-like composite structure (Li, Leng, Wang, et al. 2021), such structure is stronger than pure bitumen and is more robust under relatively small strain. However, the epoxy resin polymer structure is also stiffer and more rigid compared with bitumen, thus it is more sensitive to larger strain. Therefore, it is concluded that the WEBERs containing waterborne epoxy are more resistant to fatigue under smaller strain, while they are more sensitive to larger strain amplitude compared with the pure bitumen emulsion residue. It should be noted that such large strains (>5%) are normally not expected in asphalt pavement, except for extreme conditions. As a result, it is inferred that the waterborne epoxy resin can increase the resistance to fatigue damage of bitumen emulsion residues in normal conditions.

**Table 3.** Fatigue life prediction based on VECD model

Sample ID	A	B	$N_{f2.5\%}$	$N_{f5.0\%}$
WEBER-0	85717.9	3.588	3202	266
WEBER-1	112330.5	3.784	3506	255



WEBER-3	200624.5	4.083	4758	281
WEBER-5	747132.9	4.955	7972	257



**Figure 8.** Fatigue life predication at different strain

#### 4. Conclusions

To understand the effect of waterborne epoxy resin on the fatigue performance of waterborne epoxy bitumen emulsion residues (WEBERs), frequency sweep test, stress-controlled time sweep and linear amplitude sweep (LAS) tests were conducted. The following conclusions can be drawn from the results of this study,

- The WEBERs become more elastic with larger concentrations of waterborne epoxy resin.
- In all the time sweep tests at 300 kPa, 400 kPa, and 500 kPa, the fatigue life of the WEBERs increased with larger contents of waterborne epoxy resin.
- From the LAS tests, it can be concluded that the fatigue life of WEBERs containing waterborne epoxy resin was larger than that of the pure bitumen emulsion residue when

the strain amplitude was lower than 5%. In contrast, the reverse was true when the strain amplitude becomes larger than 5%.

It should be noted that the large strains (>5%) are normally not expected in asphalt pavement, except for extreme conditions. As a result, it is inferred that the waterborne epoxy resin can increase the resistance to fatigue damage of bitumen emulsion residues in normal conditions.

### **Acknowledgment**

The authors would like to acknowledge the sponsorship of the Hong Kong Innovation and Technology Commission through the Guangdong-Hong Kong Technology Cooperation Funding Scheme (TCFS) project GHP/116/18GD.

### **Data availability statement**

The data that support the findings of this study are available upon reasonable request.

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