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A comparative study of the induction healing behaviors of hot and warm mix asphalt

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HIGHLIGHTS

- Induction heating can greatly enhance the self-healing ability of WMA.
- Induction heating shows significant gradient heating characteristics.
- The optimal heating temperature of WMA is lower than that of WMA.
- The healing of asphalt mixture is highly strain-dependent.

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ABSTRACT

In this paper, the induction healing behaviors of both warm mix asphalt and hot mix asphalt were investigated and compared through damage-healing-damage test. The steel fibers used for making asphalt mixture conductive were first optimized according to the thermal conductivity and induction heating speed of the mixture. Then, the gradient induction heating characteristics, cracking and fatigue resistance, and self-healing ratios of both warm mix asphalt and hot mix asphalt were studied. It is found that warm mix asphalt and hot mix asphalt have similar induction heating speed. The Aspha-Min based warm mix asphalt shows slightly higher fatigue resistance but slightly lower self-healing ratios than hot mix asphalt mixture. Both warm and hot mix asphalt mixtures have very high fatigue life ratios with induction heating, which means that induction heating can improve the durability of WMA. The optimal induction heating temperature of warm mix asphalt is slight lower than that of hot mix asphalt due to the remaining active ingredient, which also deceases the healing ratio slightly. It is also found that the healing ratios of both warm and hot mix asphalt mixtures are highly strain-dependent and the mixtures obtained best healing behaviors at medium microstrain amplitudes.

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1. Introduction

It is well known that asphalt concrete can repair the damage inside and recover its strength and fatigue life autonomously during rest periods. The self-healing phenomenon of asphalt concrete was first reported in the 1960s [1]. Since then, self-healing of asphalt mixtures has always been a hot research topic. These researches involved in demonstrating the self-sealing characteristics of asphalt concrete with both laboratory experiments and field measurement, explaining the mechanisms of self-healing of asphalt concrete, investigating the factors influencing the self-healing rate of asphalt concrete and exploring the self-healing techniques to promote healing [2–10]. A lot of researches have demonstrated that temperature is the dominant factor influencing the self-healing properties of asphalt concrete: an increase in the test temperature not only increases the self-healing rate but also shortens the total time needed for full healing [7–10]. So, it is feasible to enhance the self-healing capacity and speed of asphalt concrete by increasing its temperature.

To enhance the self-healing capacity of asphalt concrete through increasing the temperature, an induction heating approach was developed by researchers at Delft University of Technology. Steel fibers were added to asphalt mixtures and induction heating was applied to increase the healing capacity of asphalt concrete when cracks occurred in asphalt mastic [11–21]. As shown in Fig. 1, Garcia and Liu et al. found that induction heating can greatly increase the self-healing properties of porous asphalt

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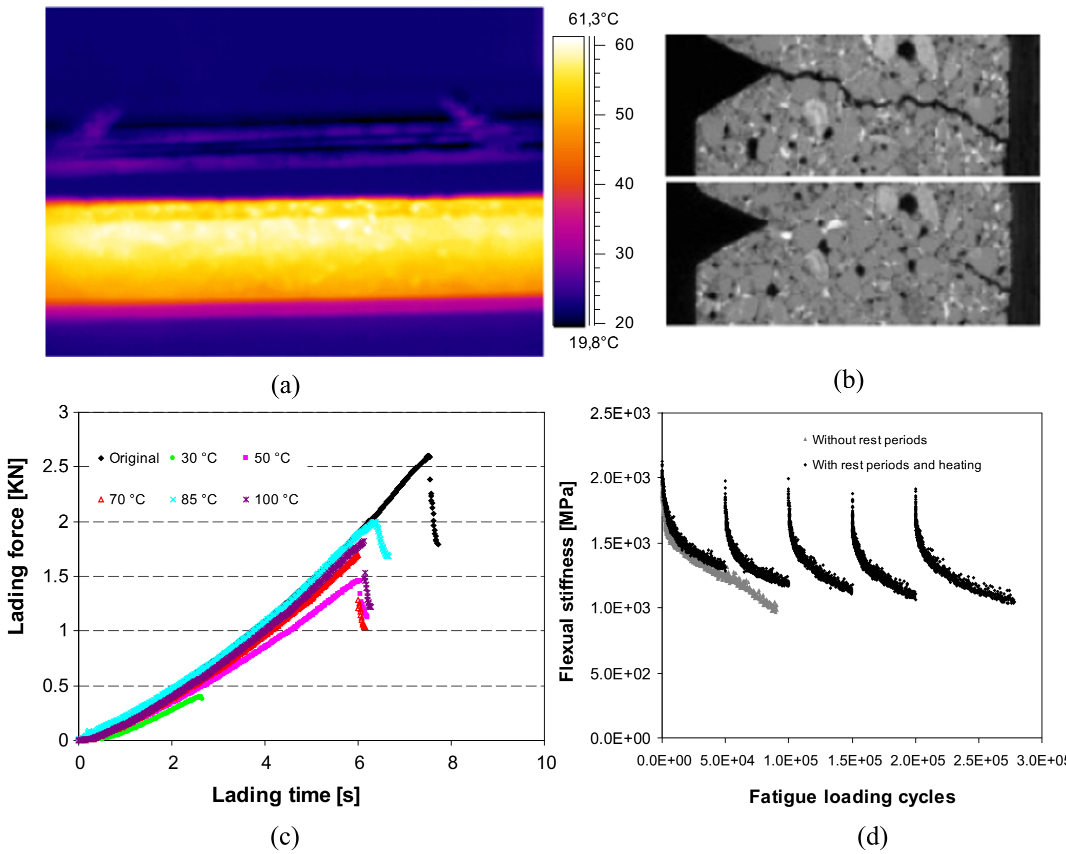

Fig. 1. Self-healing of asphalt concrete with induction heating, (a) induction heating of asphalt concrete containing steel wool fibers, (b) cracking closure due to induction heating, (c) strength recovery due to induction heating, (d) fatigue life extension due to induction heating [16–18].

concrete by closing the cracks and increasing its recovered flexural strength ratio and fatigue extension ratio [16–18]. It also has been proven that induction heating can prevent the raveling of porous asphalt concrete efficiently [19]. Garcia found that induction heating didn't aggravate the ageing of the bitumen due to a very short heating time [20]. Dai et al. reported that induction heating can be repeated for at least 6 times when cracks returned [21].

The first trial section with the induction heating concept was constructed on highway A58 near Vlissingen in the Netherlands in 2010 and the first onsite induction heating treatment was applied on it in June 2015 [22]. According to NL Agency, the Netherlands can save about 90 million euro annually by investing in self-healing asphalt concrete with a 50% extended life span and twice of the price compared with standard asphalt concrete [23]. And it is estimated that China will save a maintenance expense of 1000 billion RMB in the life span by applying self-healing concrete in 10% of its asphalt pavement. Thus, induction healing asphalt concrete, being a smart material and an advanced maintenance concept for asphalt pavement, is promising to greatly improve the quality of the pavement, reduce the maintenance activities and extend its service life.

In recent years, many technologies have been developed to reduce the temperature of production and application of asphalt concrete on site, by using the so-called Warm Mix Asphalt (WMA). Several patents for Warm Mix Asphalt have been developed based on foaming techniques and/or organic/wax additives and/or chemical additives [24,25]. It allows significant lowering of the production and paving temperature by 20–30 °C, through reducing the viscosity of bitumen and/or increasing the workability of mixture. This leads to enormous environmental benefits: (1) Warm-mix asphalt provides a reduction of 24% on the air pollution impact of Hot Mix Asphalt (HMA) and a reduction of 18% on fossil fuel consumption. It also reduces smog formation by 10%. (2) WMA is estimated to provide a reduction of 15% on the environment impacts of HMA [24]. Despite the promising performance, WMA has not yet gained full acceptance in asphalt industry, mainly because a large number of questions, especially about the quality control and durability, need still to be answered. The main Drawback of WMA is the reduced long-time performance related to moisture, cracking and low temperature behavior. In order to reach widespread implementation it is necessary to prove that WMA can compete with the performance of the well-established hot mix asphalt.

The purpose of this paper is to study the self-healing behavior of WMA with induction heating and to prove that induction heating can be used to improve the durability of WMA by healing the crack. For that, the induction heating speed, fatigue resistance and induction healing behaviors of WMA were studied. The properties of HMA were studied as well for comparison purpose.

2. Experimental

2.1. Materials

The HMA used in this research is standard asphalt mixture AC-16 containing 6% steel fiber and the gradation of the AC-16 mixture is shown in Table 1. Penetration grade bitumen with penetration 63.4 (0.1 mm) and softening point 45.1 °C was used and the bitumen content is 5.2%. Three types of short steel fibers supplied by Shanghai Auticar-yiher Metal Products Co. Ltd were added into the mixture to make it conductive and suitable for induction heating. The properties of the steel fibers are shown in

Table 1
Gradation of the WMA and HMA.

Sieve size (mm)	19	16	13.2	9.5	4.75	2.36	1.18	0.6	0.3	0.15	0.075
Passing (%)	99.9	96.8	90.4	75.2	49.9	28.7	22.2	18.0	10.8	7.2	5.3

Table 2
Properties of the steel fibers used in this research.

Type of fiber	Diameter (μm)	Average length (mm)	Density (g/cm^3)
A02	40–60	3.5	7.8
A08	20–50	3.3	7.8
Q1-80	70–130	4.2	7.8

Table 2. The WMA used in this research consisted of standard asphalt mixture AC-16, 6% steel fiber (volume content of bitumen, optimized in previous research) and 6% warm mix additive (mass ratio of bitumen, recommended by the manufacturer).

A kind of Aspha-Min warm mix additive XT-W3 supplied by Changzhou XinTuo Modified Pavement Materials Co., Ltd. was used in this research. The warm mix additive contained 20% crystalliferous water, which will be released when the temperature is above 80 °C. Due to the release of the crystalliferous water, the bitumen will be foamed and the mixing temperature for the mixture will be decreased by 30 °C. One special benefit of XT-W3 is that it can be directly added into the mixture during mixing. In this research, the HMA was mixed at 160 °C, while the WMA was mixed at 130 °C. Both Marshal Samples (Φ101.6 mm × 63.5 mm) and beam samples (250 mm × 35 mm × 30 mm, 380 mm × 63.5 mm × 50 mm) were prepared for the subsequent research.

2.2. Thermal characteristics measurement

The thermal properties of asphalt concrete with different steel fibers were measured by a Thermal Constants Analyzer (TPS 2500S, Hot Disk, Sweden). In this method, the TPS element behaves both as a temperature sensor and as a heat source. The TPS element consists of an electrical conducting pattern of thin nickel foil (10 μm thick) in the form of a double spiral, which resembles a hot disk, embedded in an insulating layer made of Kapton (70 μm thick) [Fig. 2(a)]. Fig. 2 illustrates the schematic representation of the thermal properties measurement for asphalt concrete samples.

Marshall Sample were cut in half and ground with 1200# silicon carbide paper to obtain plain surfaces. The pieces of the Marshall sample were located in contact with the two surfaces of the sensor, which is connected to the thermal constant analyzer. The test was performed at 20 °C with a recommended power 0.17 W and testing duration 40 s. The temperature of the thin nickel foil increases slightly when current flows in it, and the heat generated transfers to the asphalt pieces. The thermal diffusivity is dependent on the heat conduction characteristics of the asphalt concrete samples. According to the temperature and the response time of the sensor recorded, the thermal conductivity and the thermal diffusivity can be obtained by the mathematical model. And the heat specific is calculated by the following equation:

$$c_v = \lambda/\alpha \quad (1)$$

where, c_v is the specific heat in MJ/(m^{3}K), λ is the thermal conductivity in W/(mK), α is the thermal diffusivity in mm^2/s. The effect of different types of steel fibers on the thermal conductivity, thermal diffusivity and specific heat were investigated. In this study, four repetitions were tested for each sample as shown in Fig. 2(b) and the average values were used for the analysis.

2.3. Induction heating test

The objective of induction heating test is to study the heating efficiency of the both WMA and HMA samples (63.5 mm thick). The induction heating experiments were performed by using an induction heating system with a capacity of 7.9 kW and at a frequency of 123 kHz (Fig. 3). An infrared camera was used to measure the heating speed of the sample. The distance between the self-developed coil of the induction heating machine and the top surface of the sample was varied from 30 mm to 20 mm and 10 mm to study its effect on the heating speed of the sample. The strength of the magnetic field varies with the distance

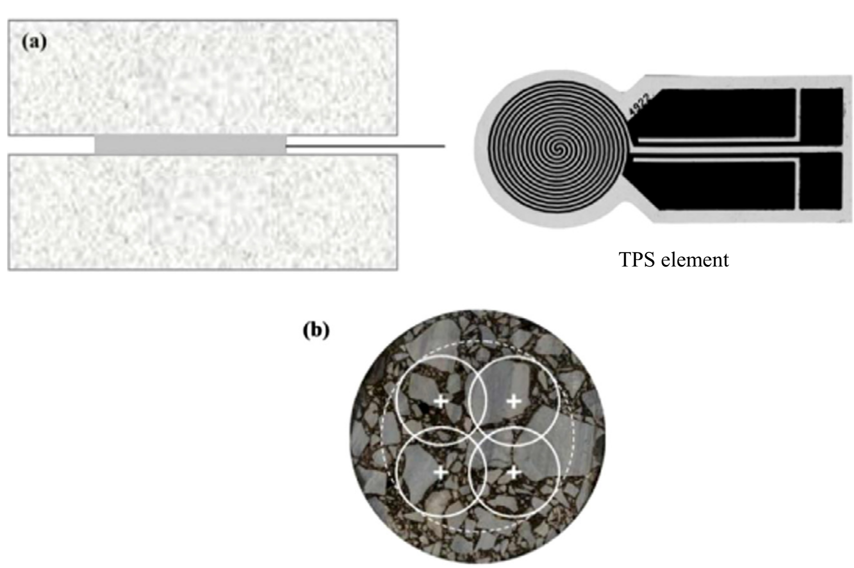

Fig. 2. Schematic representation of the thermal properties measurement for asphalt concrete samples.

Fig. 3. Setup of induction heating test.

between the coil and the sample, which will result in different heating speeds through the samples' depth. Three samples were induction heated in each case. To better understand this heating gradient, the average induction heating speeds at the top surface, middle and bottom of the sample were measured.

2.4. Three point bending test

The three point bending test was performed with a Universal Testing Machine (UTM) at −10 °C on asphalt beams (250 mm × 35 mm × 30 mm). The flexural tensile strength and strain were determined by applying a displacement at a rate of 50 mm/min until the peak load was reached. Three samples of each mixture were tested and the average value was obtained. After the test, the flexural tensile strength at failure and the maximum flexural tensile strain could be calculated using Eqs. (2) and (3).

$$R_B = \frac{3LP_B}{2bh^2} \quad (2)$$

$$\varepsilon_B = \frac{6hd}{L^2} \quad (3)$$

where R_B is the flexural tensile strength at failure in MPa, ε_B is the maximum flexural tensile strain, d is maximum deflection of the center of the beam in mm, P_B is the peak load in MPa, L is the length of the support span in mm, b is the width of the sample in mm, h is the thickness of the sample in mm.

The fractured beams during three point bending test were induction heated to different temperatures for healing and fractured again to test the recovered fracture strength. The strength recovery ratio (the recovered fracture strength divided by the initial fracture strength) was used to characterize the healing capacity of the mixture.

2.5. Four point bending fatigue test

Before starting the fatigue-healing test, the fatigue resistances of warm and hot mix asphalt were first compared in four-point bending fatigue test. The test was done at 15 °C with a frequency of 10 Hz on beam samples (380 mm × 63.5 mm × 50 mm) according the Chinese Specification JTG E20-2011. The initial value of the calculated modulus was calculated from the measured values for force, displacement and phase lag after the hundredth cycle (n = 100). The fatigue test was continued until the calculated modulus dropped to half its initial value. After testing the fatigue life of the beams at 5 strain amplitudes (250-400-600-800-1000 microstrain), the fatigue lines of the beams were determined according to the following equation:

$$N_f = a\varepsilon^{-b} \quad (4)$$

where, N_f is the number of loading cycles to fatigue, ε is the microstrain amplitude used in fatigue testing, a and b are fatigue constants.

2.6. Healing test procedure in four point bending

The experimental method of testing the induction heating activated healing of fatigue damage in asphalt beams consisted of three steps: (1) fatigue testing at 15 °C and 10 Hz was performed on asphalt beams until the complex modulus dropped to half of its initial value; (2) the fatigue damaged beams were induction heated to different temperatures, and (3) finally the heated beams were cooled to 15 °C and tested again under fatigue until the complex modulus dropped to the same value as the first fatigue testing. The second fatigue life is a healing indication caused by resting and induction heating. The healing index fatigue life extension ratio can be defined as the second fatigue life divided by the original fatigue life.

3. Results and discussion

3.1. Optimization of steel fibers used for induction heating purpose

It is recommended that short steel fibers be used in asphalt mixture for induction heating purpose [14]. To determine the suitable steel fibers, three types of available commercial chopped steel fibers (namely A02, A08 and Q1-80) were incorporated into hot mix asphalt mixture and the thermal characteristics and induction heating speeds of mixtures produced were investigated. Fig. 4 shows the thermal conductivity, thermal diffusivity and specific heat of asphalt concrete with 6% different types of chopped steel fibers. It can be seen in Fig. 4 that the asphalt mixtures with different types of chopped steel fibers show similar thermal constants. The asphalt mixture with chopped steel fibers Q1-80 has slightly higher thermal conductivity, making it easier to transfer heat to promote self-healing.

Fig. 5 shows the average induction heating speeds at the top surface of asphalt concrete samples with different types of chopped steel fibers, where the heating distance is 10 mm. It is clear in Fig. 5 that the asphalt mixture with chopped steel fibers Q1-80 (the thicker one) has slightly higher induction heating speed than other mixtures. It means that thicker and longer steel wool fibers are more useful to enhance the induction heating speed of asphalt concrete, which is in agreement with Garcia's research [14]. To have higher induction heating and thermal transfer efficiencies, chopped steel fibers Q1-80 were used in the subsequent healing tests.

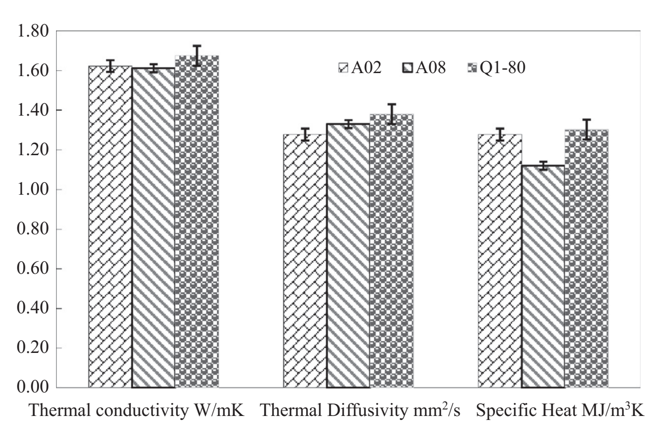

Fig. 4. Thermal constants of asphalt concrete with different types of chopped steel fibers.

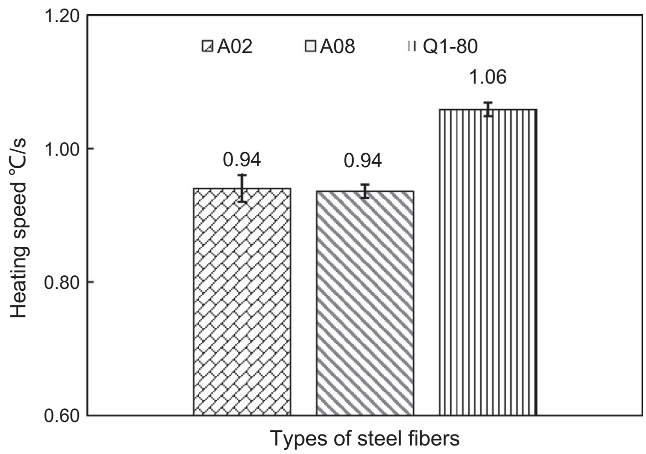

Fig. 5. Induction heating speeds of asphalt concrete with different types of chopped steel fibers.

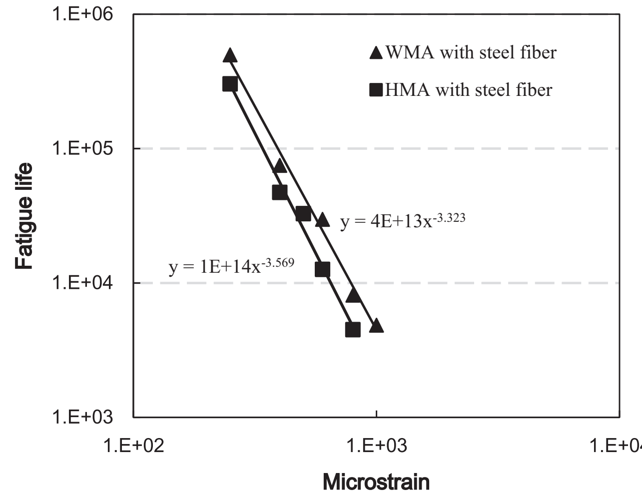

Fig. 6. Fatigue lines of WMA and HMA.

3.2. Low temperature cracking resistances of HMA and WMA with steel fibers

The flexural-tensile stress and the maximum flexural-tensile strain of HMA and WMA containing 6% steel fibers Q1-80 are shown in Table 3. The flexural-tensile stress and strain of HMA mixture is 8.55 MPa and 2322 με, while the flexural-tensile stress and strain of HMA mixture is 8.12 MPa and 2100 με. It is clear that the addition of warm mix additive to WMA mixture slightly decreases its flexural-tensile stress and flexural strain at failure. It means that the low temperature properties of asphalt mixture will be decreased by addition of warm mix additives. The reason for this is that the decreased mixing and compaction temperature of HMA result in a weaker adhesion between bitumen and aggregates and a slightly higher air voids content. The decreased flexural-tensile stress of WMA aggravates the concern of the low temperature cracking of the pavement. In the subsequent fracture-healing test, the potential of using induction heating to heal cracks in WMA will be investigated.

3.3. Fatigue resistances of HMA and WMA with steel fibers

Four point bending fatigue tests were conducted on HMA and WMA samples to compare their fatigue resistances. Fig. 6 shows the fatigue lines of both HMA and WMA containing 6% steel fibers Q1-80. The initial strain corresponding with a fatigue life of 10^6 cycles ε_6 was determined according to the fatigue lines and the fatigue resistances of the mixtures were evaluated and compared with the references.It can be seen from Fig. 6 that the WMA have better fatigue resistance than HMA samples. The active gradients in warm mix additive increase the specific surface area of bitumen and the contact area between bitumen and sand (aggregates), thus improving the coating of the aggregates. As a result, the cohesion strength and fatigue resistance of asphalt mixture can be improved to some extent by adding warm mix additives. The ε_6 of WMA and HMA is 194 microstrain and 174 microstrain respectively. Pang et al. conducted four point bending fatigue test on 3 mixtures and obtained ε_6 ranging from 158, 168 to 184 microstrains [26]. The fatigue resistances of the mixtures used in this research are comparable to the results from Pang's work.

Table 3
Cracking resistances of HMA and WMA with steel fibers.

Type of mixtures	Flexural-tensile stress (MPa)	Maximum flexural-tensile strain (με)
WMA	8.12	2100
HMA	8.55	2322

3.4. Induction heating characteristics of asphalt mixture

The magnetic field intensity is not homogeneous under the coil of the induction heating generator, which results in inhomogeneous heating in the sample. Even at the top surface of the sample, there is also a temperature gradient. Fig. 7 shows a typical infrared image of the sample after induction heating and Fig. 8 shows the temperature distribution at the surface of the sample, where most parts (85.3%) at the top surface of the sample have a temperature between 83.4 °C and 101.2 °C and the mean temperature is 91.8 °C. The asphalt mastic shows a higher temperature than aggregates, which cannot be heated with induction energy.

As it is closer to the coil and suffered more intensive magnetic field, the top surface of the sample can be heated much faster than the lower part. To have a look at the heating speeds at the different depths of the sample, a sample was heated with a heating distance of 30 mm between the coil and the top surface of the sample. Infrared camera was used to monitor the temperature variations at different depths of the sample. Fig. 9 shows the average temperature increasing speeds at different depths of the sample. The heating speeds at the top (30 mm from the coil), in the middle (61.8 mm from the coil) and at the bottom (93.5 mm from the coil) of the samples is 0.42 °C/s, 0.26 °C/s and 0.15 °C/s respectively. This

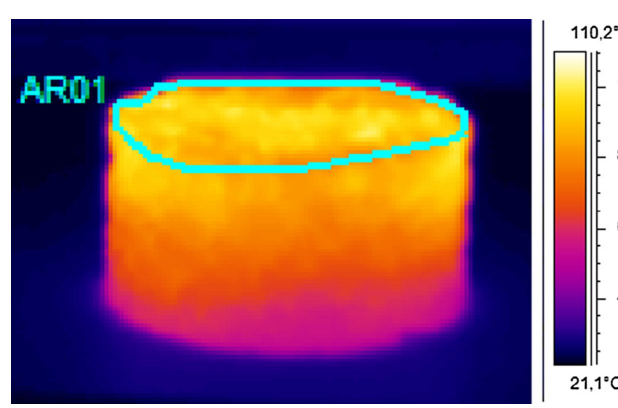

Fig. 7. Typical heating image of the sample.

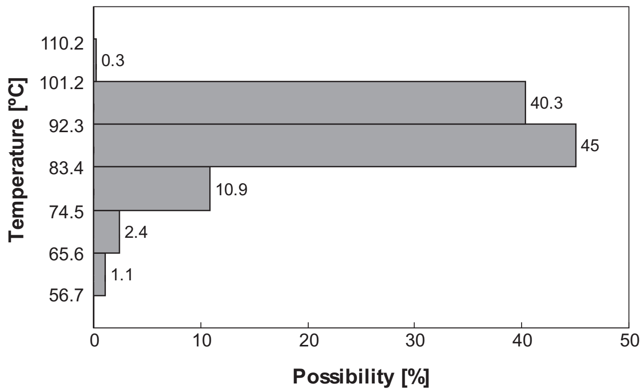

Fig. 8. Temperature distribution at the top surface of the sample.

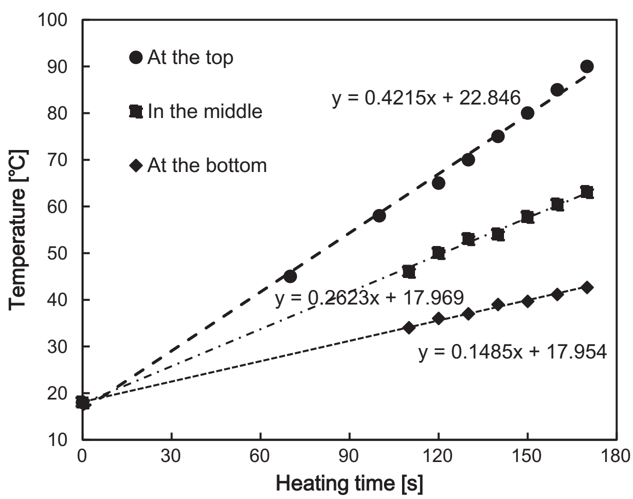

Fig. 9. Different temperature increasing speeds at different depths of the WMA sample.

proves the gradient heating characteristic of induction heating, which will result in different healing ratios at different parts of the sample.

The distance between the coil of the induction heating equipment and the surface of the sample has a significant influence on the heating efficiency of the sample. To get an idea how the heating distance influences the induction efficiency of the samples, the samples were induction heated at a distance of 10 mm, 20 mm and 30 mm respectively. Fig. 10 shows induction heating speeds at the top surface of the WMA samples at different heating distances between the coil and the samples. The heating speeds at the top surface of the sample at a distance of 10 mm, 20 mm and 30 mm is 0.98 °C/s, 0.66 °C/s and 0.42 °C/s respectively. It means that a higher induction heating efficiency can be achieved with a smaller heating distance. As a result, higher healing efficiency will be obtained when the sample is heated at a smaller heating distance.

Fig. 11 shows the induction heating speeds at the top surface of the HMA sample at different heating distances. It can be seen from Figs. 10 and 11 and that the WMA and HMA have almost the same induction heating speed in each case. The nuance lies in the fact that the HMA sample has a slight lower air voids content, which result in a slight higher induction heating speed of the HMA sample. Air bubbles can be clearly observed at the surface of WMA sample when it is heated more than 80 °C. In this case, the remaining warm mix additive coated by bitumen starts to release the

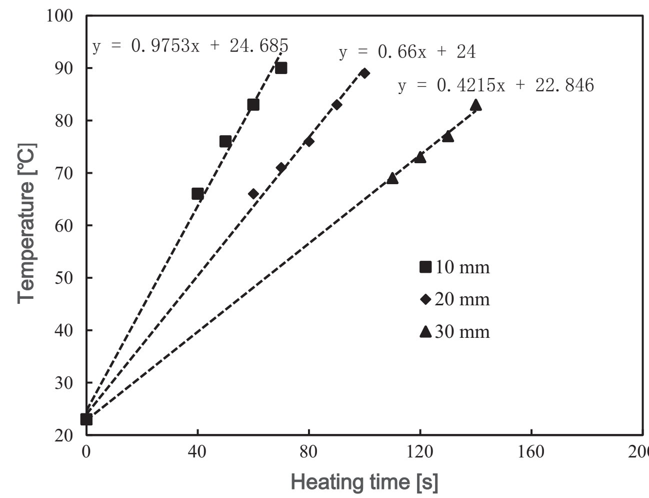

Fig. 10. Induction heating speeds of the WMA at different heating distances between the coil and the samples.

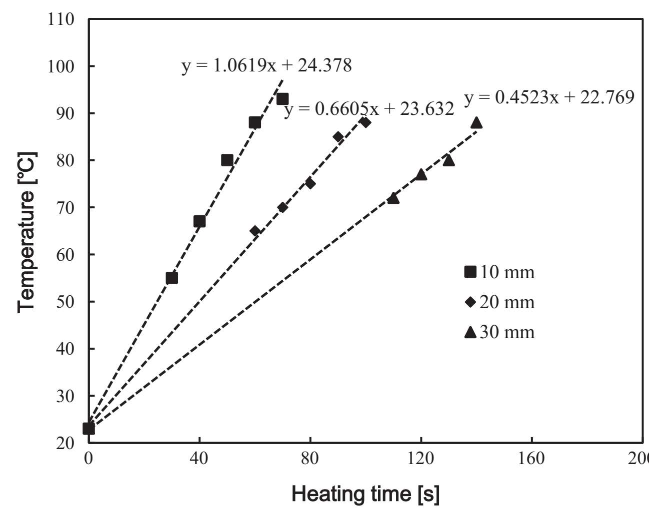

Fig. 11. Induction heating speeds of the HMA at different heating distances between the coil and the samples.

crystalliferous water, generating some foams in the sample. Due to the remaining warm mix additive after mixing, the WMA sample can probably be healed at a lower heating temperature than HMA asphalt.

3.5. Fatigue life extension ratios of HMA and WMA at different heating temperatures

To quantify the effect of heating temperature on the healing rates and to compare the healing capacities of HMA and WMA samples at different heating temperatures, the fatigue life extensions of the fatigue damaged samples with different heating temperature were investigated. For that, the beam samples were fatigued with 600 microstrain and 10 Hz at 15 °C. Then, the samples were induction heated to different temperatures and rested at 15 °C for 3 h. Finally, the fatigue lives of the healed beams were measured again with the same strain amplitude.

Fig. 12 shows the induction healing ratios (fatigue life recovery ratios) of HMA and WMA samples at different heating temperatures. As shown in Fig. 12, the healing ratios of both HMA and WMA samples are highly temperature dependent. The healing

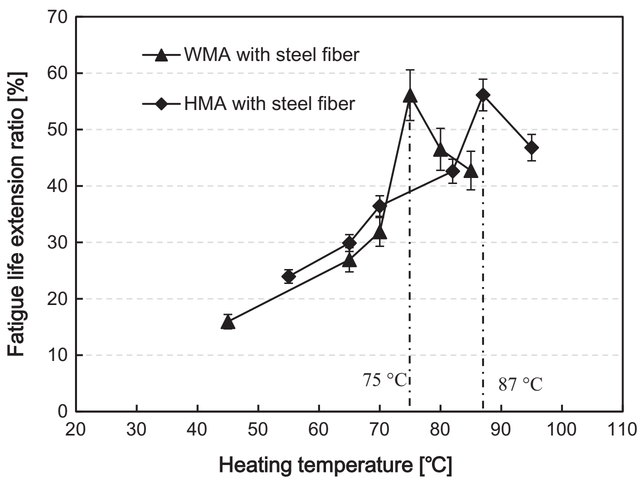

Fig. 12. Fatigue life extension ratios of the samples at different heating temperatures.

ratios increase with the increasing heating temperatures and obtain maximum healing ratios before decreasing. The reason for this decrease can be attributed to the geometry damage of the sample caused by overheating. In this case, the asphalt mortar suffered excess expansion due to overheating and the swelling problem showed up in the sample. The optimal fatigue life ratios of HMA and WMA samples are 56.17% and 56.09% at 87 °C and 75 °C respectively. The two kinds of mixture showed the same optimal fatigue life ratio in this case, but the WMA samples obtained the optimal healing ratio at a much lower heating temperatures. When the average temperature reaches 75 °C, the remaining warm mix additives in the WMA samples starts foaming and reduce the viscosity of the binder, which accelerates the flow of bitumen and promotes healing.

To quantify the effect of the microstrain amplitude on the healing rates of HMA and WMA samples at the optimal heating temperatures, the fatigue life extension ratios of the samples with different microstrain amplitudes in fatigue testing were measured at 15 °C and 10 Hz and plotted in Fig. 13. In this experiment, the HMA and WMA samples were heated to 87 °C and 75 °C respectively for healing. It can be seen from Fig. 13 that the both mixtures obtained maximum fatigue life extension ratios at 400 microstrain. At a lower microstrain amplitude, a great amount of energy was dissipated during fatigue testing and the samples suffered significant damage. As a consequence, the fatigue life extension ratios is decreased at a lower microstrain amplitude. At higher microstrain amplitudes, bigger cracks were generated in the fatigue testing which is more difficult to heal. So, the fatigue life extension ratios of the samples decreased with the further increasing of the microstrain amplitude.

Generally, the HMA samples showed higher fatigue life extension ratios than WMA samples. The main reason could be that the HMA samples were healed at a higher temperature.

3.6. Strength recovery ratios of HMA and WMA samples at different heating temperatures

Fig. 14 shows the strength recovery ratios of the samples at different heating temperatures. The fractured beam samples could not recover it strength at room temperature and lower temperatures, because the samples were completely fractured and some stones were broken (self-healing can only heal micro-cracks). The strength recovery ratios of both HMA and WMA beams increase with the increasing heating temperature and reach optimums before decreasing. The optimal heating temperature for HMA and WMA beams is 85 °C and 75 °C respectively. Compared with HMA sample, the WMA sample obtained its optimal healing ratio at a lower induction heating temperatures due to the foaming effect of remaining warm mix additives. When average temperature at the top surface of the WMA sample reaches 75 °C, the temperature in the mastic has reached around 90 °C, at which temperature the remaining active ingredients of warm mix additive take effect. As a result, the optimal healing temperature of WMA sample is lowered due the decreased viscosity of the bitumen. It is estimated that the optimal heating temperatures of WMA will gradually be increased with increasing heating times due to the decrease of the active ingredients of warm mix additive in the mixture and finally get close to the optimal heating temperatures of HMA. However, the maximum strength recovery ratio of WMA is also decreased compared with HMA. With the further increasing of the induction heating temperature, more and more active ingredients of warm mix additive remained in WMA samples take effect and the foams generated at the crack surface will prevent the closure of the crack. As a result, the healing ratios of the WMA sample decreased sharply with the increasing temperature above 75 °C.

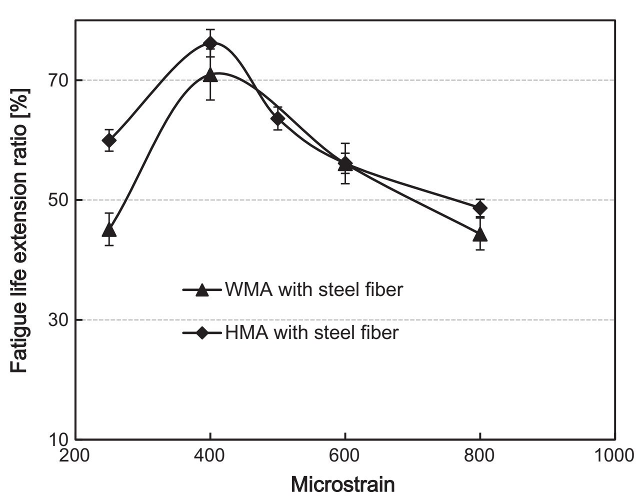

Fig. 13. Fatigue life extension ratios of the samples under different microstrain amplitudes.

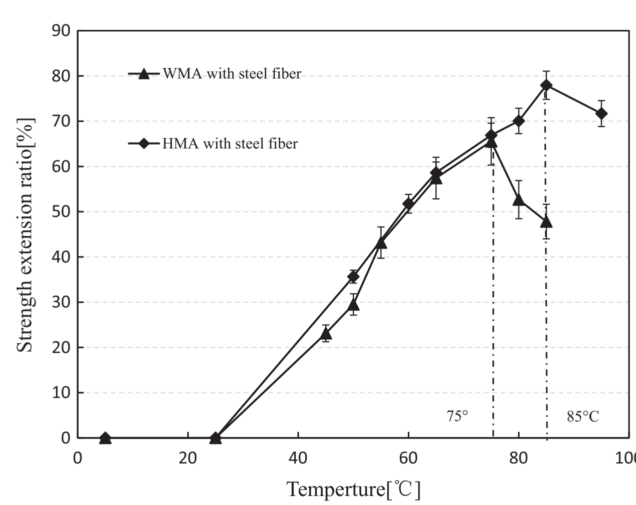

Fig. 14. Strength recovery ratios of the samples at different heating temperatures.

4. Conclusions

In this paper, the induction healing behaviors of warm mix asphalt were investigated and compared with hot mix asphalt through damage-healing-damage test. The steel fibers used to make asphalt mixture conductive was first optimized according to the thermal conductivity and induction heating speed of the mixture. Then, the cracking resistance, fatigue resistance, induction heating characteristics and self-healing ratios of both warm mix asphalt and hot mix asphalt were studied. Based on the test results, the following conclusions can be drawn.

1) The Aspha-Min based WMA shows slightly higher fatigue resistance but lower low temperature cracking resistance than HMA.
2) The addition of warm mix additives to asphalt mixture doesn't influence its induction heating speed. Induction heating of asphalt mixture shows strong gradient heating characteristics: the top parts of the sample can be heated much faster than the lower parts, which will result a higher healing ratio in the top parts of the sample. The gradient healing behavior of asphalt concrete during induction heating still needs further research in the future.
3) Both WMA and HMA have very high healing ratios with induction heating, which means that the durability of WMA can be improved by induction heating. The optimal induction heating temperature of WMA is lower than that of HMA due to the remaining active ingredient in the warm mix additives, which also deceases the strength recovery ratio of WMA. It is believed that the foams generated at the crack surface will prevent the closure of the crack in WMA.
4) The fatigue life recovery ratios of both WMA and HMA mixtures are highly strain-dependent and the mixtures obtained best healing behaviors at medium microstrain amplitudes. At lower microstrain amplitudes, the sample suffered more fatigue damage. At higher microstrain amplitudes, bigger cracks occurred the sample and they were difficult to heal.

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