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ABSTRACT

Conventional asphalt mixture has poor microwave absorbing performance and microwave heating efficiency. Based on the characteristics of dielectric loss of scrap tire pyrolysis carbon black (PCB), it is proposed to improve the microwave absorbing performance and self-healing rate of bitumen. The phase composition and electromagnetic parameters of PCB were tested to reveal its microwave heating mechanism. The preparation parameters, heating characteristics and self-healing properties of PCB modified bitumen were studied through the dispersion uniformity test, microwave heating test and SCB test. The main phases of PCB are microwave absorbing carbon and silicon, indicating that PCB is a good electric loss microwave absorbing material. 40 min is the recommended mixing time of 15% PCB modified bitumen. PCB's dosage, microwave frequency and microwave heating time have significant effects on the microwave heating characteristics of PCB modified bitumen. PCB can improve high-temperature stability, thermal conductance, heat storage capacity and self-healing rate of bitumen.

1. Introduction

Asphalt pavement has excellent road performance, it is widely used in road engineering and has become the most common pavement type [1,2]. Its general service life is 8 to 15 years[3,4]. However, due to the coupling effect of the rapid increase of vehicle ownership and changeable climate conditions, the service pressure of asphalt pavement increases sharply and the performance of asphalt pavement decreases, which gradually produces pavement distresses and greatly shortens its service life[5]. The performance of bitumen determines the service performance of asphalt pavement[6], so higher requirements are put forward for bitumen performance.

Bitumen modification is one of the methods to effectively improve the performance of asphalt pavement[7,8]. The researchers use additives to improve the performance of bitumen and thus improve the road performance of asphalt mixtures. Silica gel, polymer, rubber and other materials are usually used to modify bitumen[9]. For example, styrene–butadienestyrene block copolymer (SBS) modified bitumen has excellent low-temperature anti-cracking performance and high-temperature rutting resistance[10,11], and styrene-butadiene rubber (SBR) modified bitumen has outstanding low-temperature performance [12]. Bitumen can also be modified by polyethylene (PE) materials, such as bitumen modified by diselenide-crosslinked polyurethane elastomer (DCP), and its self-healing performance is greatly improved compared with that before modification [13,14]. In addition, bitumen can be modified by double modification, such as obtaining nano-organic palygorskite (O-PAL) and SBS modified bitumen, which can make O-PAL composite SBS modified bitumen has good water stability, aging resistance and stability [15,16]. The above is to improve the road performance and function of bitumen by adding admixtures.

Relevant studies showed that carbon black has a good affinity with bitumen[17], and can be used as an admixture of bitumen. Carbon black can alleviate the aging effect of ultraviolet light on bitumen and improve the durability and crack resistance of asphalt mixture pavement[18]. Through rutting tests, Geckilh[19] and Li[20] concluded that adding carbon black to bitumen can improve the rutting resistance and fatigue

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resistance of asphalt mixture. After pyrolysis of scrap tires, the carbon black added in the production of new tires will be transformed into scrap tires pyrolysis carbon black (PCB)[21]. PCB is a by-product of the pyrolysis of scrap tires. Excessive accumulation of scrap tires has certain safety hazards, while burning scrap tires will damage the environment. The use of PCB can promote the development of the pyrolysis scrap tire industry, and can also reduce the space, safety and environmental problems caused by excessive scrap tires[22,23]. At the same time, using PCB in asphalt mixture can enhance the rutting resistance, medium temperature fatigue performance and high-temperature performance of virgin asphalt mixture[9,24]. It has a large production and a low price, and has the potential to replace commercial carbon black[25,26]. Its application to bitumen materials may have great economic benefits.

Bitumen has self-healing performance, which can automatically repair its internal micro-cracks and restore its structural integrity and service performance[13,27]. However, its self-healing rate has typical temperature dependence and the self-healing rate increases with the increase of temperature[4]. Therefore, the method of increasing bitumen temperature can be used to improve its self-healing rate. The heating process of bitumen has gradually developed from traditional oven heating to infrared heating and induction heating. Both infrared heating and induction heating have good heating effects on bitumen [28,29]. However, induction heating of asphalt mixture does not have the characteristics of selective, deep and non-destructive heating, and may not effectively treat the internal distresses of asphalt pavement[30]. In this technology, the damaged road surface is cleaned before placing the bituminous mix. Thereafter, the mix is cured using a microwave curing vehicle for a certain duration and finally compacted for serviceability requirements. First, clean the damaged part of the road surface. After drying, place the prefabricated microwaveable asphalt mixture there, use the microwave curing vehicle to microwave heating for a certain time, and finally compact it. Compared with induction heating, the application of microwave heating technology in asphalt pavement maintenance can make asphalt pavement have better self-healing characteristics[4]. Wang[31] heated the asphalt mixture by microwave and infrared respectively. The results showed that the asphalt mixture heated by microwave has good uniformity, heating efficiency and action depth. Relevant studies have shown that adding microwave absorbing materials to asphalt mixture can significantly improve the self-healing performance of asphalt mixture. For example, after adding microwave absorbing materials such as hot burned steel slag[32], carbon-based material[33], steel wool fiber[34] and activated carbon powder[35]to the asphalt mixture, the heating rate and self-healing performance after microwave heating are significantly improved compared with the asphalt mixture without microwave absorbing material.

Microwave heating can make the heated object actively become a heating body without heat conduction of thermal energy[36]. Microwave refers to electromagnetic waves with frequency of 0.3 to 300 GHz and wavelength of 0.001 to 1 m[37]. Impedance matching and attenuation characteristics determine the wave absorbing properties of the material. Impedance matching is processed at the material interface so that electromagnetic waves can be completely incident without being reflected. Attenuation characteristics refer to the absorption of electromagnetic waves by the material and the loss of electromagnetic waves, microwave energy loss can be divided into dielectric loss and magnetic loss[38]. The dielectric loss is determined by the dielectric constant of the medium and electric field. The dielectric loss mechanism is the relaxation loss caused by dielectric polarization. The magnetic loss is determined by the permeability of the medium and magnetic field, and the magnetic loss mechanism is hysteresis loss and ferromagnetic resonance[39]. Electromagnetic parameters such as dielectric constant and permeability are the main parameters to quantify the microwave absorption efficiency of materials, which are related by Equations 1, 2 and 3[40].

$$\varepsilon = \varepsilon' - j\varepsilon'' \tag{1}$$

$$\mu = \mu' - j\mu'' \tag{2}$$

$$\tan\delta = \tan\delta_E + \tan\delta_M = \frac{\varepsilon''}{\varepsilon'} + \frac{\mu''}{u'} \tag{3}$$

In the Equations above, ε is the dielectric constant, ε' and ε" are respectively the real and imaginary parts of the dielectric constant. μ is magnetic, and μ' and μ" are respectively the real and imaginary parts of the magnetic permeability. Tanδ is the loss angle tangent, $\tan\delta_E$ and $\tan\delta_M$ are respectively the dielectric loss angle tangent and the magnetic angle loss tangent. The real part values of dielectric constant and permeability determine the ability of wave-absorbing material to store electric and magnetic energy, while the imaginary part values of dielectric constant and permeability determine the dielectric and magnetic loss ability of wave-absorbing material[36,37,41]. Dissipation factor (Tan*δ*) is the ratio of lost energy to stored energy. It determines the absorbing capacity of the absorbing material, and its loss type can be determined by the tangent value of the electromagnetic loss of the absorbing material[42]. There are three main path modes for the effect of electromagnetic waves on the object, and only the electromagnetic wave of the third path can successfully enter the interior of the material and convert the electromagnetic wave energy into heat energy to heat the object[39]. The first and second paths of electromagnetic waves determine that the wave-absorbing material should have impedance matching characteristics so that the reflectivity and transmittance of electromagnetic waves on the material surface can be reduced to the greatest extent and the absorption capacity can be improved. The third path of the electromagnetic wave determines that the wave-absorbing materials should have attenuation characteristics. After the electromagnetic wave enters the material, the electromagnetic wave energy is converted into heat energy by rapidly increasing the dielectric loss or magnetic loss[43]. Electromagnetic parameters determine the impedance matching and attenuation coefficient of microwave absorbing materials. The better the impedance matching, the lower the microwave reflectivity and transmittance, the greater the attenuation coefficient and the higher the absorption[43,44].

The absorbing performance of microwave absorbing materials at different microwave frequencies is determined according to the reflection loss (RL) at that frequency. The smaller the RL, the higher the efficiency of the electromagnetic wave being absorbed and converted into heat energy[40,45,46]. The calculation Equations of RL are shown in Equations 4 and 5.

$$Z_{in} = Z_0\sqrt{\frac{\mu_r}{\varepsilon_r}}\tanh\left(j\frac{2\pi fd}{c}\sqrt{\mu_r\varepsilon_r}\right) \tag{4}$$

$$RL = 20\lg\left|\frac{Z_{in} - Z_0}{Z_{in} + Z_0}\right| \tag{5}$$

In Equations 4 and 5, Z_{in} is the impedance of the absorbing layer, Z_0 is the impedance of free space, ε_r and μ_r are the complex relative permittivity and complex relative permeability respectively, j is an imaginary unit, f is the electromagnetic wave frequency, d is the thickness of the absorbing layer, and c is the speed of light. According to the transmission line theory, the final absorbing performance of the wave-absorbing material is determined by the RL, the smaller the RL, the better the absorbing performance of the material[39]. When the RL of a certain microwave frequency range is between −5 dB and 0 dB, only 68% of the electromagnetic waves in this range can be absorbed by the absorbing material. When the RL of a certain microwave frequency range is less than −10 dB, 90% of the electromagnetic waves in this range can be absorbed by the absorbing material. When the RL of a certain microwave frequency range is less than −20 dB, 99% of the electromagnetic waves in this range can be absorbed by the absorbing material[40,45]. It can be seen from Equation 5 that the RL of the absorbing material is directly related to the impedance Z_{in} of the microwave absorbing material. From the analysis of Equation 5, it can be seen that when Z_{in} is close to 1, the smaller the RL value, the better the microwave absorbing performance.

In the related research of carbon black, the researchers inferred that both carbon black and PCB have strong microwave absorbing properties [47-49], and some road workers applied carbon black admixture and microwave heating technology to the road field at the same time[50-53]. After adding PCB into bitumen and heating it by microwave, it can improve the self-healing performance of PCB modified bitumen. However, the research on the microwave absorption mechanism of carbon black is limited to the study of electromagnetic parameters, and there are few comprehensive studies on the phase composition and electromagnetic parameters of carbon black. The research on the preparation parameters of carbon black modified bitumen is rarely involved, and the finished product quality of carbon black modified bitumen (PCB dispersion uniformity) is not clear. The research on carbon black modified bitumen combined with microwave heating focuses on the road performance and self-healing performance of carbon black modified asphalt mixture, but there is little research on the self-healing performance of carbon black modified bitumen.

PCB will be added to bitumen as a microwave absorber, which absorbs microwave energy under the action of the microwave and transfers the generated heat to bitumen. Microwave heating of bitumen is intended to enhance its microwave loss ability, so that PCB modified bitumen can absorb higher microwave energy and improve its self-healing performance under the action of the microwave. The main work of this study consists of three parts. The first part is to use the statistical variance method to determine the stirring time of PCB modified bitumen. Then, the performance of 70# base bitumen and 15% PCB modified bitumen is tested by three parameters and viscosity test, and studying the influence of PCB on the physical properties of bitumen. The second part is to study the microwave absorbability of the PCB by testing the phase composition of the PCB. Test the electromagnetic parameters of the PCB to study the electromagnetic parameters and microwave absorption characteristics of the PCB, and use these two tests to reveal the microwave absorption mechanism of the PCB. The third part is to study the microwave heating characteristics of bitumen with four different PCB dosages through the test of bitumen heating rate. The semicircular bending (SCB) test is performed on the bitumen beam specimens with four different PCB dosages, and the self-healing performance of the bitumen is evaluated by the value of maximum braking force. The ultimate goal of this study is to determine the stirring time of PCB modified bitumen, reveal PCB's microwave heating mechanism and demonstrate that PCB can improve the self-healing rate of bitumen under microwave heating.

2. Materials and experimental method

2.1. Bitumen

The bitumen used in this study is 70# base bitumen, which is one of the most commonly used types of road petroleum bitumen in China. Some researchers have studied the effect of carbon black dosage on the performance of 70# base bitumen. The results showed that with the increase of carbon black dosage, the high-temperature stability of carbon black modified bitumen is significantly improved, but the low-temperature cracks resistance decreases[20,54]. The mixing ratio of carbon black and bitumen is within 15%[22]. PCB with 15% of bitumen mass was added to the bitumen to prepare 15% PCB modified bitumen. The performance parameters of 70# base bitumen and 15% PCB modified bitumen and the variation after adding 15% PCB are shown in Table 1, their viscosity results are shown in Table 2, and the results are all in line with the specification requirements.

It can be seen from Table 1 that after adding 15% PCB, the technical parameters of 70# base bitumen change significantly. The greater the penetration, the worse the high-temperature stability. The greater the ductility, the better the cracking resistance and low-temperature rheological properties of bitumen in a low-temperature environment. The higher the softening point, the better the high-temperature performance, the higher the viscosity, and the stronger the deformation resistance and high-temperature stability[55,56]. Compared with the three parameters before modification, the penetration and ductility of bitumen decreased, while the softening point increased, in which the penetration decreased by 20.40%, the ductility decreased by 76.4% and the softening point increased by 7.0%. Table 2 shows the results of bitumen viscosity. The bitumen viscosity of both temperature grades was more than 50% higher than that before modification, among which the viscosity at 135 °C increased by 53.6%, and the viscosity at 165 °C increased by 51.2%. It showed that the addition of PCB to the bitumen improves the high-temperature stability of the bitumen and reduces the low-temperature cracking resistance of the bitumen, but it still meets the requirements of the corresponding specifications.

Table 1
The performance parameters of 70# base bitumen and 15% PCB modified bitumen and the variation after adding 15% PCB.

Testing parameters	70# base Bitumen	15% PCB modified bitumen	Variation after adding PCB
25 °C penetration	70.6 dmm	56.2 dmm	14.4 dmm
10 °C ductility	40.50 cm	9.55 cm	30.95 cm
Softening point (R&B)	47.5 °C	50.5 °C	3.0 °C

Table 2
Test results of bitumen viscosity.

Temperature (°C)	Viscosity (Pa.s) 70#base bitumen	15% PCB modified bitumen
135	0.485	0.745
165	0.135	0.205

Fig. 1 shows the viscosity temperature curve of 70# base bitumen and 15% PCB modified bitumen drawn according to Table 2. The specification stipulates that the optimal compaction viscosity standard of asphalt mixture is 0.28 ± 0.03 Pa.s, and the optimal mixing viscosity standard is 0.17 ± 0.02 Pa.s. The optimal compaction temperature of 70# base bitumen was 150.0 to 155.1 °C and the optimal mixing temperature was 160.2 to 163.7 °C. The optimum compaction temperature of 15% PCB modified bitumen was 159.2 to 162.5 °C, and the optimum mixing temperature was 165.8 to 168.1 °C. The optimum mixing temperature and compaction temperature of 15% PCB modified bitumen were higher than 70# base bitumen.

2.2. Scrap tire pyrolysis carbon black (PCB)

The PCB of scrap tires is in the form of black powder, and the specific surface area of PCB is equivalent to that of commercial carbon black [23]. The PCB sample of scrap tires is shown in Fig. 2.

PCB is mainly composed of carbon, oxygen, copper and other elements, and the content of carbon elements reaches more than 80% [23,57]. The microstructure of carbon black is spherical agglomerations formed by the mutual attraction of basic aggregate structures under the action of intermolecular force. The basic particle size of carbon black is 10 to 600 nm[58]. PCB is further agglomerated into primary aggregates of 100 to 200 nm from the basic aggregate structure of 10 to 50 nm[59]. Fig. 3 shows the SEM images of PCB at different magnifications. The microsurface of PCB aggregate is rough, showing a chain branch layered structure, like the appearance of coral, and the shape is extremely complex.

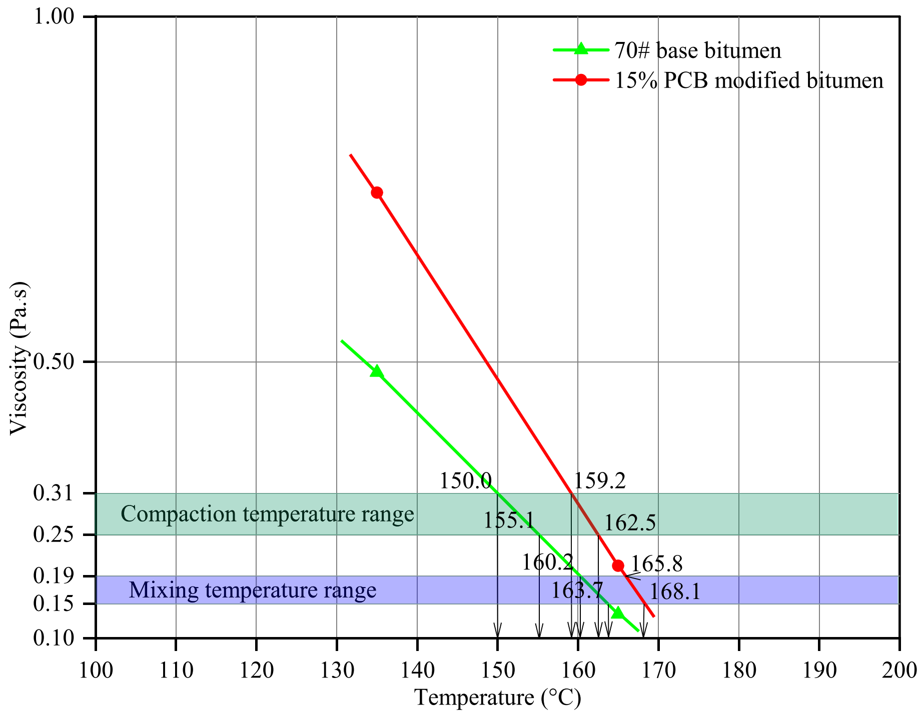

Fig. 1. Viscosity temperature curve of 70# base bitumen and 15% PCB modified bitumen.

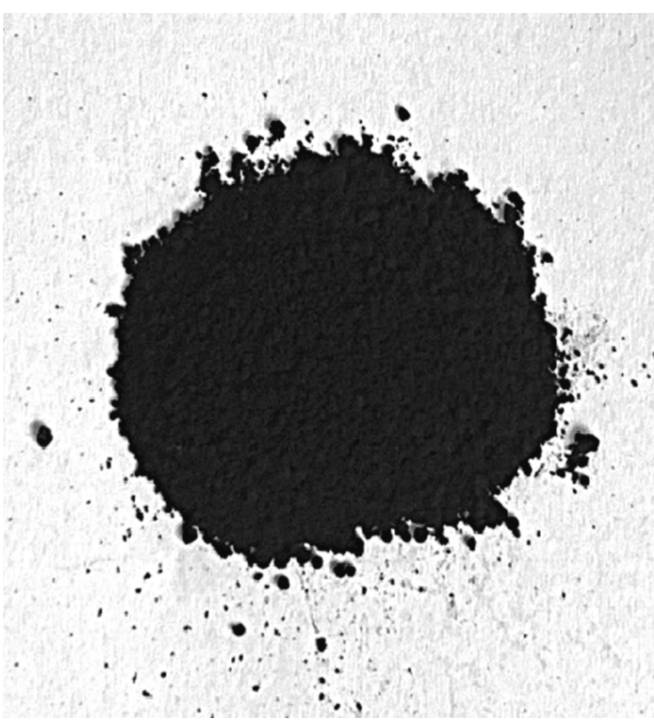

Fig. 2. PCB powder from scrap tire.

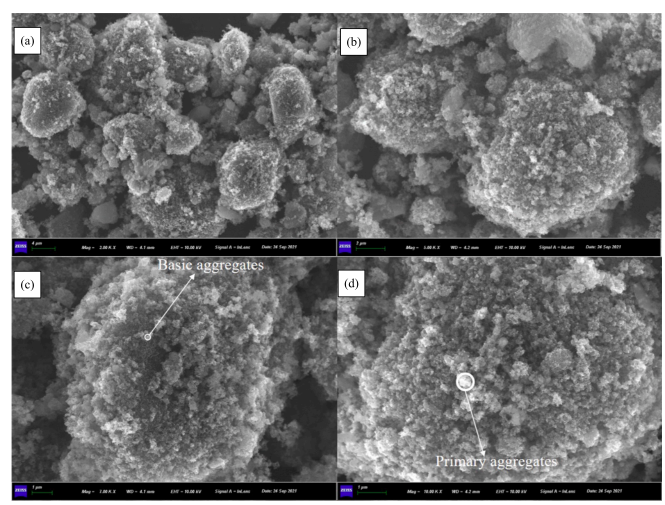

Fig. 3. SEM images of PCB at different magnification. (a. 2000 ×; b. 5000 ×; c. 7000 ×; b. 10000 ×).

2.3. *Test method for characterization of PCB*

2.3.1. *Measurement of micromorphology of PCB*

SEM images of PCB with multiples of 2000 ×, 5000 ×, 7000 × and 10000 × were taken with a Hitachi SU8010 instrument, 1 kV resolution up to 1.3 nm. Firstly, the PCB powder was put into the gold spraying instrument for gold spraying treatment to enhance its conductivity. After gold spraying, take out the sample and put it into the sample bin of the SEM scanning electron microscope. After the sample bin reached the vacuum degree that can be tested, the micromorphology of the PCB sample was tested. The topography of the sample was then viewed on a computer monitor and the desired high magnification image was acquired.

2.3.2. *Measurement of phase composition of PCB*

XRD can conduct qualitative and quantitative analyses on materials. Qualitative analysis is to indicate the composition of confirmed phases and the formation of new phases, and quantitative analysis is to indicate the content of confirmed phases in the tested materials. The phase composition of PCB was analyzed by X-ray diffraction (XRD, Bruker D8 advance). Before the test, prepare 5 g PCB sample and set the working parameters of voltage 40 kV and current 40 mA, and then test the PCB sample at the scanning speed of 5°/min and the test range of 10° to 80°.

The XRD test data of the PCB was imported into the software Jade 9.0, and the phase composition and distribution of the PCB were obtained by analyzing the software.

2.3.3. Measurement of electromagnetic properties of PCB

Microwave vector network analyzers (MVNA, Agilent E5071C) were used to test the electromagnetic properties of PCB with the frequency range of 1 to 18 GHz by the coaxial method. Make test samples of vector network analyzer before testing, mix PCB and paraffin 1:1 evenly to make test samples. During the test, first weigh the mass of the empty sample tube, and then load a certain amount of sample and weigh the mass of the sample and the sample tube again. The interval between the two is the mass of the test sample. Then, load the sample on the MVNA equipment, and start the test after writing the test program according to the requirements.

2.4. Test method for bitumen

2.4.1. Test of PCB dispersion uniformity

Before testing the microwave heating characteristics of bitumen, it was necessary to prepare modified bitumen with different dosages of PCB. The PCB modified bitumen samples with 5%, 10% and 15% PCB were prepared. When preparing PCB modified bitumen, first put the bitumen into an oven at 135 °C and heat it until it flows. After adding the required PCB into the mixing tank, add the flowing bitumen in proportion and mix it. At this point, the density of the PCB modified bitumen samples mixed with 5%, 10%, and 15% PCB was 1.058 g/cm^3, 1.078 g/cm^3, and 1.098 g/cm^3, respectively.

The dispersion uniformity of 15% PCB modified bitumen with different stirring times was tested. Prepare 9 mixing tanks, respectively add PCB and 70# base bitumen into the mixing tank in proportion, and mix them manually until they were roughly mixed. Put it to the mixing table successively and stir it at the rate of 500 rpm for 5 min to 45 min to prepare the mixing sample of PCB modified bitumen. After stirring, pour 15% PCB modified bitumen into the paper tray for sample retention. The bitumen mass of each sample is 25 g, as shown in Fig. 4, and then let it stand at room temperature. Put the samples into the microwave oven for heating for 15 s, as shown in Fig. 5, quickly intercept the thermal image with the thermal imager (emissivity 0.30), and then process the thermal image with software. The uniformity of the surface temperature distribution of 15% PCB modified bitumen after microwave heating was used to characterize the dispersion uniformity of PCB in the bitumen, to determine the preparation parameters of the modified bitumen.

Fig. 6 shows the thermal images of different stirring times under 15 s microwave heating. It can be roughly concluded by visual inspection that with the increase of stirring time, the PCB was more uniformly

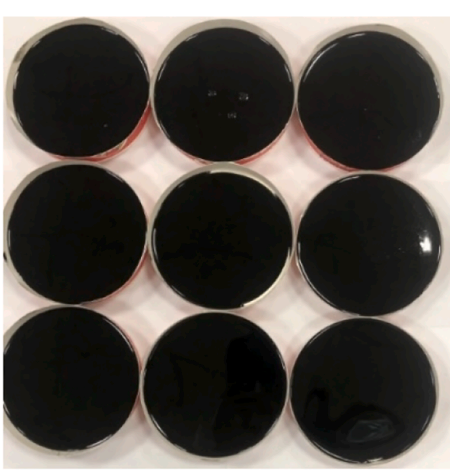

Fig. 4. PCB modified bitumen with different stirring times.

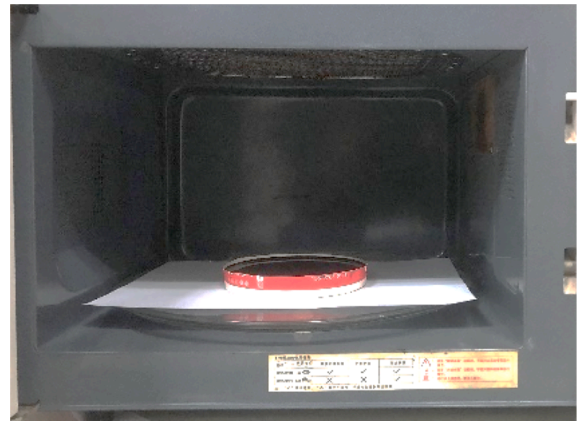

Fig. 5. Microwave heating of samples.

dispersed, and the surface temperature of the specimen was also dispersed more evenly. According to the standard deviation comparison method of statistical principle, the uniformity of each PCB modified bitumen sample was studied. The histogram shown in Fig. 7 represents the temperature distribution by the number of pixels. The X-axis is the initial value of the temperature interval and the Y-axis is the number of pixels. Each sample was divided into 25 temperature intervals.

2.4.2. Physical properties and viscosity test of bitumen

Penetration, ductility, softening point and viscosity test of bitumen were tested according to T0604-2011, T0605-2011, T0606-2011 and T0625-2011 in JTG E20-2011 respectively.

2.4.3. Heating rate test of bitumen

The test was divided into two parts. In both parts, the bitumen samples were heated by microwave for 0 to 120 s. 12 samples were prepared for each dosage of bitumen, and the test was performed every 10 s. The first part of the heating test was the microwave heating test of bitumen with different dosages of PCB under the power of 800 W microwave oven. The heating rate of PCB modified bitumen will be greatly improved in theory under microwave heating, the bitumen slurry with different dosages of PCB is heated by microwave to study the microwave heating effect of bitumen with different dosages of PCB.

The second part of the heating test was the microwave heating test of 15% PCB modified bitumen under different microwave powers. Under microwave heating conditions, the power of the microwave oven also affects the heating rate of the PCB modified bitumen. The influence of power on the heating effect of PCB modified bitumen can be studied by changing the microwave output power. When doing the second part of the microwave heating test, the power of the microwave oven should be controlled to be 800 W (the first part has been done), 640 W, 480 W, 320 W, and 160 W.

Due to the different bitumen mass, the microwave heating effect is also different. To reduce the error caused by the bitumen mass, the mass of the bitumen should be controlled to be the same. Pour four kinds of bitumen with different PCB dosages into 12 test cups, and control the bitumen mass in each cup to be equal, as shown in Fig. 8.

2.4.4. Thermophysical properties test

TPS2500S was used as the test instrument, and the bitumen samples were tested by 60 °C transient plate heat source method (hot-disk method) according to ISO22007-2 standard. Before the test, 15% PCB modified bitumen and 70# base bitumen were heated in an oven at 135 °C for 4 h. Before pouring the bitumen into the mold, the circular mold shall be covered with high temperature resistant fresh-keeping film, and the inner diameter of the mold is 5 cm. Pour 20 g of heated 15% PCB modified bitumen and 70# base bitumen into the mold respectively, and let it stand for cooling. After cooling, the upper surface

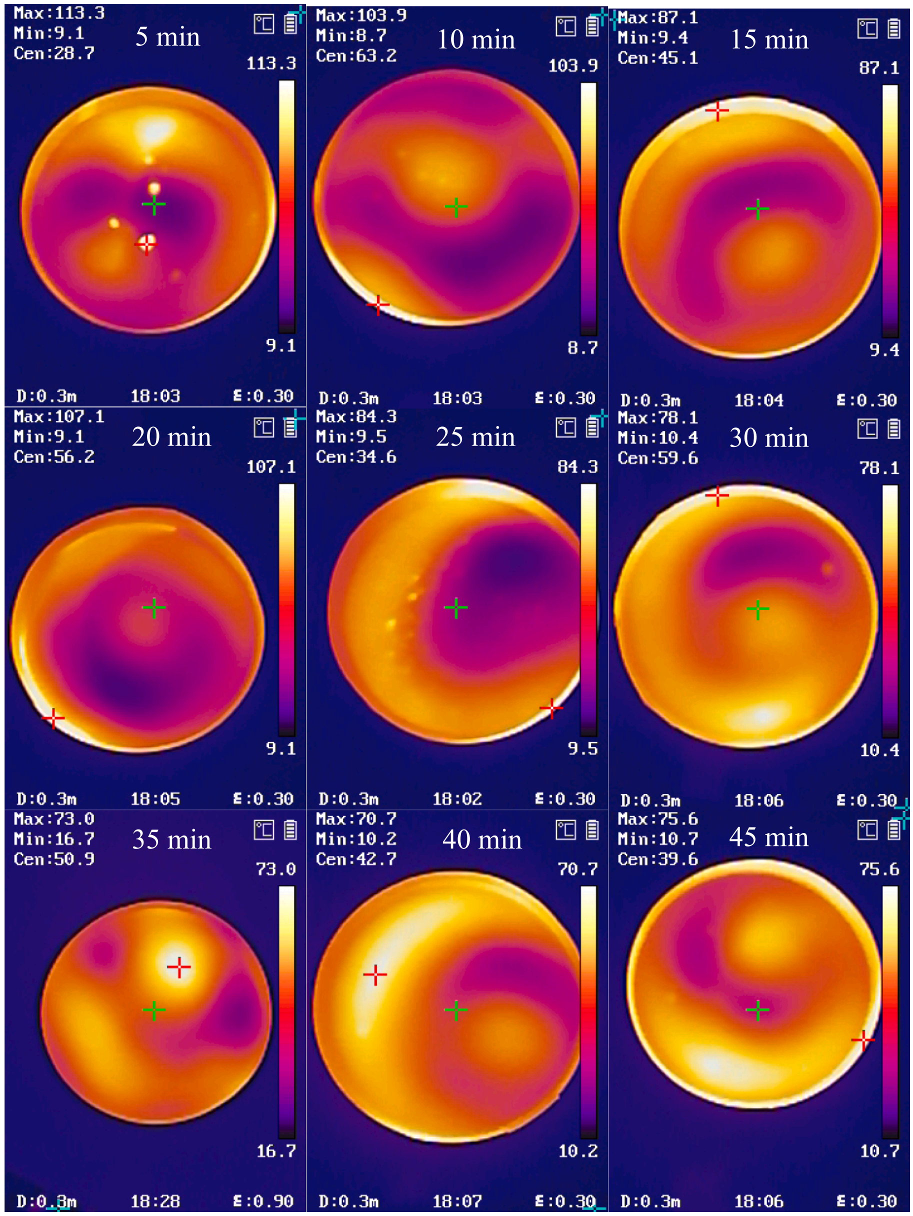

Fig. 6. Thermal images of 15% PCB modified bitumen at different mixing times.

of the bitumen sample was wrapped with excess high-temperature resistant fresh-keeping film to prevent bitumen overflow during the test. Three cake-shaped bitumen samples were made for each group, and the average test results were taken. During the test, the six samples were folded in half, the smoother side was in contact with the probe of the instrument, and the bitumen sample was squeezed to make it in close contact with the probe.

2.4.5. SCB test

PCB and 70# base bitumen were mixed in the proportions of 5%, 10% and 15% respectively, and the PCB modified bitumen was prepared after stirring evenly, the effect of PCB dosage on the self-healing performance of the bitumen was studied. Bitumen specimens for this test were formed using beam molds. Before the bitumen was poured into the mold, the inner wall of the mold needed to be coated with a release agent, and then the bitumen was poured to the level of the molding plane. After standing to room temperature, a transverse slit with a size of 20 mm × 1.5 mm × 2 mm was pre-pressed in the center of the specimen. Two specimens were prepared for each dosage, for a total of eight specimens. After that, it was placed in a −10 °C hold for half an hour, and then the specimen was taken out for demolding. After demolding, the specimens were placed at −10 °C again for 4 h. SCB test was performed on the specimens, and the maximum breaking force data of the SCB test was recorded. Put the destroyed specimen into the paper trabecular mold, and seal the grouting surface with heat-resistant tape. The destroyed surface should be in close contact while sealing. After standing for 12 h to room temperature, put it in the microwave oven for 120 s. After continuing to stand for 12 h to room temperature, repeat the above seam pressing, heat preservation process and bending tensile destroy process. The specific test process is shown in Fig. 9.

2.5. Technical map

Fig. 10 is the technical map of this study.

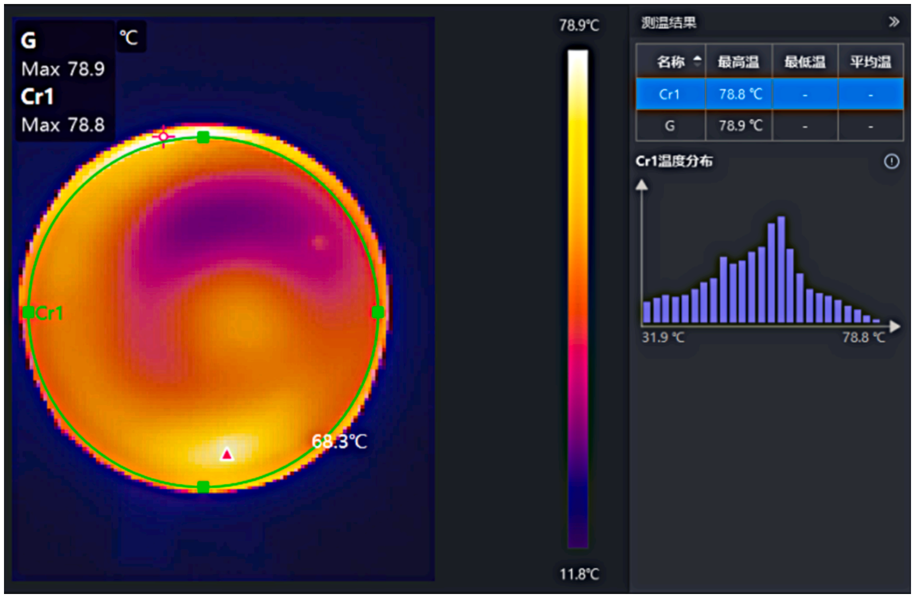

Fig. 7. Processing results of the temperature profile after stirring for 30 min.

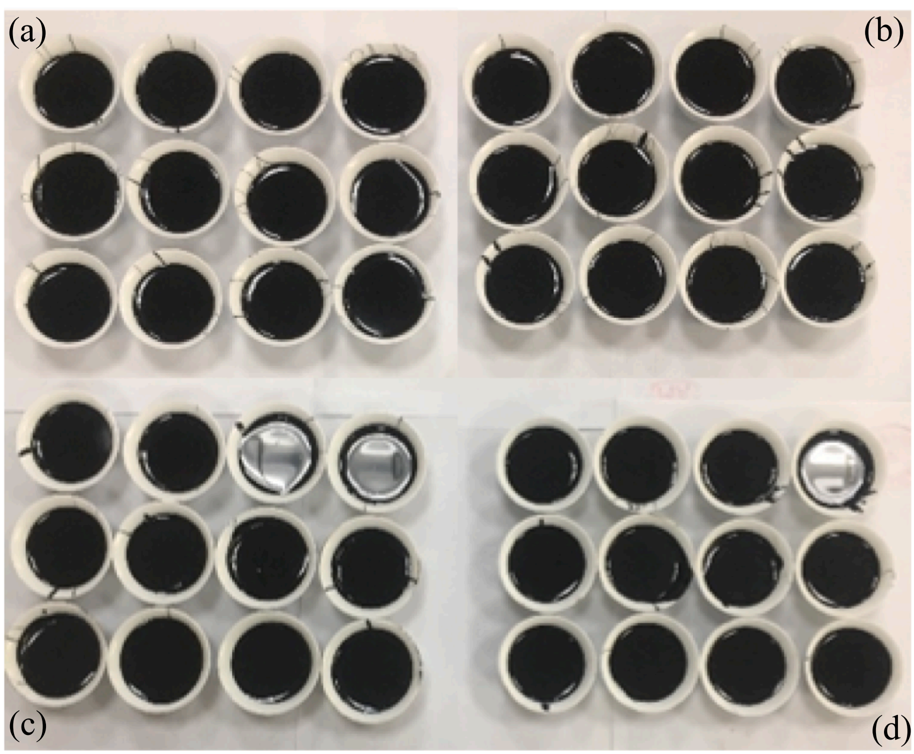

Fig. 8. Modified bitumen with different dosage of PCB. (a.0%; b.5%; c.10%; d.15%).

3. Results and discussions

3.1. Microwave heating mechanism of PCB

3.1.1. The phase composition of PCB

Fig. 11 shows the XRD qualitative analysis results of PCB. A total of three phases were detected in the PCB samples. The main phase is carbon, which is amorphous carbon without a card number, and the secondary phase is silicon (Silicon, syn JCPDS 27–1402) and calcite (Calcite, syn JCPDS 05–0586), represented by (A), (B) and (C), respectively. It can be seen from Fig. 11 that a total of 5 diffraction peaks were detected in the three phases of PCB. Among them, amorphous carbon was flat hump and had no crystal surface.

The research group used X-ray fluorescence (XRF) to test the chemical composition of the PCB in the early stage, and found that the carbon element in the PCB accounted for 95.2%, and the silicon element accounted for 2.8%, and the rest accounted for 2%. The specific surface area of the PCB is 68.353 m^2g. Combined with the XRD qualitative analysis results of the PCB, it was concluded that the main chemical elements of the PCB are carbon and silicon. The microwave absorbability of PCB can be analyzed from three aspects: carbon element, silicon element and silicon carbide. In terms of carbon elements, 99%

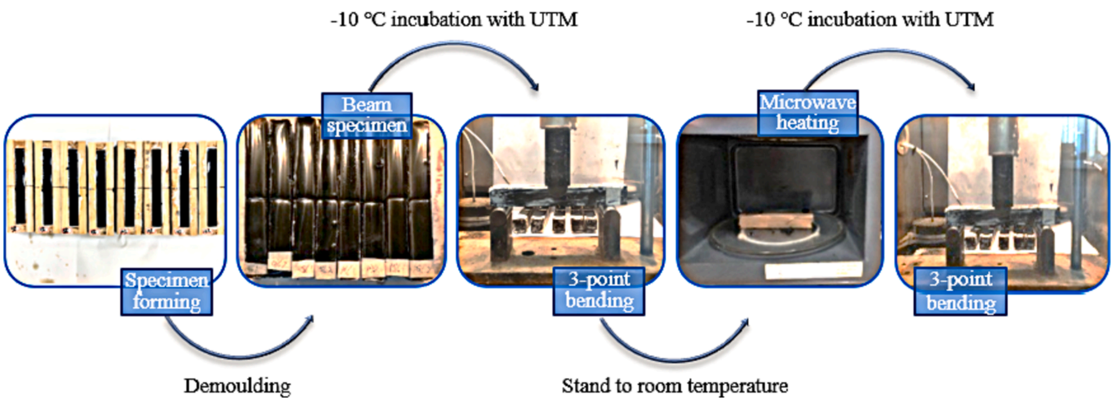

Fig. 9. Healing cycle of bitumen.

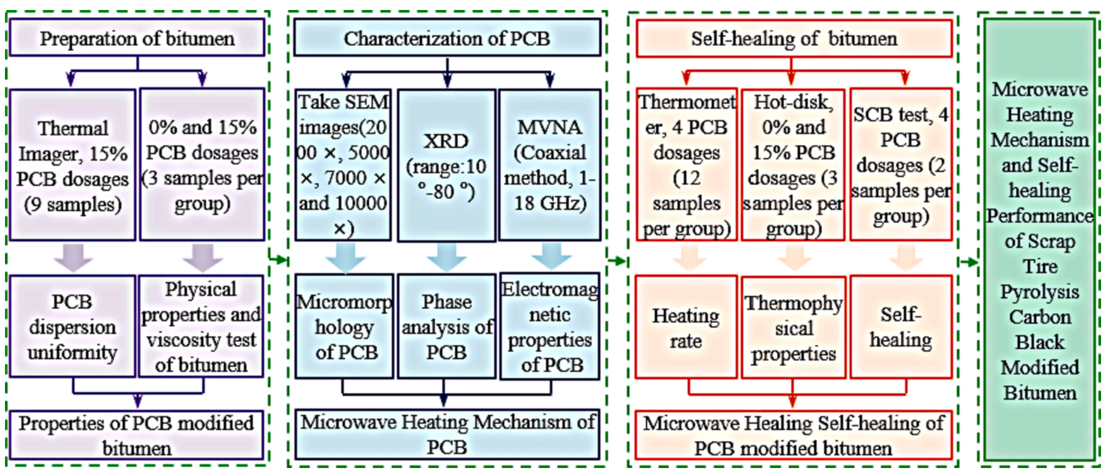

Fig. 10. Technical map.

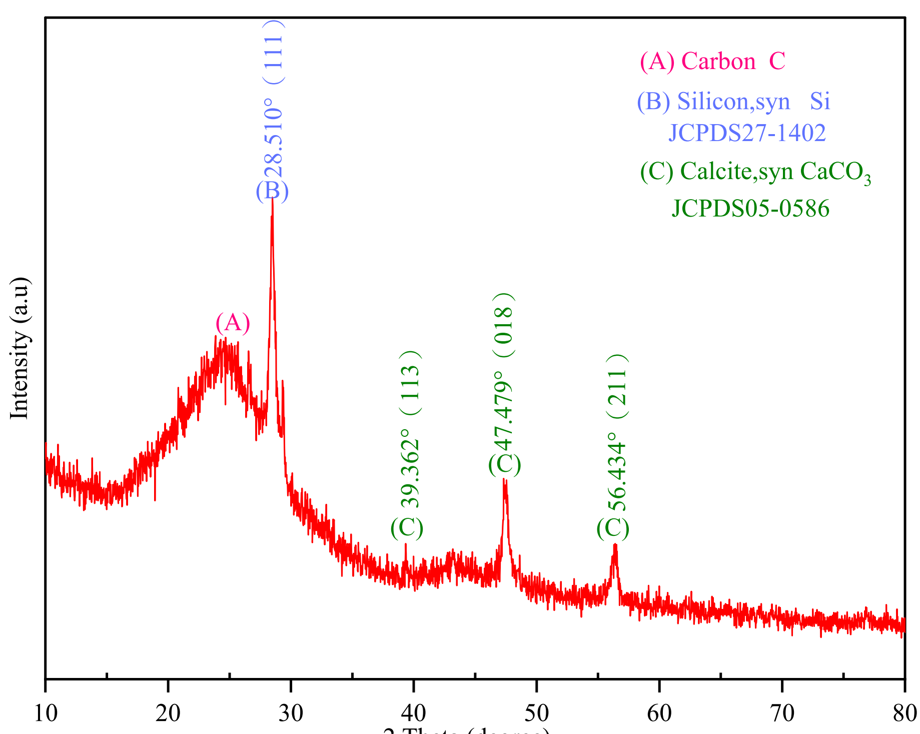

Fig. 11. Qualitative analysis of PCB by XRD.

carbon-based microwave absorbing materials such as carbon fiber [60,61], carbon nanotube[62,63] and graphene[64] absorb microwave and generate heat in the way of dielectric loss under the action of the microwave. It can be seen that carbon element absorbs microwave in the way of dielectric loss. In terms of silicon element, Luo[65] tested the electromagnetic parameters of silicon powder and concluded that silicon powder absorbs microwave in the way of dielectric loss, and the microwave absorbing performance of silicon powder is good. Under the action of microwave, the heating rate of silicon powder was 2.3 to 4.6 times higher than that of traditional heating methods, and the heating rate of silicon powder increased significantly with the increase of microwave power[66]. Kamalieet[67] also concluded that silicon has dielectric properties. It can be seen that silicon also absorbs microwave in the way of dielectric loss. In related studies, silicon carbide (SiC) was also used to improve the microwave absorbing performance of materials [68,69]. Therefore, carbon, silicon and SiC can absorb microwave heat, that is, PCB can absorb microwave heat.

3.1.2. Electromagnetic properties of PCB

By analyzing the electromagnetic parameters of PCB, the mode of microwave energy to the heat energy of PCB under microwave radiation was studied. Fig. 12 shows the test results of the dielectric loss tangent and magnetic loss tangent of PCB. In the range of 1 to 18 GHz, the dielectric loss tangent value and magnetic loss tangent value of PCB showed a wavy decline as a whole, indicating that the dielectric loss and magnetic loss of PCB to microwave decreases as the microwave frequency increases, and the dielectric loss angle tangent was greater than the magnetic loss angle tangent. At 2.45 GHz, the difference between the dielectric loss tangent and the magnetic loss tangent was 0.13. According to the tangent value of dielectric loss and magnetic loss, the absorbing material can be identified as dielectric loss or magnetic loss microwave absorbing material. In the range of 1 to 18 GHz, there was a large difference between the dielectric loss angle tangent and the magnetic loss angle tangent, and the magnetic loss had only a small contribution, which can be ignored. Therefore, it can be judged that PCB was a dielectric loss microwave absorbing material. According to the XRD analysis results of PCB, the dielectric loss of PCB was mainly produced by phase carbon and silicon. Fig. 13 shows the test results of the loss tangent of PCB. The loss tangent value generally decreases with increasing frequency. It showed that the microwave loss of PCB decreases with the increase of microwave frequency.

Fig. 14 shows the test results of the real part and imaginary part of the dielectric constant of PCB. The changing trend of the real part ε' and the imaginary part ε" of the dielectric constant of PCB were the same, and the whole was a fluctuating decrease, indicating that the PCB had charge transfer and dielectric loss under the action of the microwave. The charge storage capacity and dielectric loss capacity of PCB under the action of microwave weakened with the increase of frequency. The real part ε' of the dielectric constant of PCB began to decrease slowly after 9 GHz, and the imaginary part ε" began to decrease slowly after 5 GHz, both of which showed obvious numerical fluctuations in the range of 14 to 18 GHz. The real and imaginary parts of the dielectric constants of PCB were in the range of 1 to 18 GHz, and the overall value differed by about 10. The content of carbon element in PCB is more than 80%, resulting in a large difference between the real part and the imaginary part of the dielectric constant of PCB, so PCB has a strong charge storage capacity. Fig. 15 shows the test results of the real and imaginary parts of the magnetic permeability of PCB. The real part μ' and the imaginary part μ" both increased first and then decreased in the range of 1 to 2.45 GHz. The real part μ' of the dielectric constant of PCB fluctuated slightly around the value 1. The imaginary part μ" varied relatively gently in the range of 8 to 14 GHz, and rose in a fluctuating manner in the range of 14 to 18 GHz. The difference between the real and imaginary parts of the permittivity of PCB was about 10 at 2.45 GHz, and the difference between the real and imaginary parts of the magnetic permeability was about 0.8 at 2.45 GHz. The imaginary part μ" of the magnetic permeability of PCB decreased with the increase of frequency as a whole, indicating that the magnetic storage capacity and magnetization ability of PCB under the action of microwaves weakened with the increase of frequency.

3.1.3. Electromagnetic loss properties of PCB

Fig. 16 shows the effect of PCB thickness on microwave reflection loss, and the reflection loss of PCB with different thicknesses in the range of 1 to 18 GHz showed numerical fluctuations. When the thickness of the PCB coating was 2 mm, the maximum absorption and attenuation of

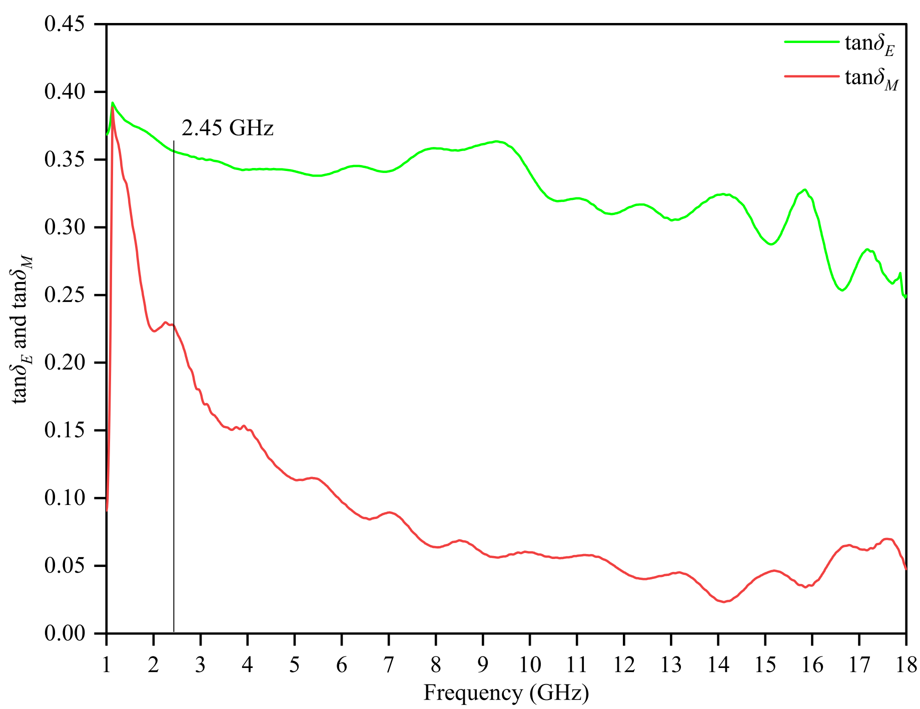

Fig. 12. Test results of the dielectric loss tangent and magnetic loss tangent of PCB.

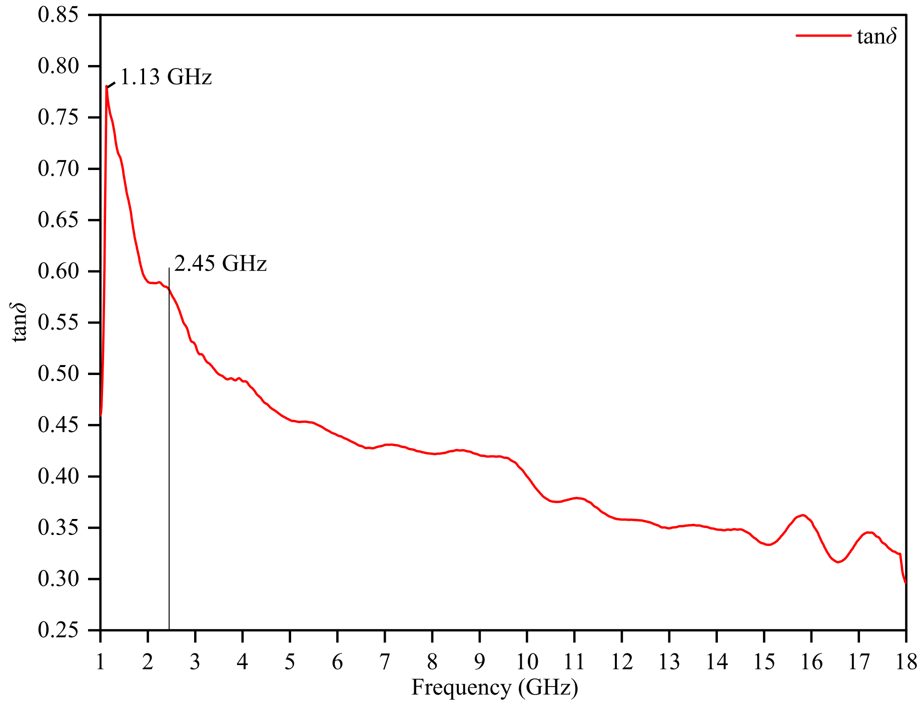

Fig. 13. Test results of loss tangent of PCB.

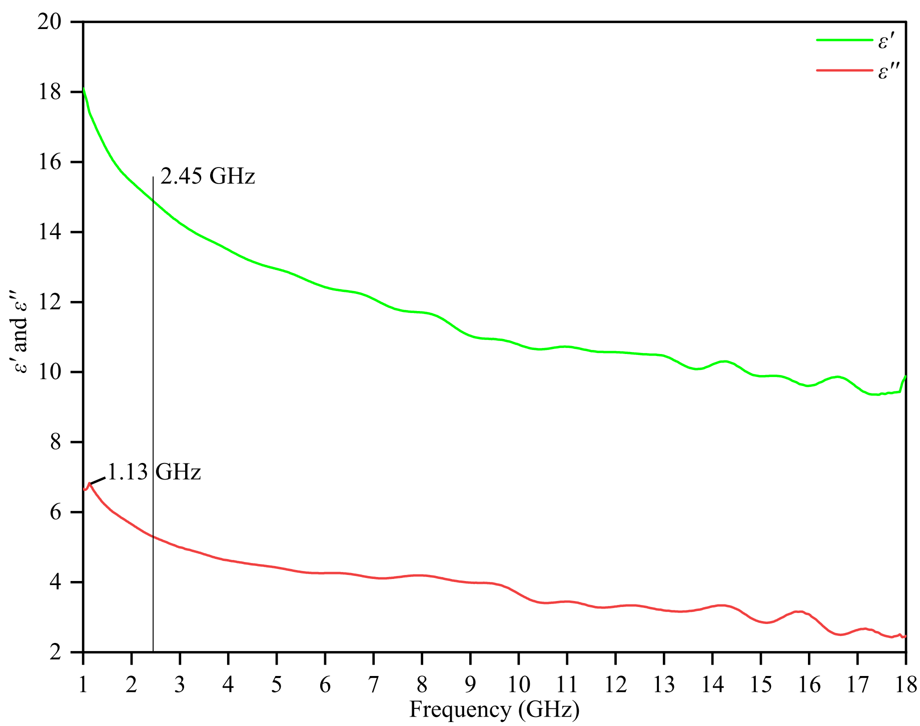

Fig. 14. Test results of real and imaginary parts of the dielectric constant of PCB.

electromagnetic waves occurred, and the reflection loss was about −30 dB. In the range of 1 to 18 GHz, the absorbing properties of PCB varied with microwave frequency and thickness, and the reflection loss curve fluctuated greatly. When the thickness of the PCB coating was less than or equal to 1 mm, the numerical fluctuation was relatively insignificant, and the frequency range where the reflection loss was lower than −5 dB did not appear on the two reflection loss curves. It showed that when the thickness of PCB coating was less than or equal to 1 mm, only 68% of electromagnetic waves can be absorbed and attenuated. When the thickness of the PCB coating was between 1.5 mm and 4 mm, the frequency range where the reflection loss was lower than −10 dB appeared on the reflection loss curve. In this microwave frequency range, the PCB coating of the test thickness can absorb and attenuate 90% of the electromagnetic waves. Eight groups of samples with different thicknesses of PCB coating had poor absorbing performance in the low-frequency range of 1 to 4 GHz. It can be seen that when the thickness of the PCB

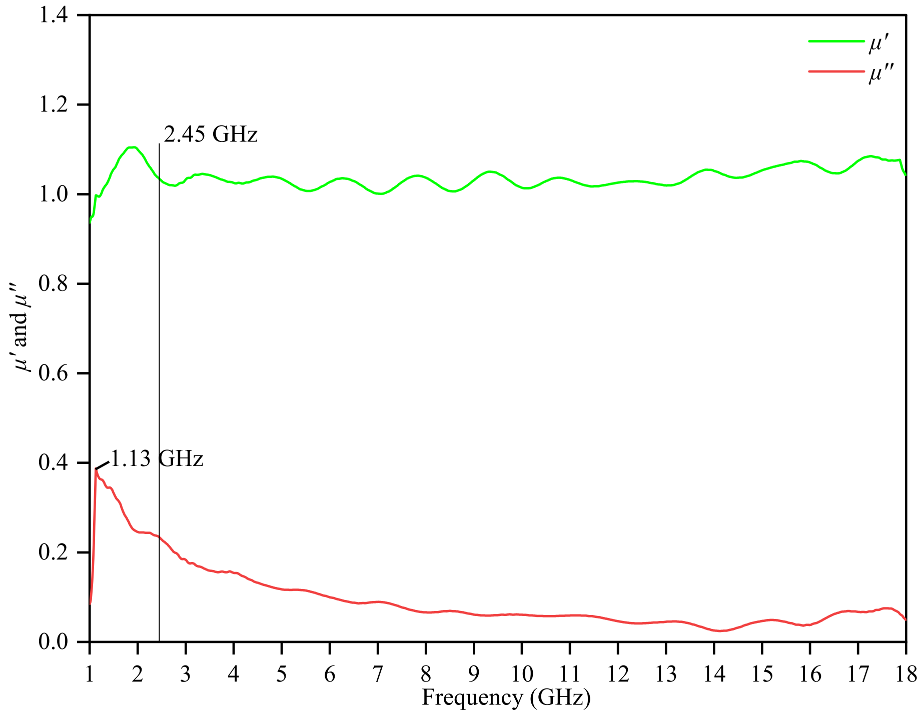

Fig. 15. Test results of real and imaginary parts of magnetic permeability of PCB.

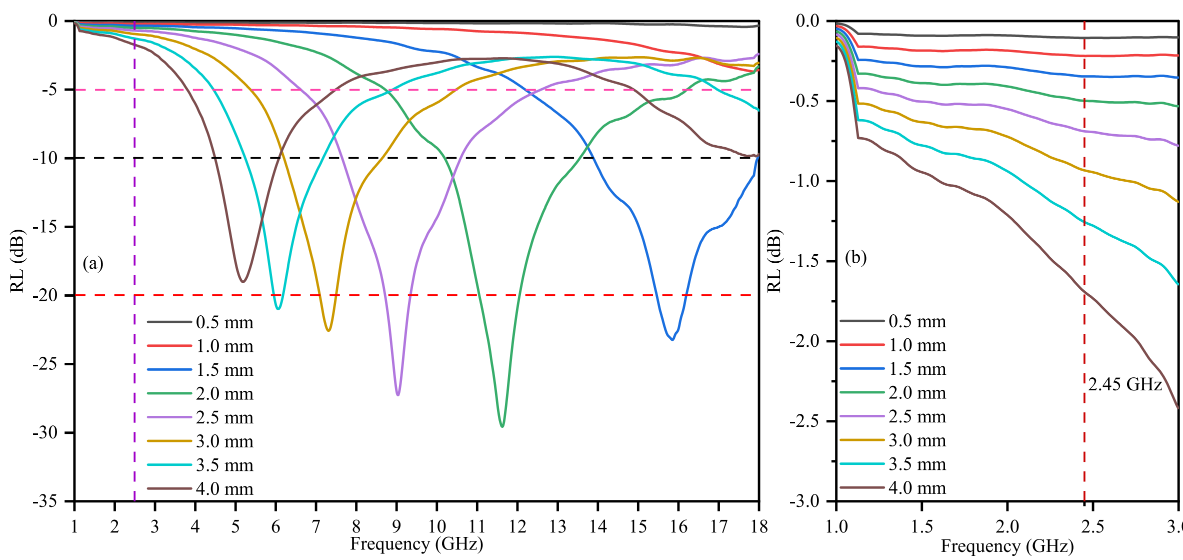

Fig. 16. Effect of PCB thickness on microwave reflection loss (a.1 to 18 GHz; b. 1 to 3 GHz).

coating was 2 to 4 mm, the abscissa and ordinate values of the maximum reflection loss decreased with the increase of the thickness, and the effective absorption band widened with the increase of the thickness. When the thickness of the PCB coating was 1.5 to 3.5 mm, the reflection loss curves showed a frequency range where the reflection loss was lower than −20 dB. The current thickness of PCB coating can absorb and attenuate 99% of electromagnetic waves in this frequency range. It can be seen that when the thickness of the PCB coating was 1.5 to 4.0 mm, the absorbing performance of the PCB was better, and when the thickness of the PCB coating was less than 1.0 mm, the absorbing performance of the PCB was poor. In general, the microwave absorption performance of PCB was better as a whole.

In summary, the strong dielectric loss characteristics of PCB can be used to enhance the overall dielectric loss performance of bitumen. PCB absorbed microwave energy to generate a lot of heat, and at the same time, the heat of PCB was transferred to the non-absorbing bitumen through heat transfer, which was to generate dielectric loss inside the PCB modified bitumen, so that the PCB modified bitumen can be heated by microwaves.

3.2. Dispersion uniformity of PCB modified bitumen

Fig. 17 shows the temperature distribution results of the bitumen. The stacked graph represents the temperature distribution by the number of pixels, the X-axis is the initial value of each temperature interval, and the Y-axis is the number of pixels. When the stirring time was within 5 to 30 min, the range of the starting and ending temperature was roughly reduced with the increase of the stirring time, and the

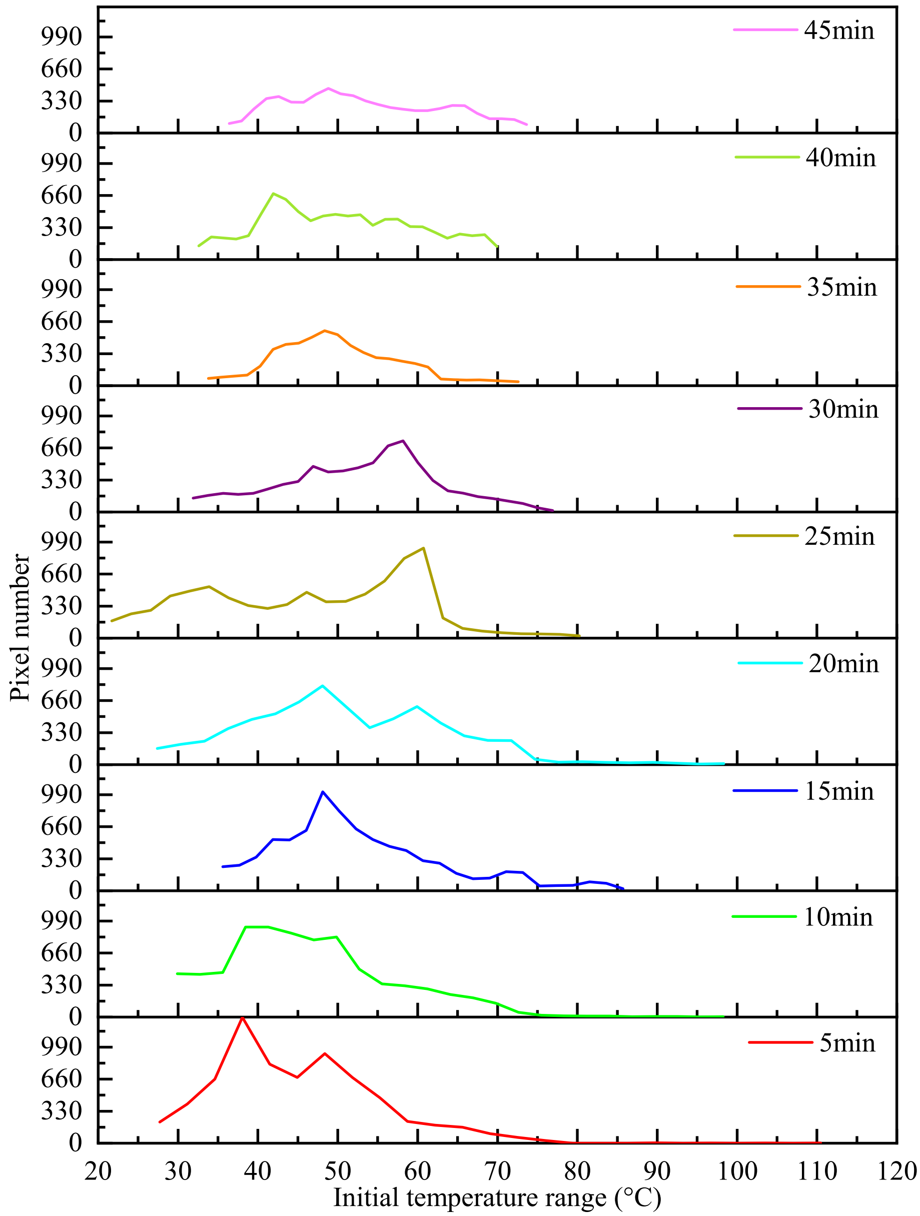

Fig. 17. Temperature distribution of test piece.

dispersion uniformity of the PCB was better. With the increase of stirring time, the peak of the number of pixels of the specimen moved to the right, and when the stirring time reached 35 min, the peak of the number of pixels of the specimen did not move significantly. When the stirring time was 35 to 45 min, there was no significant change in the temperature range between the beginning and end.

Fig. 18 shows the effect of stirring time on the dispersion uniformity of carbon black. After 10 min of stirring, the standard deviation in the 10th minute was 90.19% of that in the 5th minute. After 30 min of stirring, the standard deviation at the 30th minute was 52.22% of that at the 5th minute. After 45 min of stirring, the standard deviation at 45 min was 27.97% of that at 5 min. With the increase of mixing time, the smaller the standard deviation of surface temperature distribution, the better the uniformity of temperature distribution, indicating that the better the dispersion of PCB in bitumen.

When the stirring time was 45 min, the dispersion uniformity of PCB was improved compared with 35 min and 40 min. When the stirring time was 25 min, the dispersion uniformity of PCB was not significantly improved compared with 15 min and 20 min. The stirring time of 40 min

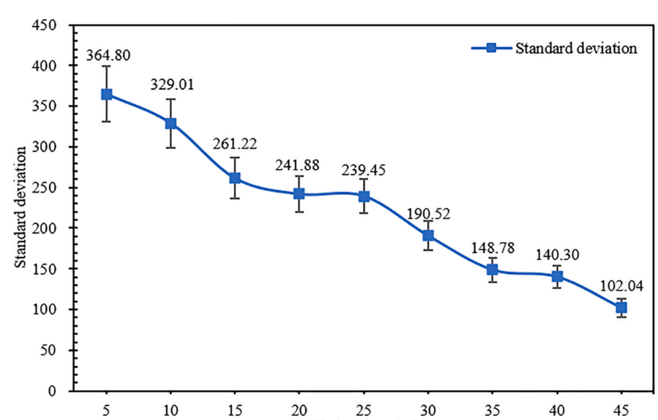

Fig. 18. Effect of stirring time on uniformity of PCB.

was selected as the preparation parameter of PCB. This not only satisfies the condition that 35 min has been relatively uniform, but adding 5 min stirring time based on 35 min will make the bitumen more uniform to be sufficient uniformity, and avoiding too long stirring time has no significant help to the uniformity. The modified bitumen used in other tests in this study was stirred at 500 rpm for 40 min.

3.3. Microwave heating self-healing of PCB modified bitumen

3.3.1. Heating rate

Fig. 19 shows the effect of PCB dosage on bitumen under microwave heating. Under the action of microwave heating with a power of 800 W, the heating rate of 70# base bitumen was slow, with only a small change, and the temperature was only 41.8 °C at 120 s. When the microwave heating power was constant (800 W), with the increase of the dosage of PCB, the heating rate of the temperature of the bitumen also increased. The heating rate of the bitumen with 15%, 10% and 5% PCB was 590%, 438% and 258% higher than that of 70# bitumen respectively. For every 5% increase in the dosage of PCB, the temperature of the bitumen at 120 s increased by 20 to 30 °C, indicating that PCB can significantly increase the microwave heating rate of the 70# base bitumen.

Fig. 20 shows the effect of microwave heating power on 15% PCB modified bitumen. The temperature change of 15% PCB modified bitumen after microwave heating under the power of 160 W was the smoothest, and that under the power of 800 W was the most obvious. When the dosage of PCB in bitumen was certain, the growth rate of bitumen slurry temperature increased with the increase of power. The heating rates of 800 W, 640 W, 480 W and 320 W were increased by 565%, 436%, 246% and 101% respectively compared with 160 W. The higher the microwave power, the higher the temperature of the bitumen after heating.

3.3.2. Thermophysical performance parameters

Fig. 21 shows the thermophysical performance test results of 70# base bitumen and 15% PCB modified bitumen. At the test temperature of 60 °C, the thermal conductivity, thermal diffusion coefficient and specific heat capacity of 15% PCB modified bitumen were higher than 70# base bitumen, and the thermal conductivity was increased by 0.01153 W/m•k, indicating that the thermal conductivity of 15% PCB modified bitumen was strong, while the thermal insulation ability was weak. The thermal diffusivity increased by 0.0140 mm^2/s, indicating that the heat dissipation ability of the 15% PCB modified bitumen was good, which was conducive to the rapid transfer of the heat generated by the PCB due to wave absorption to the surrounding bitumen, improving the fluidity of the bitumen. It can be seen that in the process of pavement construction, the PCB modified bitumen can be cooled quickly in the natural

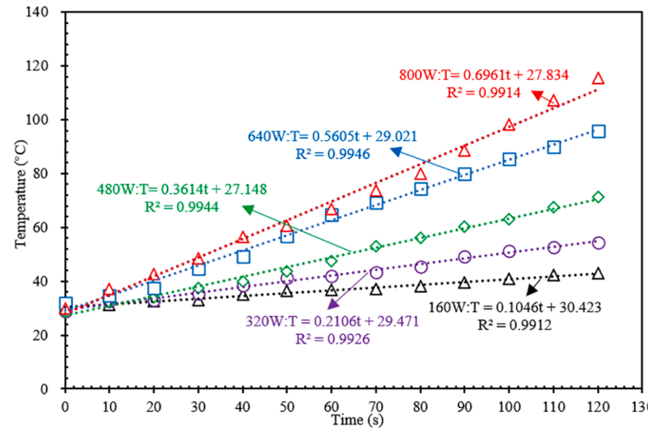

Fig. 20. Effect of microwave heating power on 15% PCB modified bitumen.

state to reduce the construction and maintenance time, and was more suitable for road maintenance than conventional asphalt mixture. The specific heat capacity increased by 0.0224 MJ/m^3K, indicating that the 15% PCB modified bitumen had better heat storage capacity, which was conducive to rapid temperature rise. Therefore, PCB had the advantage of improving the thermal conductivity and heat storage capacity of bitumen.

3.4. Self-healing properties of PCB modified bitumen

Fig. 22 shows the test results of bitumen self-healing rate with different dosages of PCB. 15% PCB modified bitumen had the best self-healing performance and 70# base bitumen had the worst self-healing performance. After 120 s of microwave heating, 70# base bitumen hardly heated up, and PCB modified bitumen heated up rapidly after rapid wave absorption. It can be seen that PCB modified bitumen enhanced its self-healing performance by softening and flowing. The self-healing rates of 5%, 10% and 15% PCB modified bitumen were 188%, 290% and 359% higher than 70# base bitumen respectively. Compared with the effect of adjacent PCB samples on bitumen self-healing, the self-healing rate of 10% PCB modified bitumen was 35% higher than that of 5% PCB modified bitumen, and the self-healing rate of 15% PCB modified bitumen was only 18% higher than that of 10% PCB modified bitumen. It can be seen that with the increase of the dosage of PCB, the self-healing rate of bitumen after microwave heating continued to improve, and 5% PCB improved the self-healing rate of bitumen most significantly.

4. Conclusions

The PCB of scrap tire was added into bitumen to prepare bitumen with microwave absorbing ability, which was intended to improve the microwave heating self-healing performance of bitumen. Through the physicochemical and electromagnetic properties of PCB admixture, the microwave heating mechanism of PCB was revealed. Through heating test and microwave self-healing test, it was verified that PCB can improve the self-healing rate of modified bitumen.

(1) The main phases of PCB were carbon and silicon which can absorb microwave, so PCB can absorb microwave. In the range of 1 to 18 GHz, it's $\tan\delta_E > \tan\delta_M$, which showed that the PCB absorbs microwave energy mainly in the form of dielectric losses. And the overall difference between the tangent of dielectric loss Angle and the tangent of magnetic loss Angle was large, so there was basically no magnetic loss. The thickness of the PCB coating and the microwave frequency had obvious effects on the microwave absorbing properties of the PCB. When the thickness of the PCB coating was in the range of 1.5 to 4 mm, there was an effective bandwidth. The absorbing material can absorb and

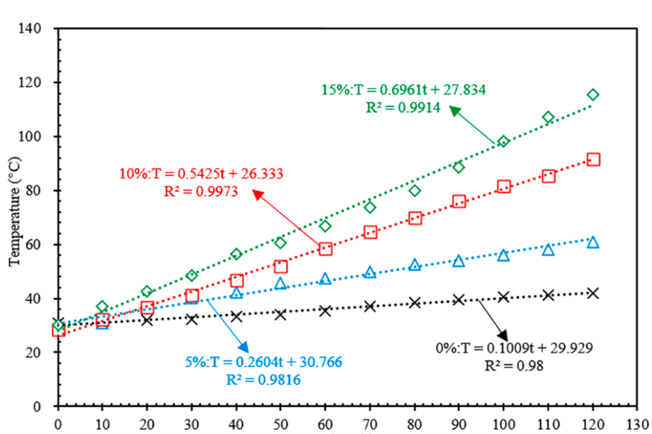

Fig. 19. Effect of PCB dosage on bitumen under microwave heating.

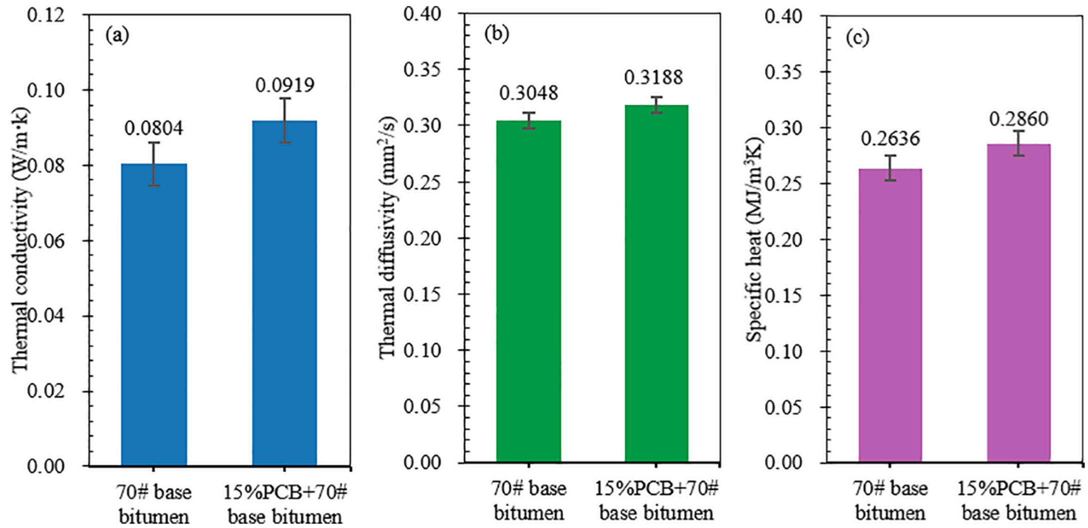

Fig. 21. Thermophysical properties of 70# base bitumen and 15% PCB modified bitumen. (a. Thermal conductivity; b. Thermal diffusivity; c. Specific heat).

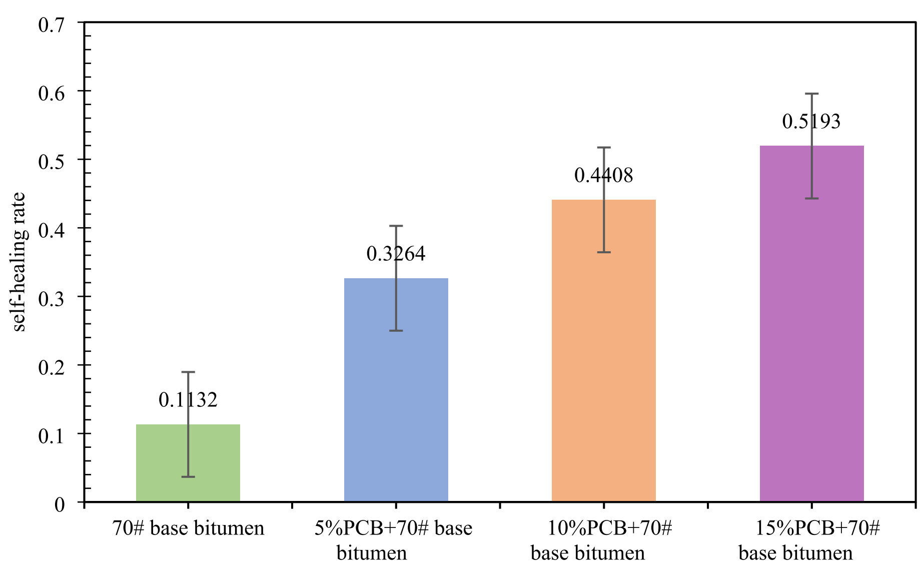

Fig. 22. Effect of PCB dosage on microwave healing properties of bitumen.

attenuate more than 90% of the electromagnetic wave within the effective bandwidth. Based on the above mechanism analysis, PCB had good microwave absorbing performance and was a strong dielectric loss microwave absorbing material. Under the action of the microwave, the PCB in the bitumen produces dielectric loss and absorbs the microwave to heat up, and the heat energy is further diffused to the bitumen that cannot absorb the microwave.

(2) When the stirring time was in the range of 5 to 40 min, the uniformity of the 15% PCB modified bitumen increased continuously with the increase of the stirring time. The recommended minimum stirring time for preparing PCB modified bitumen was 40 min. The PCB evenly existed in the bitumen, and the heat energy generated by absorbing microwaves is quickly transferred to the bitumen, which increases the temperature of the bitumen. The addition of PCB to the bitumen had a significant effect on the ductility and viscosity of the bitumen. The PCB improved the high-temperature stability of the bitumen and reduces the low-temperature cracking resistance, but it still met the specification requirements. Because the PCB absorbed a certain amount of energy and also increased the thermal diffusivity of bitumen, lead to the optimum mixing and compaction temperatures of 15% PCB modified bitumen were higher than those of 70# base bitumen.

(3) The microwave power, the dosage of PCB and the heating time all had a significant effect on the temperature rise of the bitumen. At 800 W power, adding PCB with 5%, 10% and 15% bitumen mass can increase the heating rate of the bitumen by 2.58 times, 4.38 times and 5.90 times respectively. When the dosage of PCB was certain, based on 160 W microwave power, the heating rate of bitumen can be increased by 155% on average for each additional 160 W microwave power. When the microwave power and the dosage of PCB were constant, the temperature of the modified bitumen had a linear positive correlation with the heating time. The thermal conductivity and heat storage capacity of the modified bitumen after adding 15% PCB were both improved, PCB was conducive to promoting rapid thermal conduction and heat storage of bitumen. And the improvement of the thermal diffusivity was conducive to the rapid transfer of the heat generated by the PCB to the surrounding bitumen.

(4) PCB in microwave heating, PCB in the heat generation at the same time also in the transfer of heat, so that the surrounding bitumen to obtain a higher temperature. The higher the softening degree of bitumen was, the higher the self-healing rate was. And only adding PCB with 5% bitumen mass can increase the self-healing rate of bitumen by 1.88 times. When the dosages of PCB were 0%, 5%, 10% and 15%, with the

increase of PCB doping, the self-healing effect of the bitumen after microwave heating was continuously improved, of which the addition of 15% PCB increased the self-healing rate of bitumen by 3.59 times.

At this stage, the mechanism analysis of PCB is mainly analyzed through the main phase of PCB. It also has certain other trace substances (chemical composition) that may also affect the absorption of microwave performance of PCB. To reduce the preparation time of modified bitumen, and also to reduce the influence of long-term high-temperature preparation on bitumen aging, the stirring speed during the preparation of modified bitumen can be appropriately increased. Asphalt mixture is the main part of asphalt pavement, this study only studies bitumen, the next step will be to study its microwave self-healing from the perspective of asphalt mixtures.

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CRediT authorship contribution statement

Fu Wang: Methodology, Data curation, Writing – original draft. **Hongbin Zhu:** Data curation, Writing – original draft, Validation. **Yuanyuan Li:** Conceptualization, Methodology, Data curation, Writing – original draft, Writing – review & editing, Supervision, Funding acquisition. **Dengjun Gu:** Methodology, Project administration. **Yangming Gao:** Methodology. **Jianlin Feng:** Resources, Data curation, Software, Validation. **Benan Shu:** Methodology, Data curation. **Chao Li:** Methodology, Writing – review & editing. **Shaopeng Wu:** Methodology, Validation, Funding acquisition. **Quantao Liu:** Data curation, Software. **Zixuan Xu:** Data curation, Software.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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