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Research on Microscopic Characteristics of Interfacial Transition Zone of Recycled Coarse Aggregate Asphalt Mixture

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Self-introduction

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Introduction

1.1 Research Background

●**The growing problem of construction waste and the need for aggregates**

●The increasing scarcity of natural sand and gravel resources and the growing accumulation of construction waste will gradually become one of the main conflicts in China's construction industry. The largest proportion of construction waste is waste concrete, which is used in the preparation of asphalt mixes through a specific processing process, which can promote the efficient use of waste concrete and reduce the consumption and extraction of natural sand and gravel.

●**Basic properties of recycled aggregates and multi-phase joint interfaces**

●Changes in the surface properties of the recycled coarse aggregate are the main cause of the multiple physical differences between the recycled coarse aggregate, the asphalt slurry surface and the interface between the two phases, and the presence of surface adherent mortar leads to increased micro-damage in the recycled coarse aggregate asphalt mix. The lack of understanding of the microscopic properties of the transition zone of the recycled coarse aggregate - asphalt mastic interface directly affects the study of the mechanical properties and damage patterns of recycled coarse aggregate asphalt mixtures.

1.2 Available research findings

●**Study on the characterisation of recycled aggregate**

●Recycled aggregates are multi-phase composites consisting of aggregates and cement mortars. The adhering mortar layer results in a rough and heterogeneous surface pattern with a large number of pore structures, which leads to a deterioration of the physical and mechanical properties of the recycled aggregate. The interface transition zone has a gradient distribution of mechanical properties which makes it a weak area for recycled aggregate asphalt mixtures.

●**Study on the application of recycled aggregates in asphalt mixes**

●The high temperature stability and stiffness modulus of recycled aggregate asphalt mixes are improved to a certain extent compared to natural aggregate asphalt mixes;

- Low temperature performance, elastic modulus and dynamic modulus decrease;
- Large variability of recycled aggregate leads to great differences in water stability and resilient modulus of asphalt mixture.

1.2 Available research findings

- **Study on Interface Transition Zone**
- Further research on recycled aggregate asphalt mixture shows that the bearing capacity is directly related to the bond strength of aggregate-asphalt mortar interface, and the newly introduced recycled aggregate-asphalt mortar interface is more complex and changeable than the natural aggregate-asphalt mortar interface, which is the key factor affecting its performance variation.
- The interfacial zone in the RCAM was divided into the mortar-natural aggregate interface transition zone (M-N ITZ) and mortar-asphalt mastic interface transition zone (M-A ITZ).
- **Study on Microscopic Test Technology and Characterization Method of Asphalt Mixture**
- The microstructure of the interfacial transition zone in asphalt mixtures can be explored by several instrumental techniques (SEM, NI, X-ray, etc.) to help understand the complex mechanical behaviour of the interfacial transition zone of recycled aggregate asphalt mixtures.
- At present, the research on the micromechanical properties of nano-scale interface transition zone of recycled aggregate asphalt mixture is still very limited.

1.3 Research significance

- Two kinds of interface transition zones are effectively identified to realize the quantitative characterization of the interface transition zone, and the microscopic characteristics of the thickness, average modulus, chemical properties and structural composition of the interface transition zone are obtained. The insufficient bearing capacity and failure cause of recycled aggregate asphalt mixture are explained. The causes of the weak interfacial zone between recycled aggregate and asphalt mortar are expounded, and the influence mechanism of interfacial transition zone on the performance of recycled coarse aggregate asphalt mixture is further studied.

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Materials and mixtures design

2.1 Materials

●RCA

The aggregates used in this experiment include crushed production of RCAs and natural aggregates. Among them, RCAs are all derived from waste cement concrete panels. RCAs in different particle size ranges are shown in Fig.1. The particle size range of the recycled aggregates used in this study is 9.5-26.5mm.

(a)26.5-31.5mm (b) 9.5-26.5mm (c) 0.75-9.5mm

RCAs in different particle size ranges

Analysis of technical specifications for RCA

●**Physical and mechanical properties of RCA**

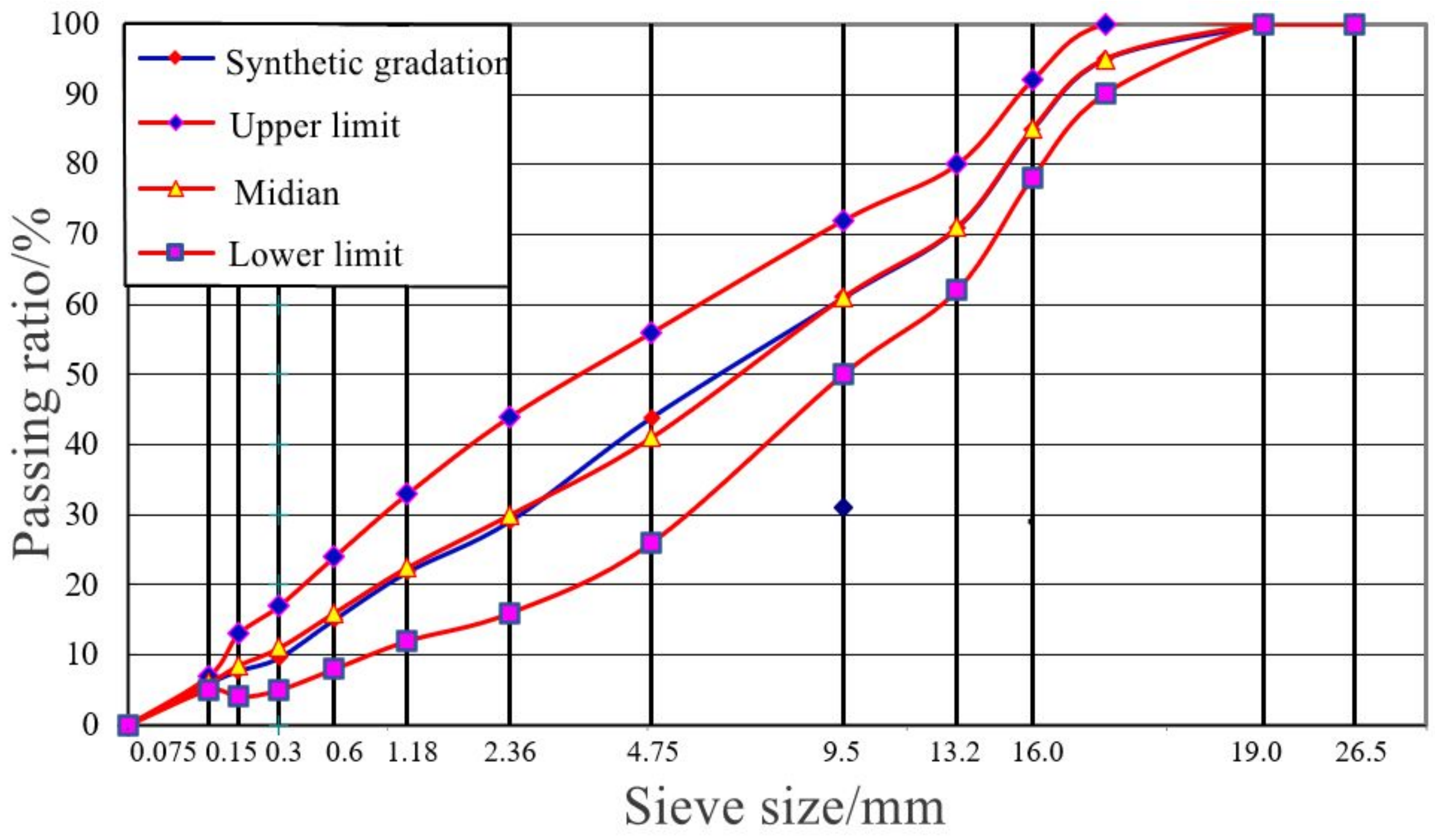
●The gross bulk density, clay content and needle flakes of RCAs all meet the requirements of the specification for first class RCA. The crushing values, wear loss rates and firmness of the recycled coarse aggregates are greater than those of the natural aggregates due to the amount of mortar and microcracking present in the RCA. Overall, the basic properties of recycled coarse aggregates in the range of 9.5-26.5 mm meet the requirements of asphalt mixes.

Technical Indices	Particle size range /mm							
	RCA				NA			
	9.5-13.2	13.2-16	16-19	19-26.5	9.5-13.2	13.2-16	16-19	19-26.5
Apparent relative density	2.686	2.679	2.689	2.691	2.716	2.715	2.707	2.716
Relative density of gross volume	2.295	2.323	2.395	2.450	2.677	2.683	2.685	2.699
Water absorption/%	6.34	5.72	4.57	3.66	0.52	0.45	0.30	0.23
Mortar adhesion rate /%	31.5	29.4	33.6	29.5	/	/	/	/
Crush value /%	21.3	23.5	22.8	24.4	10.0	10.4	9.9	10.4
Abrasion value /%	25.0	24.2	24.6	22.1	18.0	17.4	17..9	18.4

2.2 Mixture design

- To keep RCAM from cracking, rutting, and moisture damage, the asphalt binder used should have a high viscosity and good adhesion with aggregate. The asphalt binder selected was styrene butadiene styrene (SBS) modified asphalt. The limestone powder produced by Zhenjiang Gaozi is selected as the ore powder.

- In this study, RCAs ranging from 9.5 to 26.5 mm were selected to replace natural aggregates of the corresponding particle size according to the AC-20 gradation. The total replacement rate was 39%. Using the standard Marshall test method, it is determined that the best oil-stone ratio of natural aggregate AC-20 is 4.4%, the best oil-stone ratio of RCA AC-20 is 4.9%,



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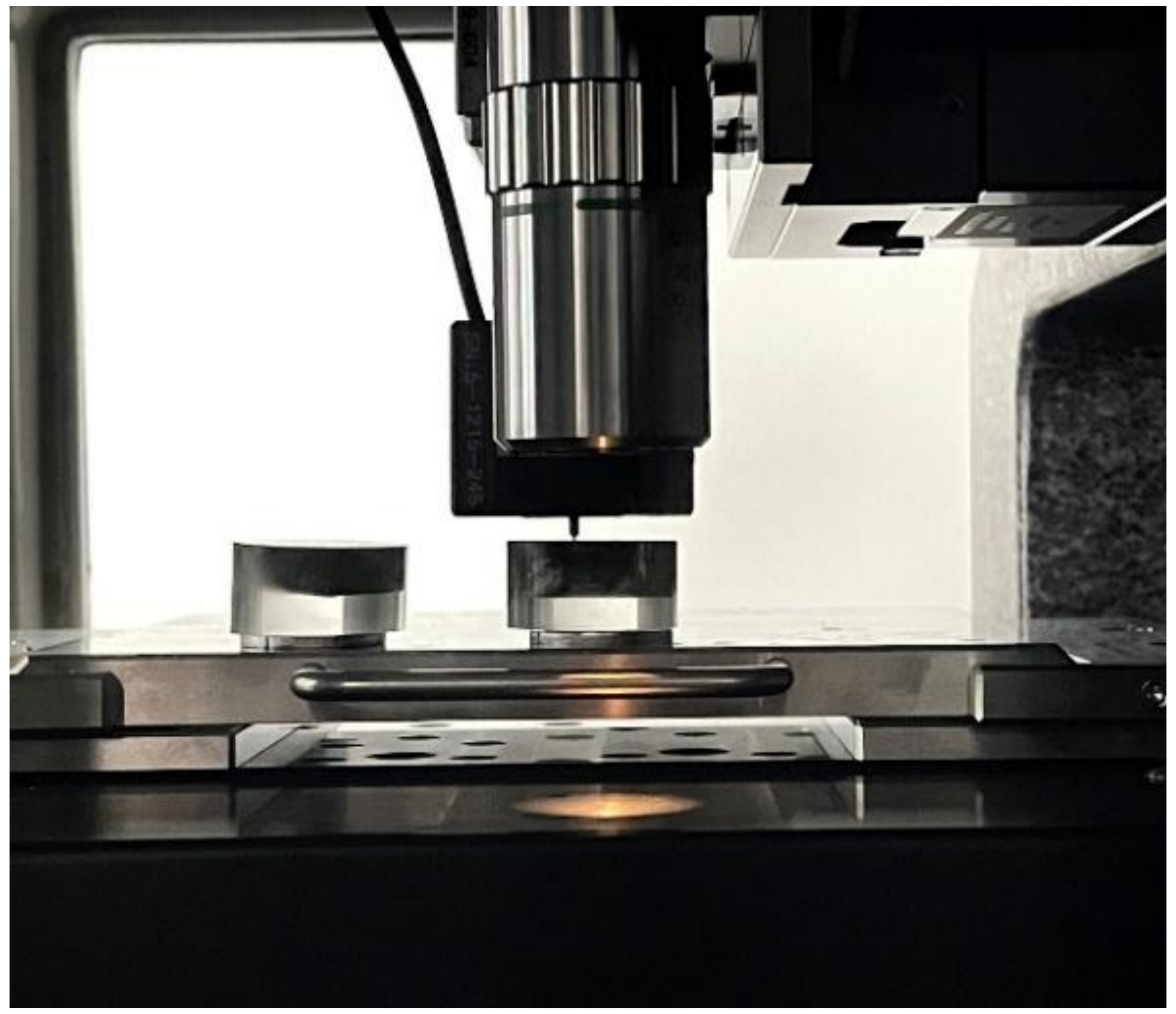
Methods

3.1 Nanoindentation test

Nanoindentation, as a new type of micro-mechanical performance testing technology suiTable for multi-phase mixtures, can identify and characterize multiple phases of asphalt mixtures and characterize the changes in mechanical properties of the transition zone between the new and old interfaces. As shown in Fig. 4, this study used Hysitron Triboindenter (TI950) to test the test piece, to determine the nanomechanical properties of the ITZ.



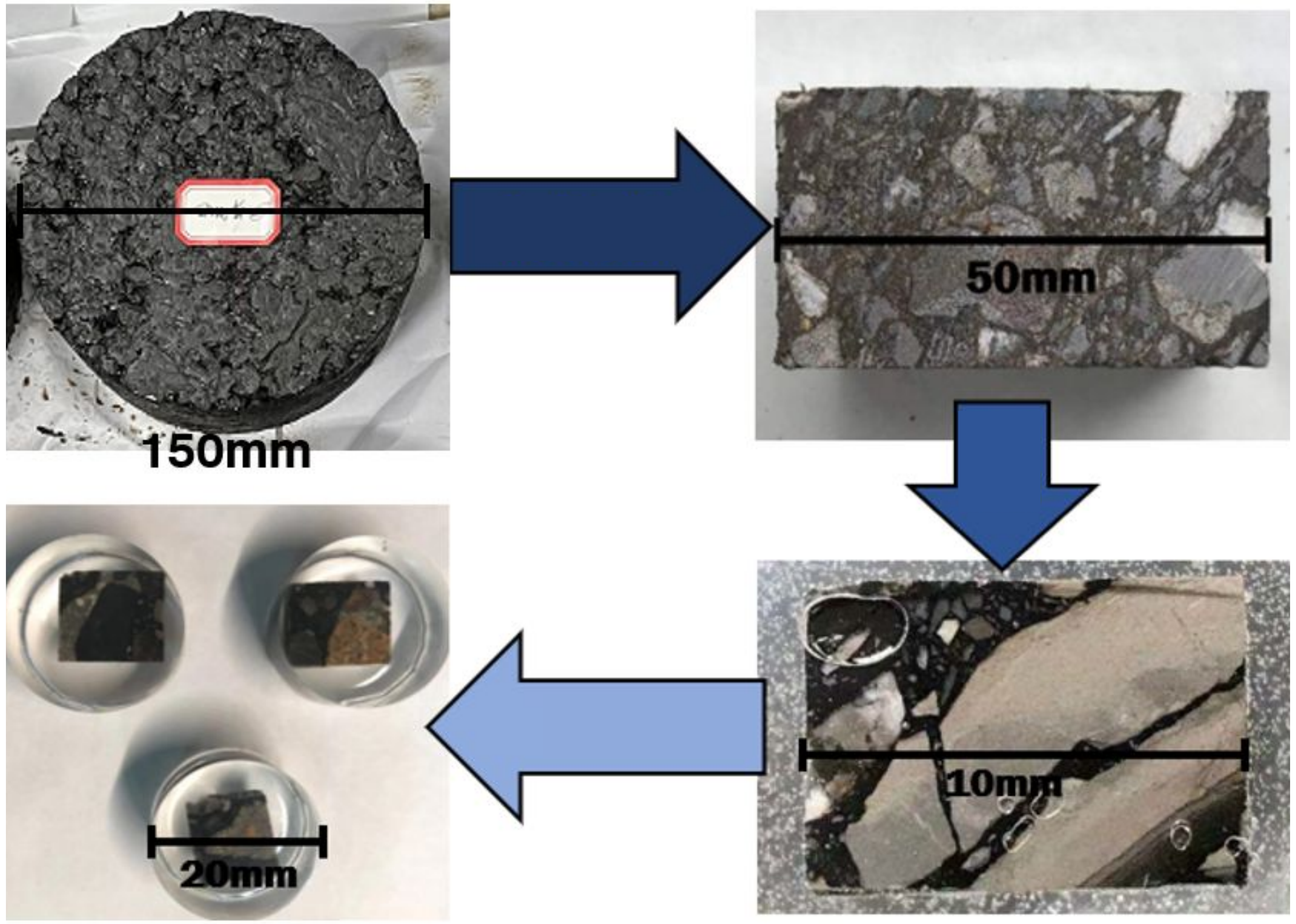
3.1.1 Ni test sample preparation

NI test accuracy has a high correlation with the flatness of the specimen surface. Therefore, it is important to try to eliminate the negative effects of uneven surfaces on test results during sample preparation.

(1) **Mixture forming**

(2) **Precision cutting**

(3) **Cured with epoxy resin**

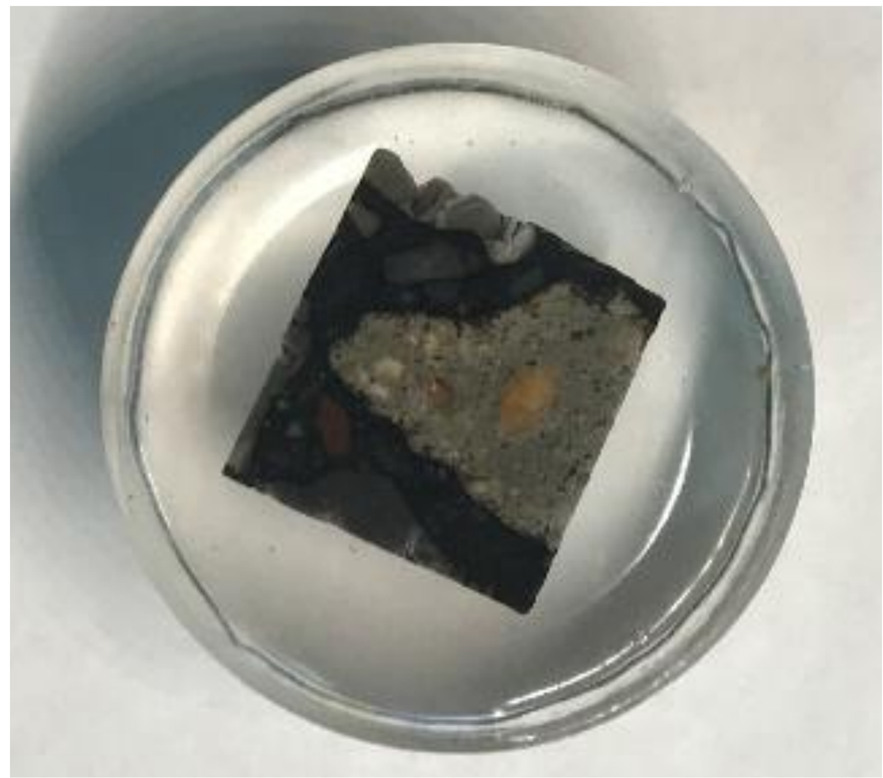


3.1.1 Ni test sample preparation

(4) Surface grinding

(5) Polishing

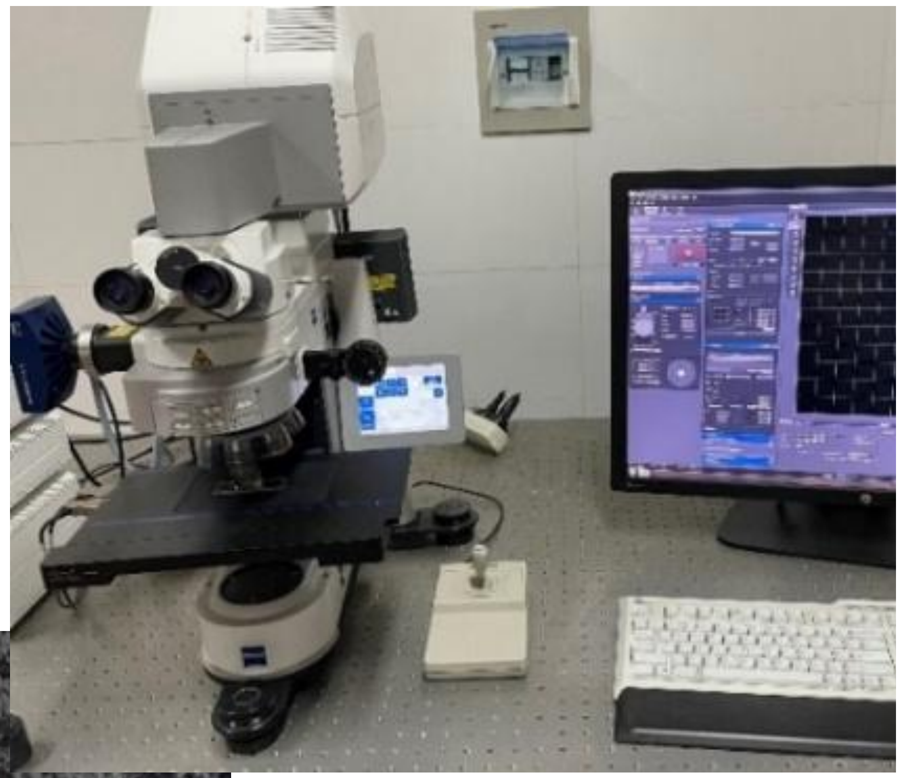
(6) Cleaning and drying

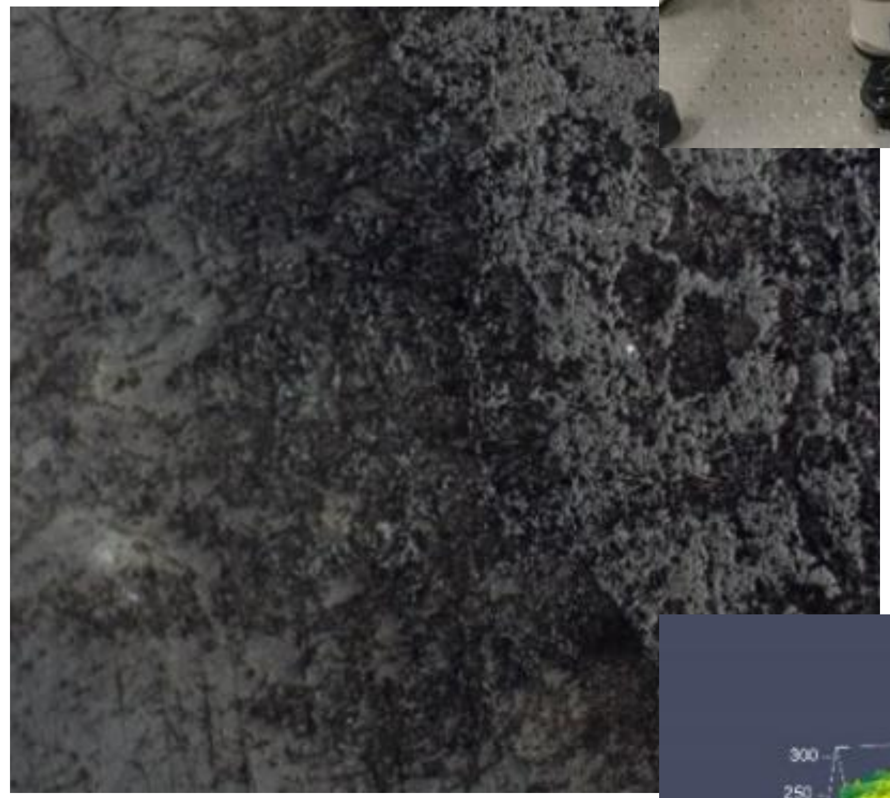


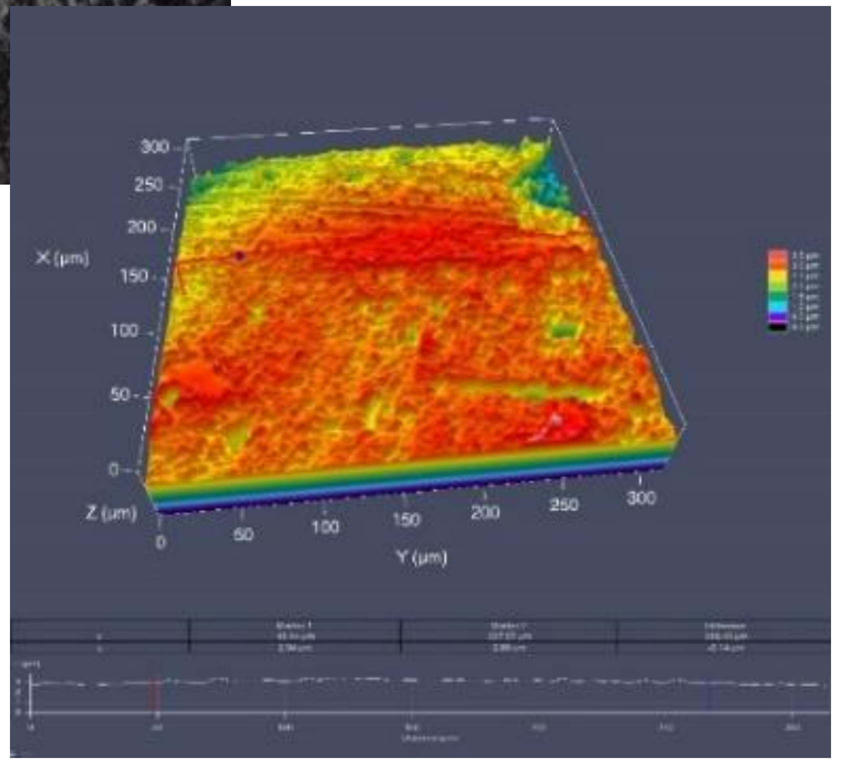
3.1.2 Elastic modulus

Surface roughness testing of specimens

To ensure the surface quality after polishing and grinding, the polished samples were further evaluated by 3D confocal laser microscopy to obtain information on the surface topography. The scans carried out within the specimen covered a range of 300 x 300 μm and a 2D visual map of the test area around the test area with 3D topography is shown in the figure below. The root mean square (RMS) of the surface topography was chosen to measure the surface roughness. An average RMS roughness of 150nm after polishing of the test piece is generally considered sufficient. The test results show that the average RMS roughness of the polished specimens in this test is 127.03 nm, which satisfies the requirements of the scored specimens.







3.1.2 Elastic modulus

The significant viscoelastic characteristics of recycled coarse aggregate asphalt materials pose difficulties for the application of nanoindentation techniques: on the one hand, due to the creeping properties of the asphalt; on the other hand, the multiple jamming of the cement mortar surface can easily cause the indenter to tilt, deviate from the test area or interrupt the experiments being carried out. In order to achieve a long duration of the experiment, avoid deviations in the test area and data variability, it is essential to set the test parameters in advance.

Test parameters

Loading indenter: Berkovich tip and modified bitumen was used for the test.

Maximum controlled load: The maximum load of the asphalt mix is suitable between 3000-5000 μN.

Loading and unloading rate: The single loading time is set to end within 20 s. The unloading rate is the same as the loading rate.

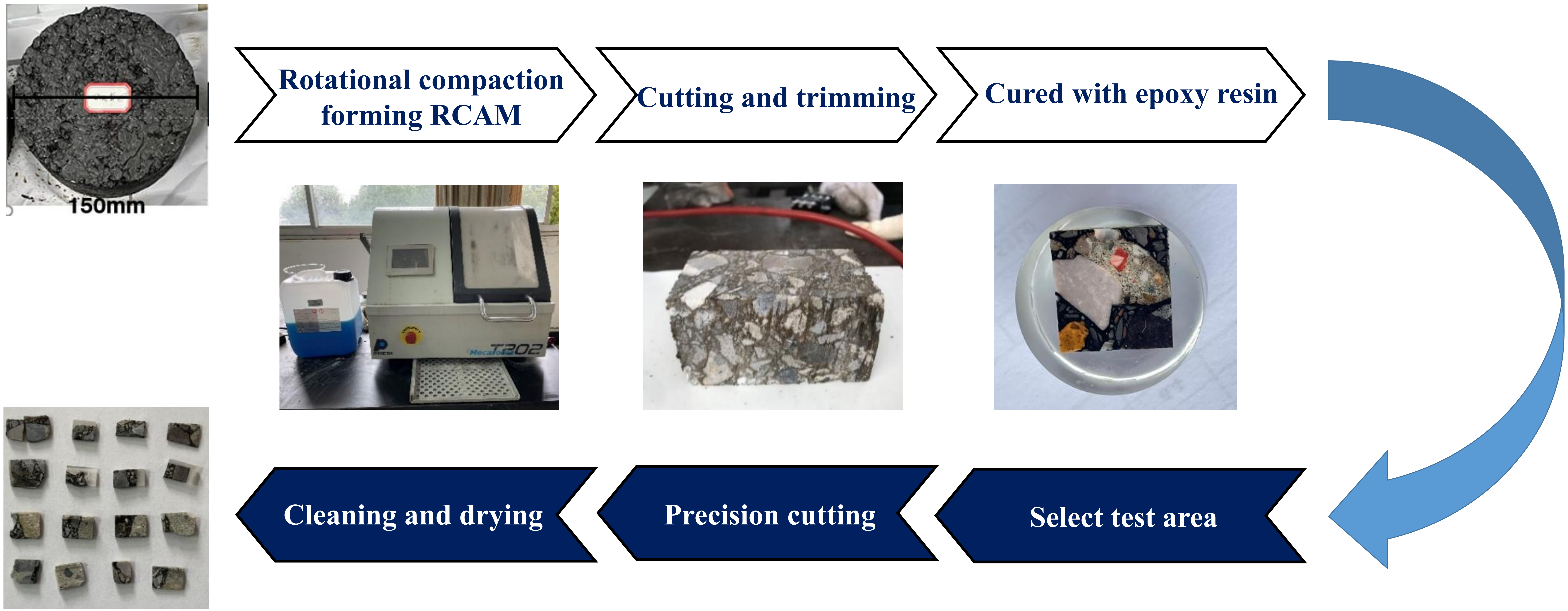
Spacing of measurement points: set the distance between adjacent points in the horizontal line to 10 μm and the distance between two test points in the longitudinal line to 10 μm.

3.2 Electron microscope observation

- **Microscopic morphological observation methods**
- In order to observe and characterise the microscopic morphology of the transition zone at the recycled coarse aggregate - asphalt mastic interface, Field Emission Scanning Electron Microscopy (FESEM) and Environmental Scanning Electron Microscopy (ESEM) were used to study and obtain detailed individual phase imaging as well as microstructure to better help understand the apparent morphological characteristics of the transition zone between the old and new interfaces.
- **Microscopic morphological observation sample observation**
- Scanning electron microscope (ESEM) images were selected at magnifications of 100-5000, with the pixel brightness of the images corresponding to the atomic number of the phase being tested below the sample surface. The pores and uncompacted structures were the darkest during the observation, followed by asphalt and aggregate particles. Prior to performing the mapping, a gold spray treatment was applied to the sample surface in order to make the image contrast unaffected by the excess charge in the sample.

3.2 Electron microscope observation

- **Sample preparation method for microscopic morphological observations**



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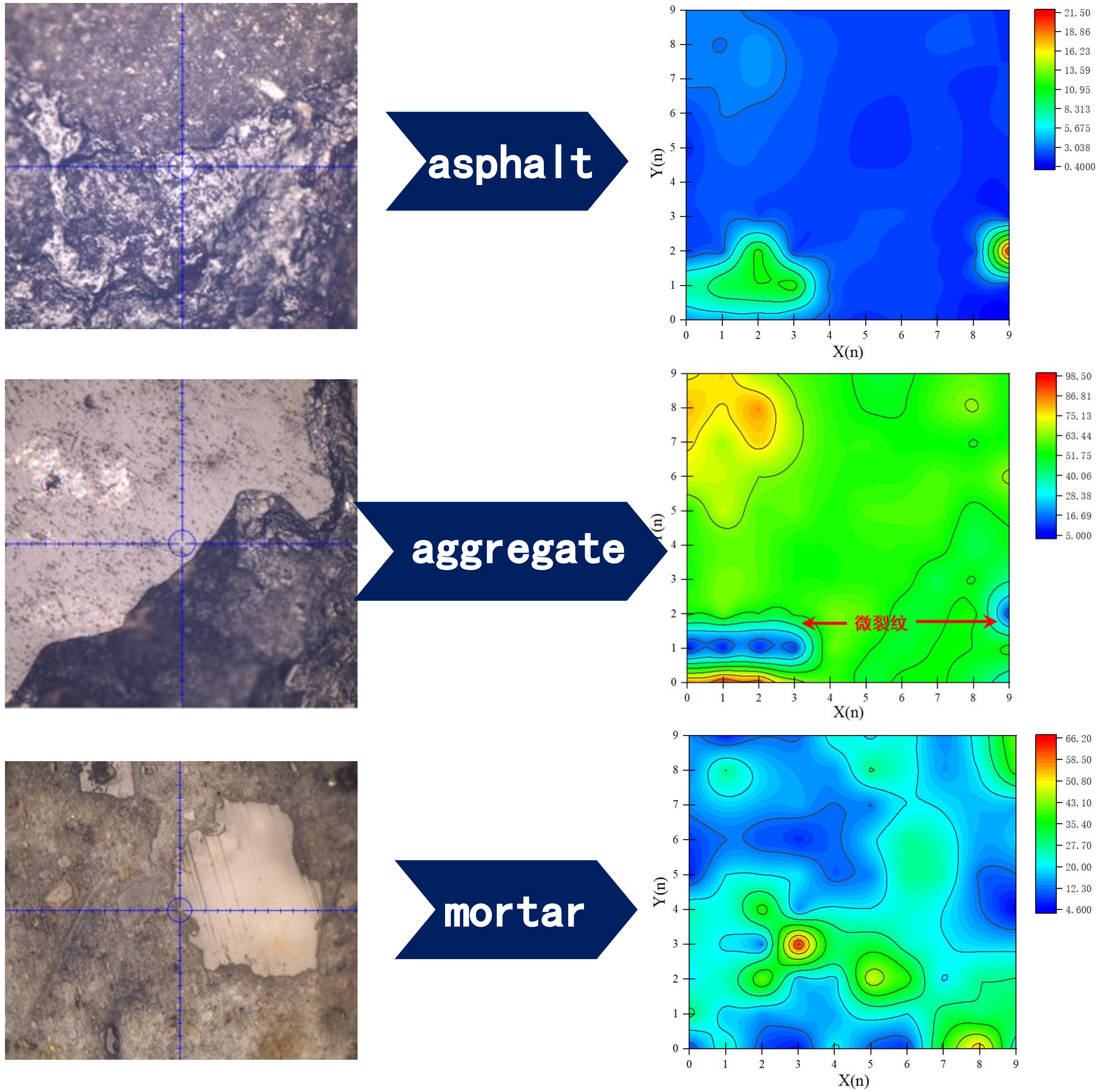
Results and discussion

4.1 Micromechanical properties of ITZs in RCAM

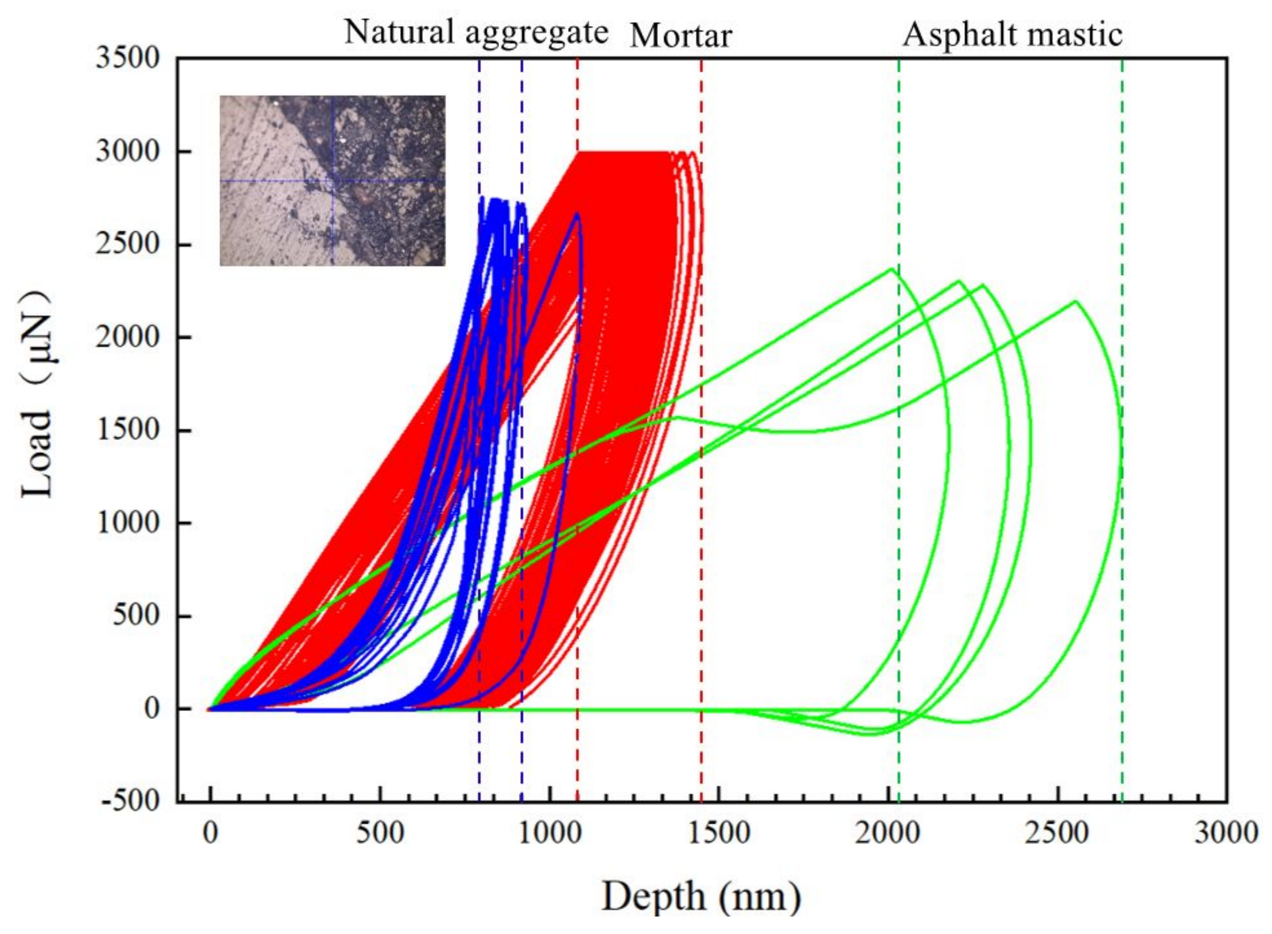
(1) Elastic-mechanical characteristics of RCAM

The joint interface of the RCA asphalt mix is composed of five different phases, in order of extension from the aggregate: aggregate, M-N ITZ, mortar layer, M-A ITZ, and asphalt. Due to the heterogeneity of the interfacial transition zone, single-phase testing was required to identify the phase composition by selecting the corresponding areas from different samples. Indentation tests on a 10*10 grid were carried out on each phase. The loading-unloading curves of each indentation were recorded, and the modulus of single material in Tab. 5 was obtained from the elastic unloading curve by using the Oliver-Pharr method.

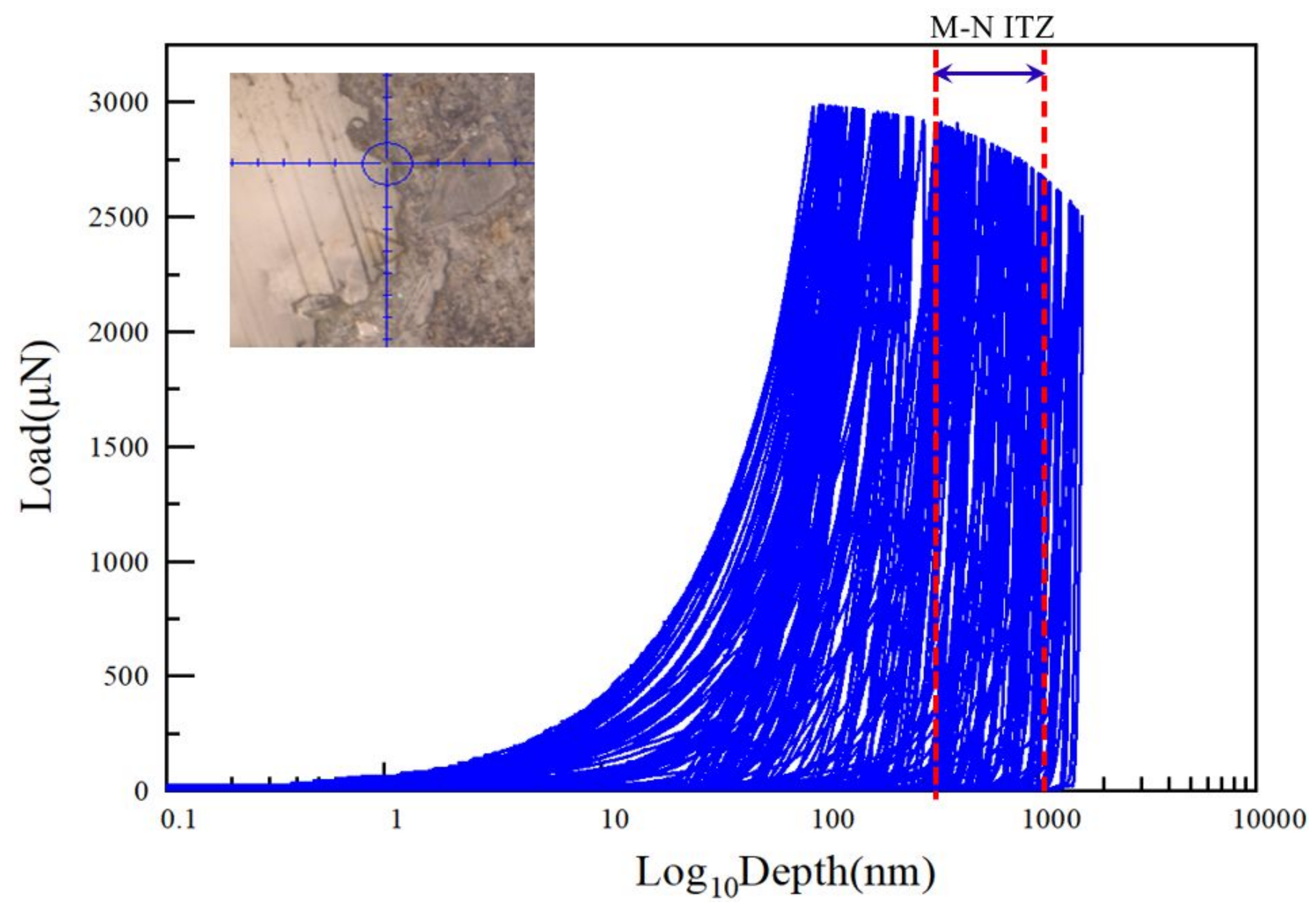


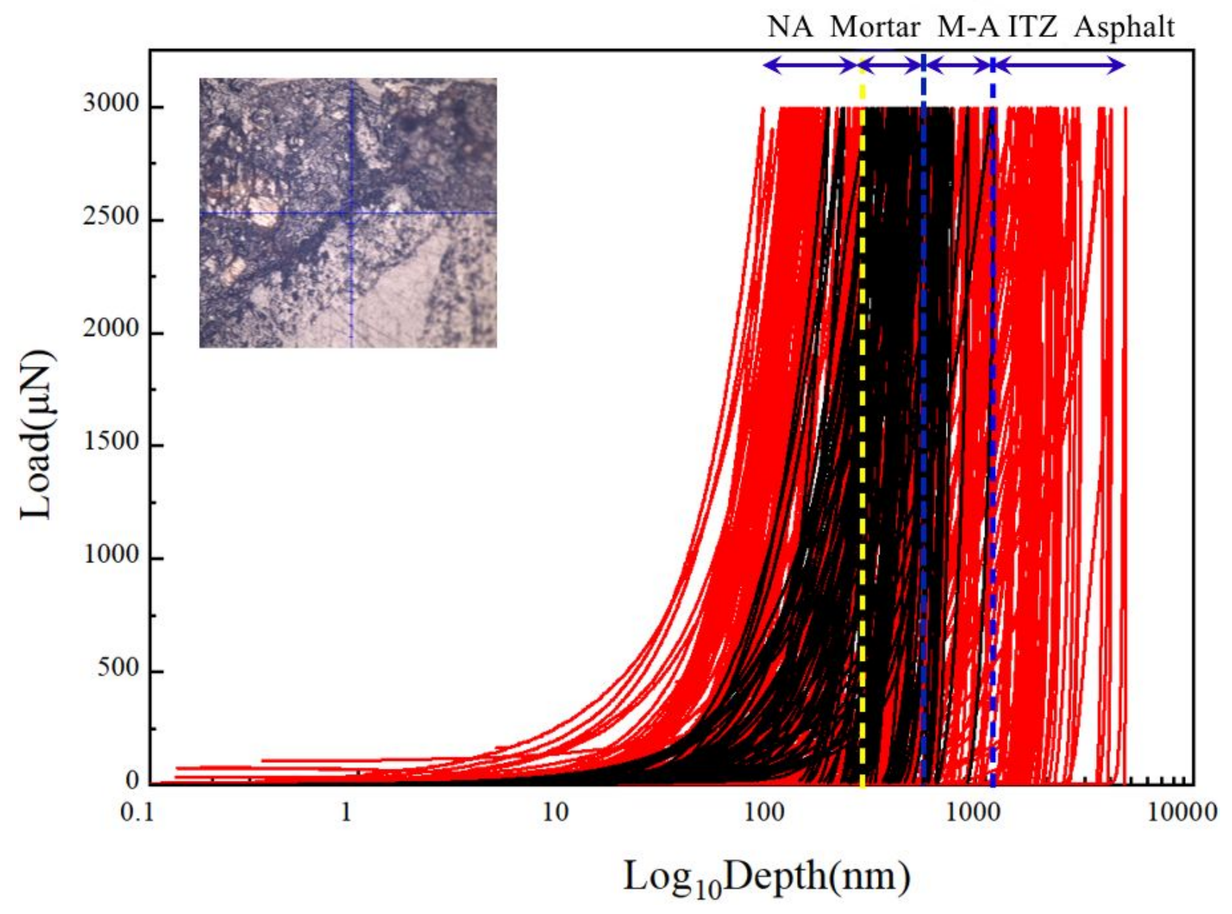
4.1 Micromechanical properties of ITZs in RCAM

- As can be seen from the loading and modulus distinctions, the load-depth curves characterise asphalt, natural aggregates and cement mortars very well and can distinguish between different areas well.
- The mechanical response distribution represented by the frequency curve was used to analyse the indentation data on the transition zone of the old interface. The load-displacement curves obtained for the ITZs are shown in the figure, with modulus intervals within the three-phase modulus range, which indicates that the surface roughness and loading method meet the requirements of the NI test.



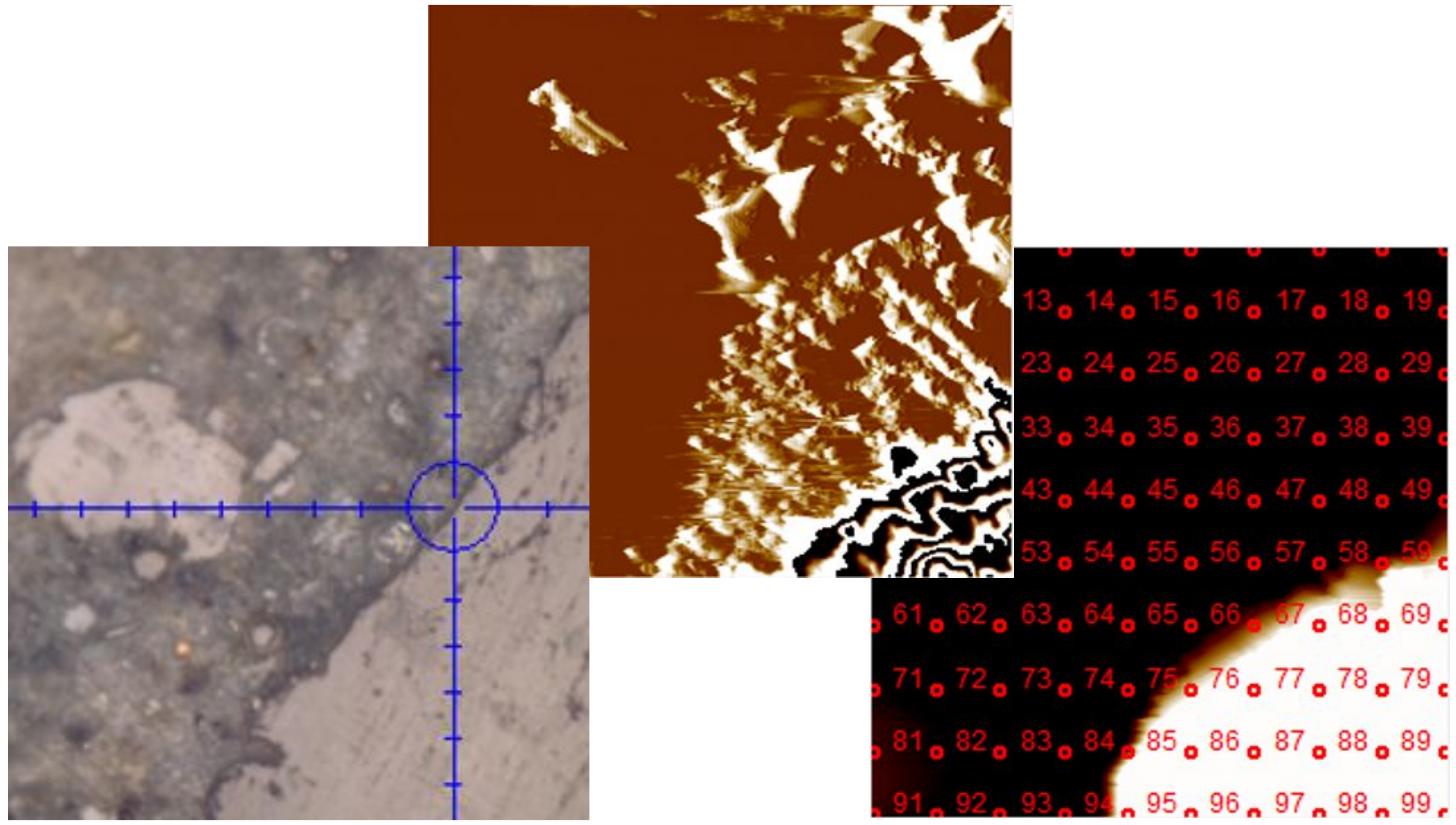
Material	Cement mortar	Modified asphalt	Aggregate
Modulus /GPa	30.23	1.62	50.65
Coefficient of Variation /%	31.2	15.8	12.5

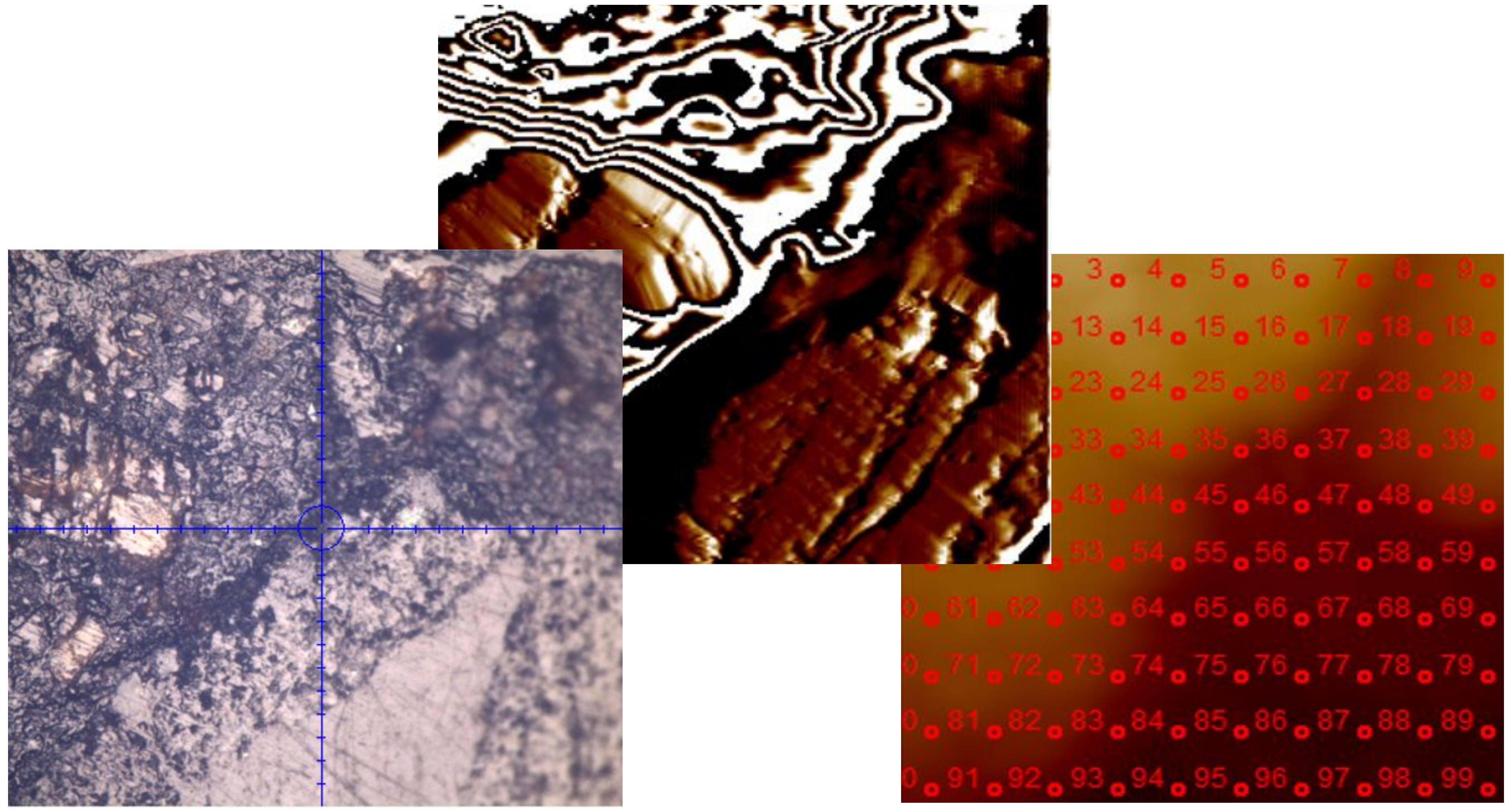




4.1 Micromechanical properties of ITZs in RCAM

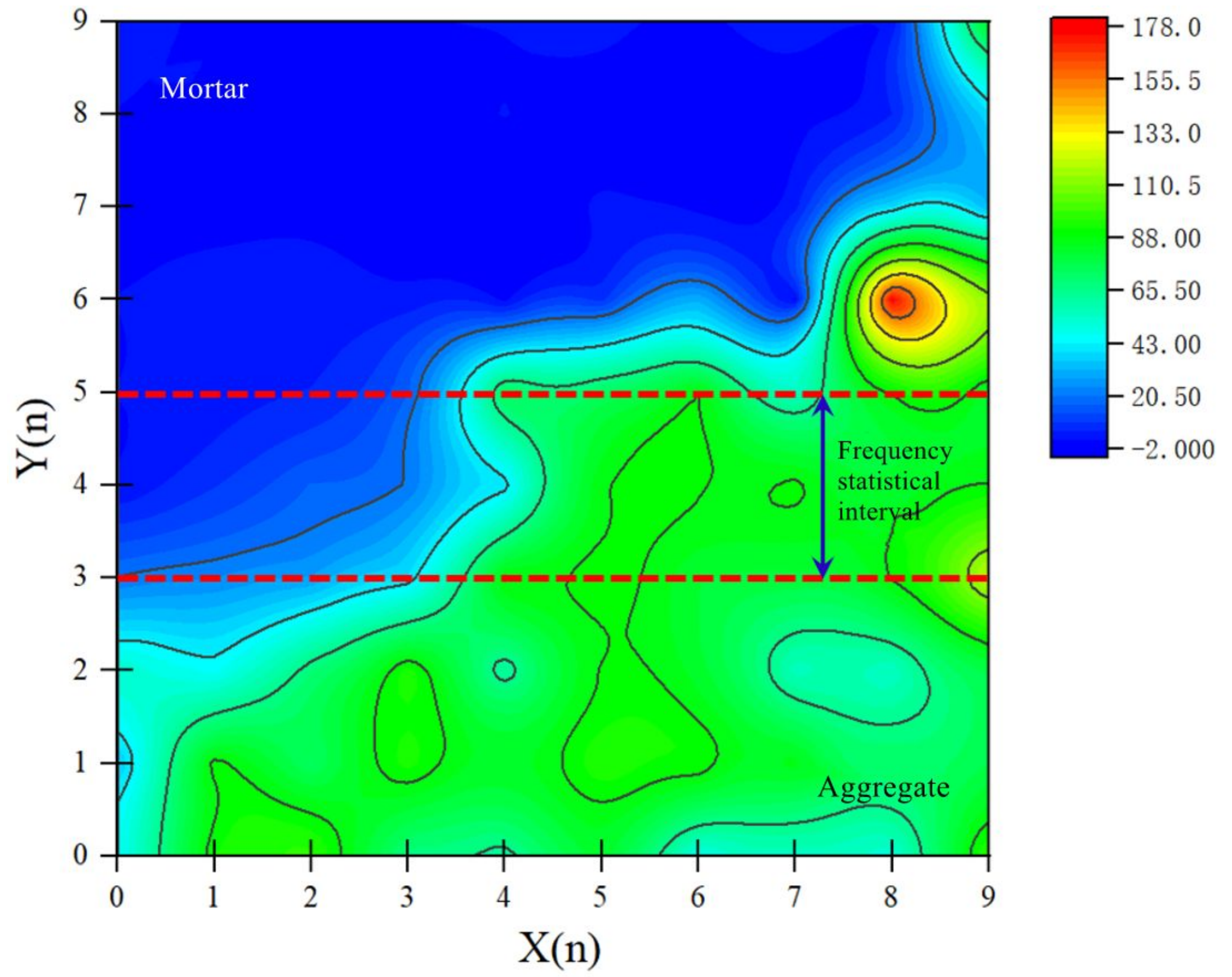
The mechanical response distribution expressed as a frequency curve was used to analyse the indentation data on ITZs. Figure 1 on the right shows the nanoindentation region of the grid area. Figure 2 on the right shows the in-situ 2D mapping of mechanical properties in the old interface transition zone using nanoindentation used to examine the different phase properties independently of each other. Figure 3 on the right shows the grid arrangement of the old interface transition zone.

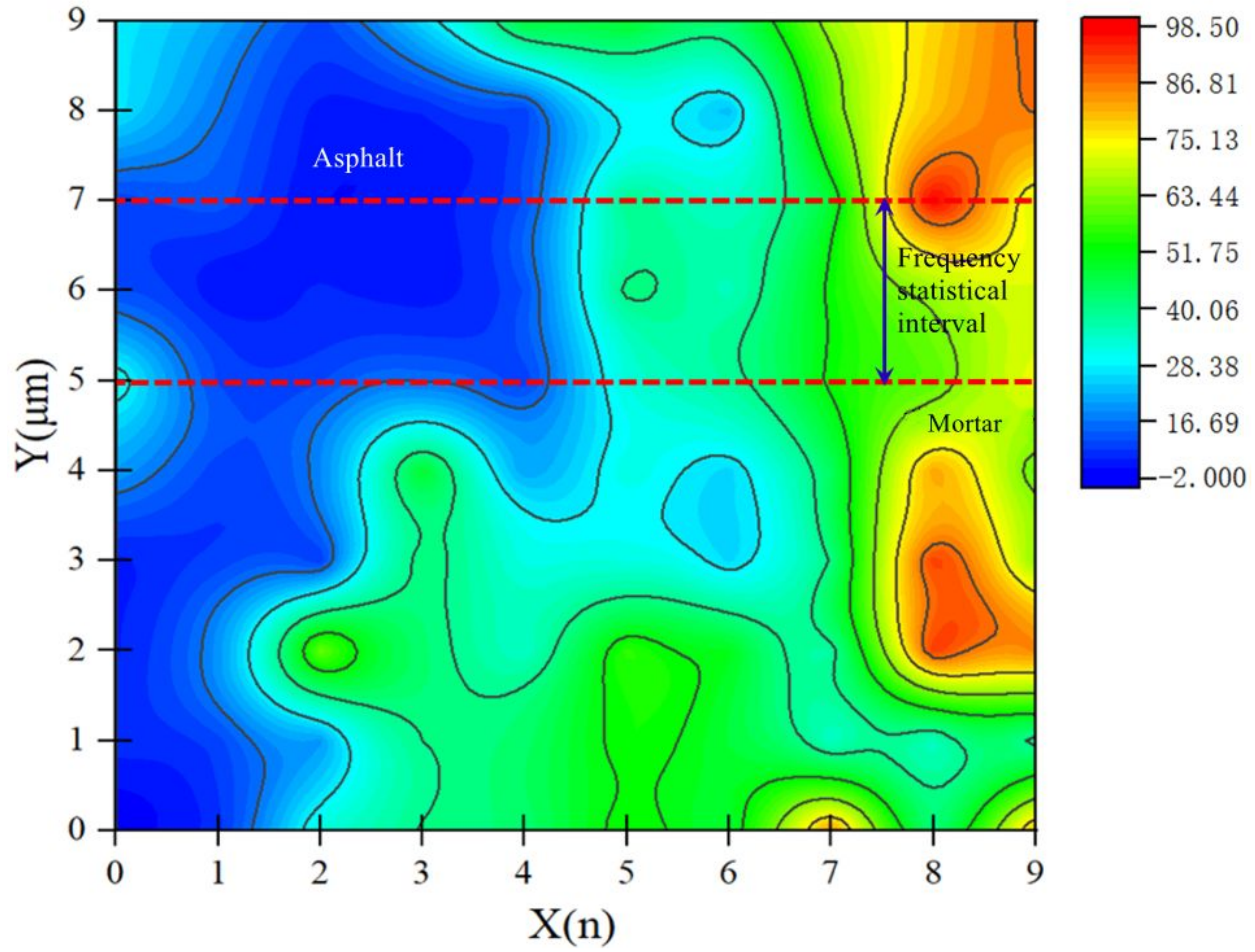




4.1 Micromechanical properties of ITZs in RCAM

Using the modulus data obtained from the tests, indentation modulus contour maps of the interfacial transition zone area were developed to examine the mechanical properties of the material phases independently of each other. As the modulus contour map is not uniformly distributed, resulting in data deviations in the longitudinal and transverse averaging statistics, this study identifies suitable intervals for frequency statistics with reference to the modulus contour image and applies the statistical averages and deviations to the modulus frequency histograms over the old interface transition zone. To identify the indentation modulus distribution in specific directions and to determine the thickness of the old interface transition zone. By analysing the lateral variation in properties around the aggregate.





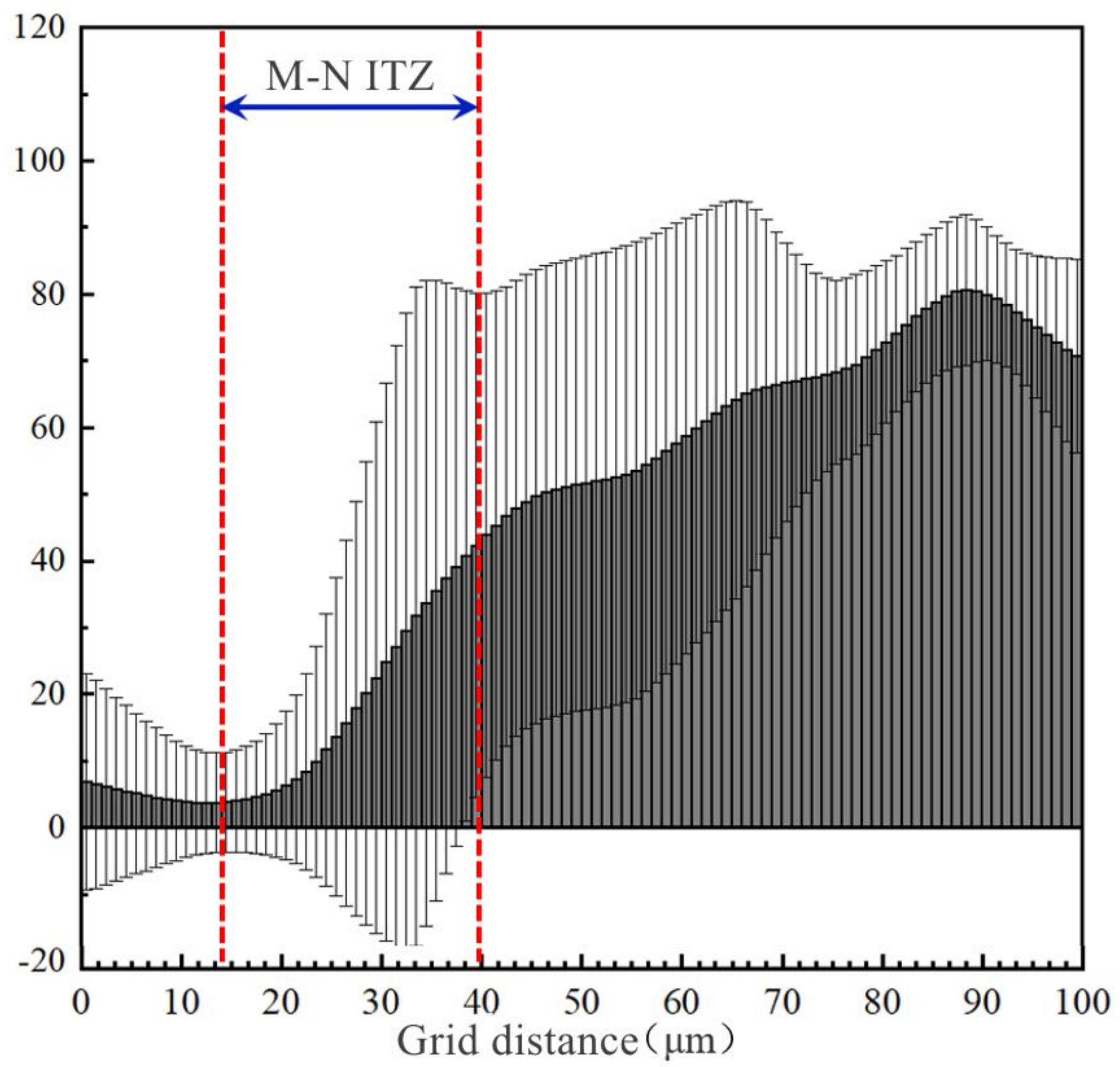
4.1 Micromechanical properties of ITZs in RCAM

(1) Elastic-mechanical characteristics of M-N ITZ

By analysing the lateral variation of properties around the aggregate, the M-N ITZ is 25 μm thick. The average modulus of M-N ITZ is 24.01 GPa, which is 1.3-1.5 times the average modulus of cement mortar.

The modulus frequency diagram represents the trend in the modulus of elasticity from the cement mortar phase to the natural aggregate phase.

- 0-15μm, in the mortar phase, when the modulus of the material is at a low level, 0-3 GPa
- 15-40μm, in the M-N ITZ, the modulus is between 10 GPa and 30 GPa
- Beyond 85μm, the modulus of the material continues to increase gradually, and begins to slow down as it reaches the dense region



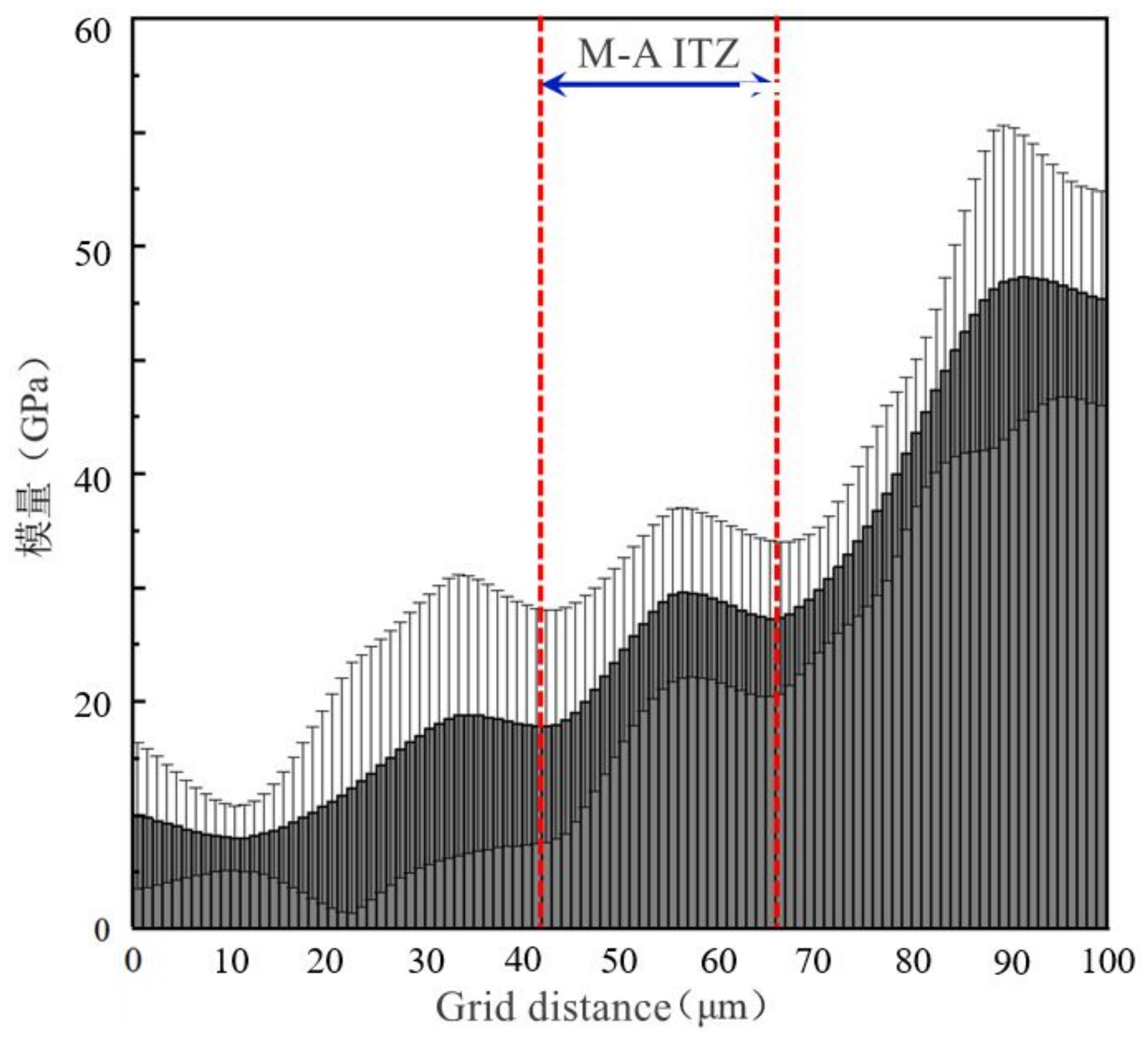
4.1 Micromechanical properties of ITZs in RCAM

(2) Elastic-mechanical characteristics of M-A ITZ

By analysing the lateral variation of properties around the aggregate, the thickness of the transition zone at the new interface is 26 μm. The results show that the average modulus of the transition zone at the new interface is 28.68 GPa.

The modulus frequency diagram represents the trend in the modulus of elasticity from the asphalt mastic phase to the aggregated phase

- 0-40μm, in the asphalt mastic phase, where the modulus is at a low level of approximately 0-5 GPa
- 40-66μm, in M-A ITZ, the modulus is between 10 GPa and 25 GPa
- Beyond 66 μm, the modulus continues to increase rapidly and more rapidly in the test area near the mortar side

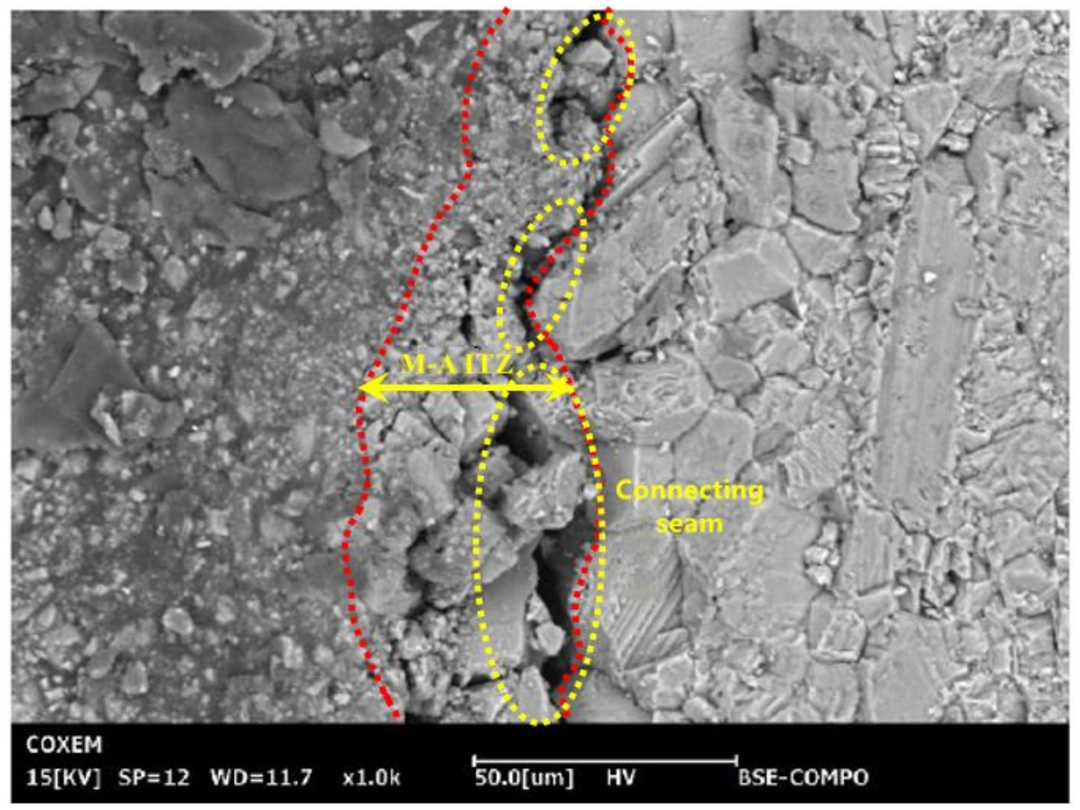


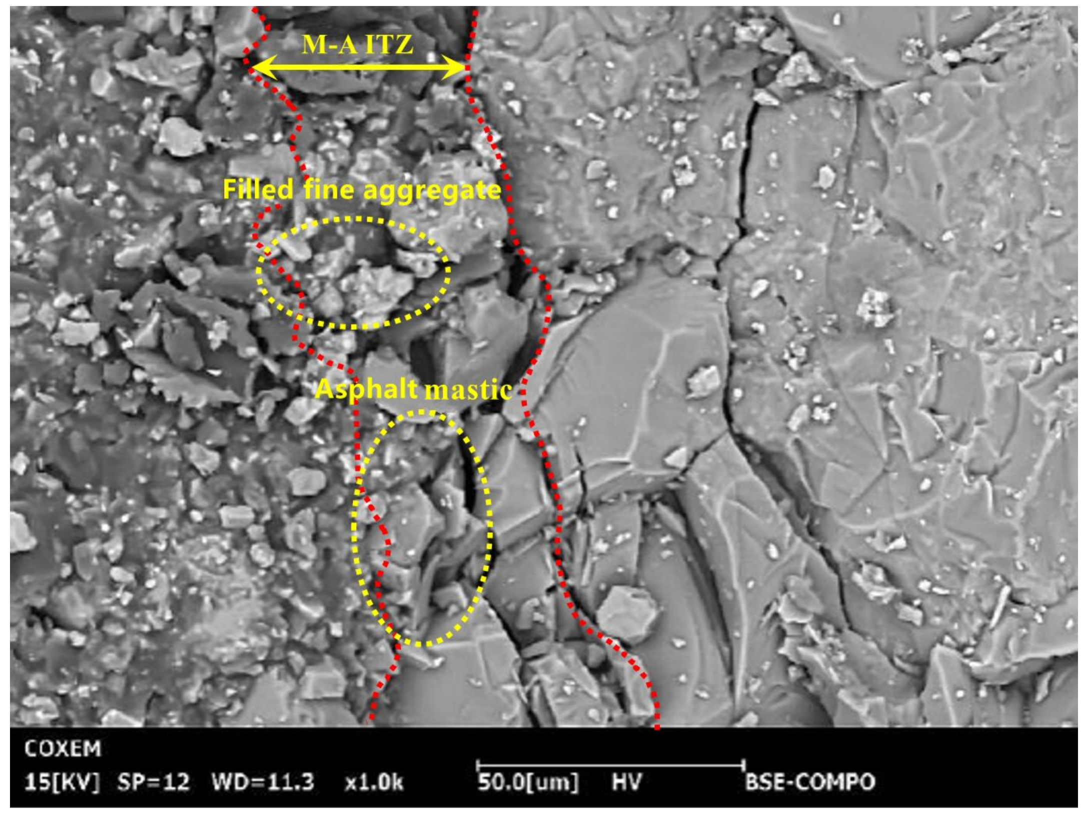
4.2 Microstructure of ITZs in RCAM

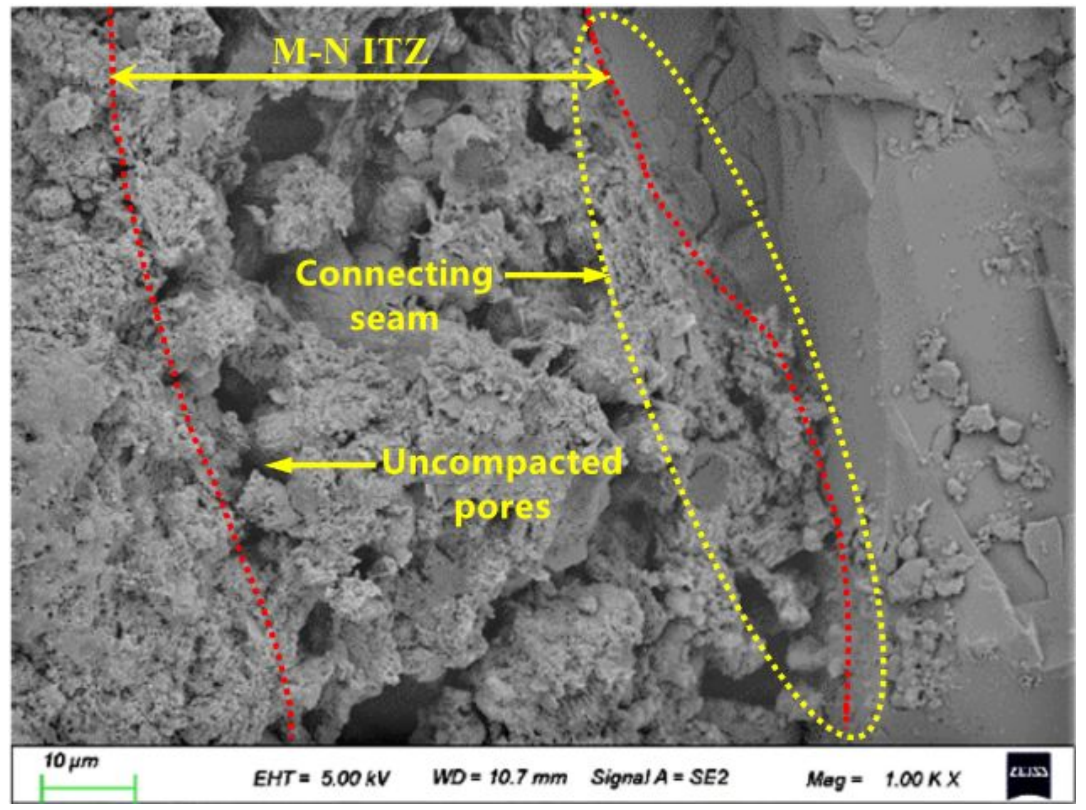
Analysis of SEM test results

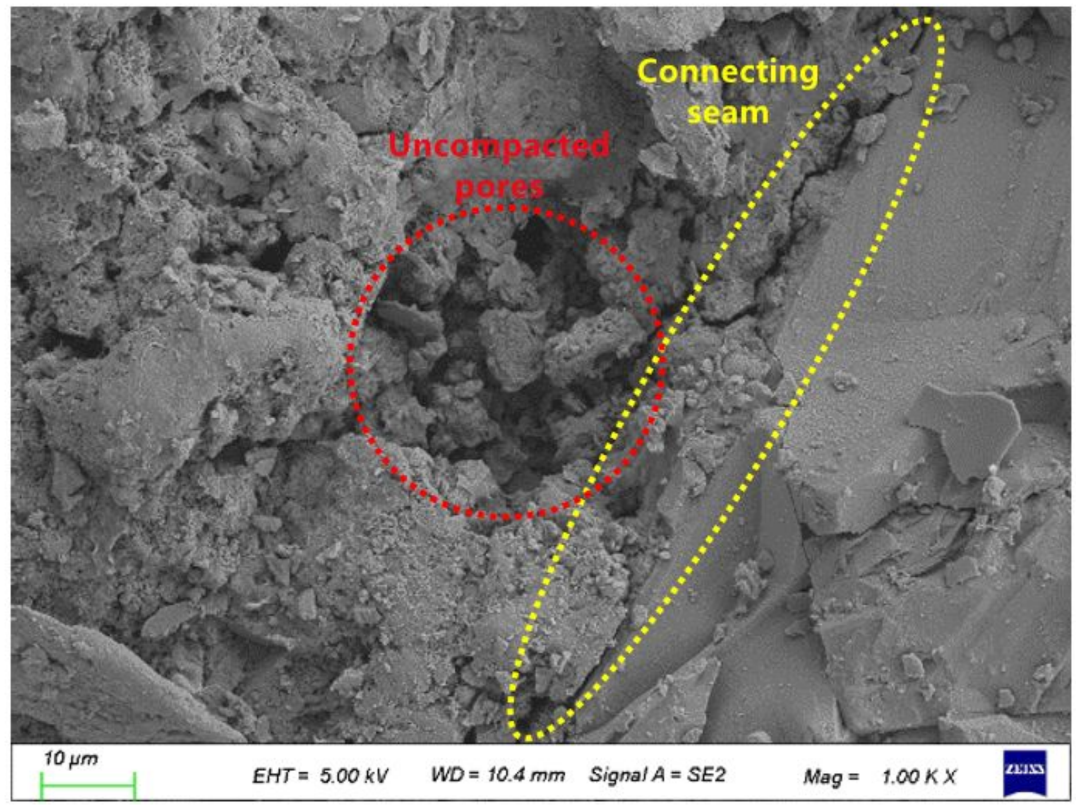
The existence of a clear boundary between the asphalt mastic and the M-A ITZ was visually verified by SEM images. The ITZ area was identified using the density variation with crystal distribution.

The M-A ITZ, as the location of the asphalt mastic-mortar phase connection, has an internal gap of 2-5 μm, which may be caused by the accumulation of poor particles of mineral dust and fine aggregates, which enter the area near the interface preventing the penetration of the asphalt mastic.









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Conclusion

5. Conclusion

1. Analysis of the modulus contour plot and modulus frequency plot of the ITZ shows that the M-N ITZ is uniformly distributed over the surface of the natural aggregate. The average modulus of elasticity of the M-N ITZ is 24.01 GPa and the thickness is 25 μm. The average modulus of elasticity of the M-A ITZ is 28.68 GPa and the thickness is 26 μm.

2. The structure of the M-A ITZ transitions from a dense area to a loose area. The M-A ITZ is mainly composed of bituminous mastic and fines. The M-N ITZ has good integrity between the interfaces of the phases and is mainly composed of mortar and filler. Some areas of the ITZ are characterised by uncompacted pores and micro-cracks, which can easily cause stress concentration and lead to structural damage.

THANK YOU

Please criticize and correct!