Analysis of Nanoindentation Test Results of Asphalt Mixture with Different Gradations

Yunhong Yu, Gang Xu, Tianling Wang, Huimin Chen, Houzhi Wang and Jun Yang *

Abstract: Nanoindentation has been applied in the field of asphalt mixtures, but, at the nano-scale, changes in the composition of the mixture and material properties can have a significant impact on the results. Therefore, it is necessary to investigate the feasibility of nanoindentation tests on different types of asphalt mixtures with different gradations and the influence of material properties and test methods on nanoindentation results. In this paper, the nanoindentation test results on three kinds of asphalt mixture (AC-13, SMA-13, and OGFC-13) with different aggregate gradations were investigated. The load-displacement curves and moduli obtained from the nanoindentation tests were analyzed. In addition, nanoindentation tests were carried out before and after polishing with different ratios of filler and asphalt (RFA) (0.8–1.6). On this basis, the morphology of asphalt specimens with different RFAs is observed by scanning electron microscopy (SEM) imaging. The results indicate that using the nanoindentation test to characterize the mechanical behavior of asphalt mixture, the confidence level of the dense-graded mixture is low, and non-dense-graded mixtures are used as much as possible. Moreover, results illustrate that the nanoindentation modulus tends to increase as the RFA increases. and the SEM chart shows that the higher the mineral powder content in the mastic, the more complex the bitumen and mineral powder interaction surface, confirming the influence of mineral powder content on the nanoindentation test results. Furthermore, the effect of polishing is almost insignificant.

Keywords: asphalt mixture; nanoindentation; gradation; the ratio of filler and asphalt

1. Introduction

Asphalt mixtures are multi-phase composites formed by combining asphalt, filler, coarse and fine aggregates in certain proportions and using specific forming methods. The performance of asphalt mixtures is influenced by a number of factors, such as asphalt, minerals, gradation and the forming process [1–3]. Pawel studied the effect of the interlocking of aggregate on the cracking of mixture, and showed that reaching the interlocking stage during compaction resulted in better rutting resistance [4]. Ghuzlan investigated the effect of grading on rutting performance [5]. Numerous domestic scholars have studied the influence of minerals on the performance of mixes at a macro level. The overall performance index of the mixture couples the aggregate and asphalt mastic properties. However, the influence of the interaction between the various parts on the mixture is not well studied. Therefore, it is necessary to find a new way to investigate it. In recent years, the rise of various microscopic test methods for studying asphalt materials has brought new prospects. Among these, nanoindentation technology is gradually becoming a powerful tool for the characterization of microscopic properties of asphalt materials. It can record the load-depth curves during indentation of the indenter onto the sample by touching different phase materials on the surface of the specimen under a high-definition microscope and subsequently fit the test curves with a mechanical model to analyze the test curves. The micromechanical
indices of each phase material can be accurately determined, and the mechanical properties can be tested at the micro- and nano-scale [6–8]. Many researchers have carried out detailed investigations on the application of nanoindentation technology in asphalt materials and asphalt mixtures, and useful findings have been put forth. In terms of viscoelastic characterization of asphalt, Jager was the first to use the nanoindentation technique to test B50 and B70 asphalt considering the indenter geometry, who found that the viscoelastic parameters correspond well with three deviation creep model parameters [9]. Veytskin used linear viscoelastic analysis to extract the viscoelastic properties of aged asphalt and asphalt mastic samples and calculated the shear relaxation modulus and dynamic modulus. It was shown that the nanoindentation results were consistent with those measured by the dynamic shear rheological (DSR) test tests in terms of magnitude and trend, thereby verifying the feasibility and accuracy of nanoindentation for studying viscoelastic properties of asphalt mastic [10,11]. For the viscoelastic characterization of asphalt mixture, Oyen and Jager et al. used the elastic-viscoelastic correspondence principle to derive the viscoelastic solutions of indentation curves for different loading phased to characterize the creep response of materials during loading [12–14]. Moreover, Yao et al. quantified the strength loss of asphalt mixture before and after freeze-thawing and the fracture properties of asphalt concrete. Their research demonstrated that nanoindentation is more detailed and accurate in the evaluation of asphalt mixture properties [15,16]. Duraid investigated the effect of warm additives on the nano-mechanical properties of asphalt mixture in three phases (aggregate, interfacial transition zone and mastic), suggested that nanoindentation can reflect the real behavior of asphalt mixture [15,17]. In terms of the interfacial transition zone (ITZ) of asphalt mixtures, some scholars are devoted to the study of the interface characteristics of asphalt mixture and the mechanical behavior of ITZ [18,19]. The accuracy of this was verified by energy dispersive spectroscopy (EDS) [20]. The above results show that the nanoindentation test has good applicability and prospects for the characterization of asphalt mixture properties. It may provide a new way to explore the properties of asphalt mixtures. However, a comparison of the research findings published earlier reveals that the sample preparation procedure of asphalt materials is more complicated, and the test parameters are not the same when the nanoindentation test was applied in each study.

Various factors highly influence the results of nanoindentation tests on asphalt materials due to the precision of nanoindentation test. Zhao et al. were the first to investigate the effect of indenter size on the test material in nanoindentation testing and found that the larger the radius of the indenter, the more plastic deformation caused by indentation load in the test [21]. Tarefder et al. systematically studied the application of the nanoindentation technique in asphalt materials. The effects of loading rate and creep satiation time on the nanoindentation test results were investigated, and the nanoindentation test parameters and settings applicable to asphalt materials were determined. The hardness and Young’s modulus values measured at a satiation time of about 200 s were found stabilized [22]. According to Yao’s test results, in addition to the maximum load, loading rate, and other test parameters, the nanoindentation test results of asphalt materials and asphalt mixtures are also affected by other factors, such as the composition of the asphalt material, interfacial attributes, and the gradation of the asphalt mixture [23].

The above research results show that asphalt mixtures are multi-phase composites with complex aggregate-asphalt interface properties, which are influenced by asphalt, aggregate, and mutual interaction. However, they ignore the influence of the gradation on the reliability of the nanoindentation results. For asphalt mixtures with varying gradations, the interface composition and structure characteristics are different, and the measured nanoindentation data will also appear significant differences. Therefore, it is necessary to investigate the suitability of different gradation asphalt mixture for nanoindentation. In addition, considering the preparation process of asphalt mixture, nanoindentation specimens must be polished to meet surface flatness requirements. For materials such as metals and bones, the surface disturbance generated by polishing has less impact on the measurement of mechanical properties of the samples. For asphalt materials, however,
such disturbance is highly probable to damage the surface of the specimens and have an unpredictable impact on the subsequent mechanical property tests. Therefore, the purpose of this paper is to investigate the variability and deviation of the test results of nanoindentation for asphalt mixtures with different gradations. According to the variability of the test results, the influence of different RFAs corresponding to different gradations of mixtures on the nanoindentation test results is discussed. On this basis, the influence of polishing is considered to guide further on the subsequent performance testing of nanoindentation for asphalt mixtures.

2. Experiment
2.1. Specimen Preparation

2.1.1. Asphalt Mixture Specimens

In this paper, three typical gradations (AC-13 continuous dense gradation, SMA-13 gap-gradation, and OGFC-13 open gradation) commonly used in China were selected to investigate the effect of mixture types on the nanoindentation test results. Two samples for each type were selected. The three gradation curves are shown in Figure 1. According to the AASHTO T312(AASHTO 2015) standard specification, a Superpave gyratory compactor was used to compact the loose asphalt mixture. The size of the cylindrical specimen is 150mm in diameter and 170mm in height (Figure 2). In order to determine the target air void and bulk specific gravity of specimen, the AASHTO T166(AASHTO 2016) standard specification was used. The air void content of the samples and the required compaction times are shown in Table 1.

![Figure 1. Gradation curves of 3 kinds of asphalt mixtures.](image)

Table 1. The experimental data required for the sample.

| Gradation | Air Void Content | Number of Gytrations | Optimum Asphalt Content |
|-----------|------------------|----------------------|-------------------------|
| AC-13     | 4%               | 100                  | 5.0%                    |
| SMA-13    | 4%               | 100                  | 6.0%                    |
| OGFC      | 20%              | 75                   | 4.1%                    |

The specimens were cut from the mixture specimens, and two parallel samples of each gradation were prepared with a thickness of 10 mm. After curing the specimens in resin, the surfaces of the specimens were polished on a metallographic polisher (500 rpm) with metallographic sandpaper under water-cooled conditions. The sandpaper grit was 150, 400, 800, 1200, and 2000 mesh, respectively. After the polishing was completed, the specimens were cleaned with an ultrasonic cleaner for 2 min, dried, and sealed for storage. As shown in Figure 2, the asphalt mixture was cored into a 1.5-cm cuboid and then cut into rectangles that were 1-cm in height to prepare the nanoindentation specimens.
2.1.2. Asphalt Mastic Specimens

In this study, mineral filler with a density of 2.657 g/cm\(^3\) and SBS modified asphalt were used to prepare asphalt mastic. The properties, such as penetration, softening point and ductility, of this asphalt are listed in Table 2. Based on the recommended RFA in the specification JTG F40-2004, the RFA for this study was determined to be 0.8, 1.0, 1.2, 1.4, and 1.6. To prepare the mastic specimens, the asphalt and mineral powder were first heated for 30 min, and then mixed with a high-speed electric mixer at 1500 rpm at 155 °C for 30 min. The final asphalt mastic samples are shown in Figure 3.

| Test                                   | SBS Modified Asphalt |
|----------------------------------------|----------------------|
| Penetration(25 °C, 100 g, 5 s)/0.1 mm  | 55                   |
| Softening point/°C                     | 87.7                 |
| Ductility(10 °C, 5 cm/min)/cm          | 41                   |
| Mass change/%                         | 0.13                 |

Table 2. Properties of SBS modified asphalt.

Figure 2. Preparation of nanoindentation specimens.

Figure 3. Nanoindentation asphalt mastic specimens.

2.2. Test Method

The mechanical properties of the specimens were tested using the NanoTest TM (Micro Materials, Wrexham, NorthEast Wales, UK). The maximum load was determined as 3 mN for the nanoindentation asphalt mixture specimens and 0.12 mN for the mastic samples, with a loading/unloading rate of 0.01 mN/s and a creep satiation time of 200 s. The load-depth curves were obtained from the nanoindentation test, and the Oliver-Pharr model was used to calculate the materials’ elastic modulus and hardness-depth curves [24]. Figure 4 shows the area of the aggregate-asphalt mastic interface in a typical asphalt mixture specimen under optical microscopy. It should be noted that the asphalt mastic phase tends to appear white under the microscope, while the aggregate phase is gray-black and may contain reflective crystalline compounds. Black lines in Figure 4 distinguish them.
Appl. Sci. 2021, 11, x FOR PEER REVIEW 5 of 16

Figure 4 shows the area of the aggregate-asphalt mastic interface in a typical asphalt mixture. The red circles indicate the selected test interface. As can be seen from Figure 6, the continuous asphalt mastic interface tends to appear white under the microscope, while the aggregate phase is gray.

For each nanoindentation sample, 16 measurement points (4×4 matrix, measurement point interval 80 μm) were arranged in a clear and flat area. Some micrographs of the test area and the distribution of measurement points are shown in Figure 5.

Figure 5. Microscopic images of the nanoindentation test area of asphalt mastic samples.

(a) Typical area 1 (b) Typical area 2

The FEI Inspect F50 (FEI, Hillsboro, OR, USA) field emission SEM was used to observe the external morphology of the asphalt mastic with different RFA. In addition, since all asphalt mastic is inorganic and non-conductive, the sample surfaces were gold-plated before the electron microscope scanning was performed.

3. Results and Discussion

3.1. Analysis of the Three Gradations

Figure 6 shows the scanned images of the three asphalt mixtures in cross-section. The red circles indicate the selected test interface. As can be seen from Figure 6, the continuous gradation asphalt mixture contains a large number of fine aggregate particles. Its aggregate-asphalt mastic interface is more complex and unclear, which causes difficulties in selecting the measurement area and subsequent tests. Moreover, the gap-gradation asphalt mixture has a clear and flat aggregate-asphalt mastic interface suitable for nanoindentation. Although the open-graded asphalt mixture has a clear and flat aggregate-mastic interface, there are more holes at the interface, which affects the selection of the test area.
gradation asphalt mixture contains a large number of fine aggregate particles. It's aggregate-asphalt mastic interface is more complex and unclear, which causes difficulties in selecting the measurement area and subsequent tests. Moreover, the gap-gradation asphalt mixture has a clear and flat aggregate-asphalt mastic interface suitable for nanoindentation. Although the open-graded asphalt mixture has a clear and flat aggregate-mastic interface, there are more holes at the interface, which affects the selection of the test area.

![Asphalt mixture pictures.](image)

Figure 6. Asphalt mixture pictures.

The load-depth curves obtained for the nanoindentation specimens of the three kinds of asphalt mixture are shown in Figure 7. The results of the nanoindentation modulus after removing the outliers are shown in Table 3. As shown in Figure 7 and Table 3, the load-depth curves of continuously graded asphalt mixture have some dispersion in the maximum depth and curve form, and the red part of the curve is the abnormal curve. After removing the outliers, the mean values of moduli have high coefficients of variation, which may be due to the distribution of some of the measurement points near the fine aggregate particles. The maximum depth and modulus values of the load-depth curves of gap-graded and open-graded asphalt mixtures are less discrete. The maximum depth dispersion of gap-graded asphalt mixtures is less than that of open-graded asphalt mixtures.

| Gradation | Modulus/GPa | Coefficient of Variation/% |
|-----------|-------------|----------------------------|
| AC-13     | 0.576       | 31.8%                      |
| SMA-13    | 0.028       | 16.3%                      |
| OGFC      | 0.014       | 11.03%                     |

Table 3. Nanoindentation modulus of different gradation asphalt mixtures.

It is worth mentioning that the nanoindentation modulus of dense-graded asphalt mixture is much higher than that of gap-graded and open-graded asphalt mixtures. This may be attributed to the dense-graded asphalt mixture contains a large amount of fine aggregate and mineral filler particles, which produce a stronger interaction (adhesion) with asphalt. Another possible reason could be that there are fine aggregate particles below the asphalt mastic phase, which affects the nanoindentation test results.

In conclusion, gap gradation and open gradation are significantly more suitable for nanoindentation test studies on asphalt mixtures, both in test operation and characterization of the mechanical properties of the asphalt mastic.
Figure 7. Nanoindentation load–depth curves of asphalt mixture with different gradations.
3.2. Effect of Different Rates of Filler and Asphalt

As given in Table 3, it can be seen that the modulus of the gap-graded asphalt mixture is about twice that of the open-graded mixture. Although both gap-gradation and open-gradation asphalt mixtures have less fine aggregate, there is still a significant difference in their nanoindentation test results due to the asphalt mastic composition between the two. It has also been shown that most SMA asphalt mixtures have an RFA of 1.6 to 1.8 [25], while the ratio of filler to the asphalt of OGFC-graded mixtures is around 1.0 [26,27]. Therefore, in order to further investigate the differences in the results between the gap-graded and open-graded asphalt mixtures, asphalt mastic specimens with different RFA (0.8 to 1.6) were prepared for nanoindentation tests before and after polishing. The effects of the RFA and polishing on the nanoindentation test results were further examined.

Figure 7 shows the asphalt mastic specimens before and after polishing. Since the mastic pieces were cast in the mold, the surface was inevitably convex or concave. Figure 8 shows the microscopic images of the test area before and after polishing the asphalt mastic specimen with the rate of filler and asphalt of 0.8. The creep curve of the loaded section is shown in Figure 9.

![Figure 8. Asphalt mastic nanoindentation specimens.](image1)

![Figure 9. Microscope image of asphalt mastic specimen. (a) before polishing, (b) after polishing.](image2)

The load-depth curves and modulus values obtained by the nanoindentation tests of asphalt mastic specimens with different RFA before and after polishing are shown in Figures 10 and 11, and Table 4. The fluctuation range of the maximum depth of the test curves for all specimens is within 15%, indicating the reliability of the test results. It is also...
close to the homogeneity characteristics of the asphalt mastic samples and the appropriate test parameter settings.

**Table 4.** Nanoindentation modulus of asphalt mastic specimens before and after polishing.

| RFA/Polish | Modulus/GPa | Coefficient of Variation/% |
|-----------|-------------|---------------------------|
|           | Before Polishing | After Polishing | Before Polishing | After Polishing |
| 0.8       | 0.0108       | 0.0086         | 17.1%  | 21.1% |
| 1.0       | 0.0117       | 0.0100         | 11.9%  | 12.6% |
| 1.2       | 0.0137       | 0.0122         | 15.4%  | 14.3% |
| 1.4       | 0.0181       | 0.0160         | 18.3%  | 16.9% |
| 1.6       | 0.0286       | 0.0252         | 16.8%  | 17.7% |

**Figure 10.** Nanoindentation creep curve before and after polishing of asphalt mastic specimens with RFA of 0.8.

**Figure 11. Cont.**
Figure 11. Cont.
Figure 11. Different rates of filler and asphalt load depth curves before and after polishing.

The red scale in the lower right corner of Figure 9a,b is 10 µm, while the white area is the asphalt phase. It can be presumed that the black particles are mineral powder particles with particle sizes below 0.075 mm. By comparing Figure 9a,b, it can be deduced that the asphalt mastic surface was almost entirely covered by asphalt before polishing. After polishing, the surface asphalt was ground away, and some mineral powder particles were exposed and smoothed flat. From this perspective, the polishing process did cause damage and perturbation to the specimen surface. As shown in Figures 10 and 12 and Table 4, even though the polishing process exposed the mineral powder particles on the specimen surface (and there may even be mineral powder particles below or around the measurement points), however, the nanoindentation test curves and moduli results of the asphalt mastic specimens did not change significantly before and after polishing. This is possibly due to the densely packed and well-mixed mineral particles in the asphalt mastic, forming a stable adhesive mixture with the asphalt. So even if the polishing operation wipes out the surface layer covered with asphalt, the indentation probe acts on the mineral powder particles during the nanoindentation test. However, considering that the overall mechanical properties of the mixture do not change much under water-cooled polishing conditions. The larger indentation depth eliminates the disturbance effect, leading to the nanoindentation test results that are not significantly different from those before polishing. It is inferred that the polishing only changed the mastic surface grayness, and the overall mechanical properties of the mixture remain unaffected. The polishing process of the specimens did not affect the reliability of the nanoindentation test results.

Figures 11 and 12 also show a significant difference in the nanoindentation modulus corresponding to different RFA. As the RFA of asphalt mastic increases, its corresponding nanoindentation modulus shows an increasing trend. In addition, the nanoindentation modulus of the RFA 1.6 was approximately twice the modulus of RFA 1.2. As can be seen from the underlined data in Figure 12. This further confirms that the nanoindentation modulus of open-graded asphalt mixture is twice as high as that of gap-graded mixtures. This may also be because the interfacial interaction mechanisms between asphalt and mineral powder include chemisorption, physical adsorption, and mechanical embedding. The three main bonding mechanisms work together to influence the final bond strength of the mastic. When the RFA and the mineral powder amount increase, the adhesion between asphalt and mineral powder is enhanced, resulting in an increasing in the strength of the asphalt mastic, which leads to the modulus increase. To substantiate this, a scanning electron microscope [7] study was carried out on the samples after nanoindentation. The results are, thus, shown in Figure 13.
It can be seen from Figure 13 that when the RFA is low, the distance between the mineral particles in the asphalt slurry is farther with more free asphalt. The enhancement effect of mineral powder on the asphalt mastic is smaller (as shown in Figure 13a). The binding interface of the asphalt mastic is clear and distinct. With the increase of RFA, the total surface area of mineral powder particles in the mastic increases. The more structural asphalt is formed by the adsorption of mineral powder, the greater is the enhancement effect of mineral powder on asphalt mastic. The bonding interface of the asphalt mastic tends to be stable, and there is smoother transition morphology. The mineral filler is well-coated by the asphalt, which matches the results of Shao et al. [28] regarding the microscopic interface of the asphalt mastic. When the RFA was increased to 1.6, the structural asphalt on the surface of different mineral powder particles interconnected to form a mesh structure, and the enhancement effect of mineral powder on the asphalt mastic reached the maximum (as shown in Figure 13e). According to their dispersion state (as shown in Figure 14), the above results are similar to the existing studies that classify mineral powders into suspended volume-filled, particle-structured, and particle-clustered failure states [29]. It can be concluded that the results of the SEM’s plot correspond to the minimum nanoindentation modulus at the RFA of 0.8 and the highest value of the nanoindentation modulus at the RFA of 1.6. The reason for this result is related to the enhanced interaction between the asphalt and the mineral powder, as has been explained earlier.

![Image of nanoindentation modulus of asphalt mastic specimens.](image-url)

**Figure 12.** Nanoindentation modulus of asphalt mastic specimens. (As can be seen from the underlined data, the nanoindentation modulus of the RFA 1.6 was approximately twice the modulus of RFA 1.2).

![Image of SEM plots for different RFA values.](image-url)

**Figure 13.** Cont.
Figure 13. SEM diagram of asphalt mastic with different rates of filler and asphalt. (a–e) 500×, (f–j) 3000×.

It can be seen from Figure 13 that when the RFA is low, the distance between the mineral particles in the asphalt slurry is farther with more free asphalt. The enhancement effect of mineral powder on the asphalt mastic is smaller (as shown in Figure 13a). The binding interface of the asphalt mastic is clear and distinct. With the increase of RFA, the total surface area of mineral powder particles in the mastic increases. The more structural asphalt is formed by the adsorption of mineral powder, the greater is the enhancement effect of mineral powder on asphalt mastic. The bonding interface of the asphalt mastic
4. Conclusions

In this paper, nanoindentation tests were carried out on asphalt mixture with different gradations and asphalt mastic with different RFA. The feasibility of nanoindentation in different types of asphalt mixes was investigated. And the test results of asphalt mastic before and after polishing were compared. Based on the findings, the following conclusions can be drawn:

(1) The nanoindentation modulus of continuous graded asphalt mixture is much higher than that of gap-graded and open-graded asphalt mixtures. The load-depth curves of continuous dense gradation asphalt mixture have some dispersion in the maximum depth and curve form. The average modulus after removing the anomalies and outliers has a large coefficient of variation. The reliability of the nanoindentation test results is low for dense graded asphalt mixture containing a high level of fine aggregates. The nanoindentation tests on asphalt mixtures preferable to use a gradation type with less fine aggregates.

(2) The difference in nanoindentation modulus between gap-graded and open-graded asphalt mixtures is reflected in their different RFA. The results and microstructure of asphalt mastic with different RFA showed that as the RFA increased, the nanoindentation modulus enlarged because of the interaction enhanced between asphalt and mastic. The amount of mineral powder in the asphalt mixture has a significant impact on the results of the nanoscale test.

(3) The differences between the nanoindentation test results of asphalt mastic with different RFA before and after polishing are minor, indicating that polishing has a negligible effect on the test results of the asphalt mixture.

Author Contributions: The authors confirm contribution to the paper as follows: study conception and design: Y.Y., G.X., J.Y.; data collection: Y.Y., T.W.; analysis and interpretation of results: Y.Y., H.W.; draft manuscript preparation: Y.Y., J.Y., G.X., H.C. All authors have read and agreed to the published version of the manuscript.

Funding: This research was funded by National Natural Science Foundation of China, grant number No. 51778140 & No. 52078130.

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: Not applicable.

Acknowledgments: The authors acknowledge the financial support of the National Natural Science Foundation of China (No. 51778140, No. 52078130), Technology Research and Development Program of China State Railway Group Co., Ltd. (Beijing 10000, China) (P2019G030), Postdoctoral Research Fund Program at Southeast University (1121002107).

Conflicts of Interest: The authors declare no conflict of interest.
References

1. De Arimateia Almeida e Silva, J.; Rodrigues, J.K.G.; de Carvalho, M.W.; de Figueiredo Lopes Lucena, L.C.; Cavalcante, E.H. Mechanical performance of asphalt mixtures using polymer-micronized PET-modified binder. *Road Mater. Pavement Des.* **2018**, *19*, 1001–1009.

2. Morovatdar, A.; Ashhtiani, R.S.; Licon, C.; Tirado, C.; Mahmoud, E. Novel Framework for the Quantification of Pavement Damages in the Overload Corridors. *TRR* **2020**, *2674*, 036119812092580. [CrossRef]

3. Lu, X.; Gong, M.; Chen, J.; Zhang, D.; Liu, Z.J.; Materials. B. Study on the decay behavior of the stability of asphalt mixture under different dry-wet cycle conditions. *Constr. Build. Mater.* **2021**, *296*, 123307. [CrossRef]

4. Polaczyk, P.; Ma, Y.; Xiao, R.; Hu, W.; Huang, B.J.; Design, P. Characterization of aggregate interlocking in hot mix asphalt by mechanistic performance tests. *Road Mater. Pavement.* **2022**, *22*, S498–S513. [CrossRef]

5. Ghuzlan, K.A.; Al-Mistarehi, B.; Al-Momani, A.S. Materials. B. Rutting performance of asphalt mixtures with gradations designed using Bailey and conventional Supapave methods. *Constr. Build. Mater.* **2020**, *261*, 119941. [CrossRef]

6. Arifuzzaman, M. Nano-scale evaluation of moisture damage in asphalt. Ph.D. Thesis, The University of New Mexico, Albuquerque, NM, USA, 2010.

7. Khorasani, S.; Masad, E.; Kassem, E.; Al-Rub, R.A. Nano-Mechanical Characterization of Mastic, Aggregate, and Interfacial Zone in Asphalt Composites. *J. Test.Eval.* **2013**, *41*, 924–932. [CrossRef]

8. Tarefder, R.A.; Zaman, A.M.; Uddin, W. Determining Hardness and Elastic Modulus of Asphalt by Nanoindentation. *Int. J. Geomech.* **2010**, *10*, 106–116. [CrossRef]

9. Ger, A.; Lackner, R; Eberhardsteiner, J.J.M. Identification of viscoelastic properties by means of nanoindentation taking the real tip geometry into account. *Meccanica* **2007**, *42*, 293–306.

10. Veytskin, Y.; Bobko, C.; Castorena, C. Nanoindentation investigation of asphalt binder and mastic viscoelasticity. *Int. J. Pavement Eng.* **2016**, *17*, 363–376. [CrossRef]

11. Veytskin, Y.; Bobko, C.; Castorena, C. Nanoindentation and atomic force microscopy investigations of asphalt binder and mastic. *J. Mater. Civ. Eng.* **2016**, *28*, 994–1000. [CrossRef]

12. Fischer-Cripps, A.C. A simple phenomenological approach to nanoindentation creep. *Mater. Sci. Eng.* **2004**, *385*, 74–82. [CrossRef]

13. Oyen, M.J. Spherical Indentation Creep Following Ramp Loading. *J. Mater. Res.* **2005**, *20*, 2094–2100. [CrossRef]

14. Lu, H.; Wang, B.; Ma, J.; Huang, G.; Viswanathan, H.J. Measurement of Creep Compliance of Solid Polymers by Nanoindentation. *Mech. Time-Depend. Mater.* **2003**, *7*, 189–207. [CrossRef]

15. Khan, Z.; Faisal, H.M.; Tarefder, R. Fracture Toughness Measurement of Asphalt Concrete by Nanoindentation. In Proceedings of the ASME 2017 International Mechanical Engineering Congress and Exposition, Tampa, FL, USA, 3–9 November 2017.

16. Yao, Z.; Zhu, H.; Gong, M.; Yang, J.; Xu, G.; Zhong, Y.J.; Materials. B. Characterization of asphalt materials’ moisture susceptibility using multiple methods. *Constr. Build. Mater.* **2017**, *155*, 286–295. [CrossRef]

17. Abd, D.M.; Al-Khalid, H.; Akhtar, R.J.; Materials. B. An investigation into the impact of warm mix asphalt additives on asphalt mixture phases through a nano-mechanical approach. *Constr. Build. Mater.* **2018**, *189*, 296–306. [CrossRef]

18. Zhu, X.; Ying, Y.; Li, L.; Du, Y.; Feng, L.J. Identification of interfacial transition zone in asphalt concrete based on nano-scale metrology techniques. *Mater. Des.* **2017**, *129*, 91–102. [CrossRef]

19. Hu, J.; Huang, Q.; Lou, N.; Luo, S.J.M. Microstructural Characteristics of Interfacial Zone in Asphalt Mixture Considering the Influence of Aggregates Properties. *Materials* **2020**, *13*, 2558. [CrossRef] [PubMed]

20. Huang, Q.; Qian, Z.; Hu, J.; Zheng, D.; Yu, J.; Materials. B. Investigation on the properties of aggregate-mastic interfacial transition zones (ITZs) in asphalt mixture containing recycled concrete aggregate. *Constr. Build. Mater.* **2020**, *269*, 121257. [CrossRef]

21. Dan, Z. Research on Nanoindentation Response of Metallic Glasses via Molecular Dynamics Simulations. Ph.D. Thesis, Jilin University, Changchun, China, June 2020.

22. Tarefder, R.A.; Faisal, H.J. Effects of Dwell Time and Loading Rate on the Nanoindention Behavior of Asphaltic Materials. *J. Nanomech. Micromech.* **2013**, *3*, 17–23. [CrossRef]

23. Zheng, Y. Characterization of Micromechanical Properties of Aged Asphalt Mixtures by Nanoindentation. Master’s Thesis, Southeast University, Nanjing, China, May 2019.

24. Oliver, W.C.; Pharr, G.M. An Improved Technique for Determining Hardness and Elastic-Modulus Using Load and Displacement Sensing Indentation Experiments. *J. Mater. Res.* **1992**, *7*, 1564–1583. [CrossRef]

25. Na, W. Design of SMA Ratios and Road Performance of Southern Mountain Highways. Master’s Thesis, Chang’an University, Xi’an, China, June 2003.

26. Lei, W. Study on OGFC Gradation Regulations and Pavement Performance. Master’s Thesis, Chang’an University, Xi’an, China, June 2009.

27. Weinian, Z. Drainage asphalt mixture (OGFC-13) ratio design. *J. Chang. Inst. Technol.* **2013**, *6*, 39–43.

28. Shao, X.Z.; Tan, Y.Q.; Sun, I.J. Research on Microstructure of Asphalt Mortar. *Highway* **2003**, *105*, 105–109.

29. Ma, X.Y.; Chen, C.J.; Yang, P.W. Fatigue life prediction of asphalt masticss based on simplified viscoelastic continuum damage model. *J. Chang. Univ. (Nat. Sci. Ed.)* **2019**, *39*, 35–43. [CrossRef]