[Rock and Soil Mechanics](https://rocksoilmech.researchcommons.org/journal)

Volume 44 | Issue 12 Article 6

2-7-2024

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Xian-hui CHEN
Faculty of Engineering, China University of Geosciences (Wuhan), Wuhan, Hubei 430074, China, xianhuiym@qq.com

Yi CHENG
Faculty of Engineering, China University of Geosciences (Wuhan), Wuhan, Hubei 430074, China, chengyi@cug.edu.cn

Xin-yue XIE
Faculty of Engineering, China University of Geosciences (Wuhan), Wuhan, Hubei 430074, China

Mi-mi CHEN
Faculty of Engineering, China University of Geosciences (Wuhan), Wuhan, Hubei 430074, China

Recommended Citation

CHEN, Xian-hui; CHENG, Yi; XIE, Xin-yue; and CHEN, Mi-mi (2024) "Correlation between macro and micro mechanical parameters of marble based on nanoindentation experiment," *Rock and Soil Mechanics*: Vol. 44: Iss. 12, Article 6.
DOI: 10.16285/j.rsm.2022.6920
Available at: https://rocksoilmech.researchcommons.org/journal/vol44/iss12/6

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Abstract

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Keywords

indentation test, continuous stiffness measurement, hardness, micro mechanical parameters, marble

Rock and Soil Mechanics 2023 44(12): 3551–3564 ISSN 1000–7598
https: //doi.org/10.16285/j.rsm.2022.6920 https://rocksoilmech.researchcommons.org/journal/

Correlation between macro and micro mechanical parameters of marble based on nanoindentation experiment

CHEN Xian-hui, CHENG Yi, XIE Xin-yue, CHEN Mi-mi
Faculty of Engineering, China University of Geosciences (Wuhan), Wuhan, Hubei 430074, China

Abstract: Nanoindentation experiment is an important means to study micro mechanical properties of rock. Up to now only a few studies have discussed the correlation between the micro mechanical properties of rock and various macroscopic strengths of the rock. Firstly, the nanoindentation experiments on four different kinds of marble were carried out by the continuous stiffness measurement technique to obtain the micro mechanical parameters of dolomite and calcite. Secondly, the microscopic data scale was upgraded by Mori-Tanaka method to obtain the homogenized elastic modulus and Poisson's ratio. Finally, the correlation between the microscopic parameters and the macroscopic mechanical experiment results was analyzed, and the applicability of predicting macroscopic properties of rock using nanoindentation data was discussed. The results show that: (1) The elastic modulus of dolomite in marble is 122.5 GPa, and the hardness is 5.4 GPa. The elastic modulus of calcite in marble is 70.3 GPa, and the hardness is 2.3 GPa. The strength and deformation properties of calcite are relatively poorer compared to those of dolomite. (2) For dolomite and calcite in marble, an indention depth of approximately 800 nm is recommended for determining the elastic modulus and hardness with the continuous stiffness measurement technique. (3) The data dispersion at the grain boundary points is larger than that at the inner points of the grain, and the hardness can better reflect the defect effect of grain boundary than elastic modulus does. (4) The homogenized results obtained by Mori-Tanaka method are somewhat reliable for predicting macroscopic elastic modulus and Poisson's ratio. (5) Nanoindentation data can reflect the influence of mineral types on rock strength. To accurately predict uniaxial compressive strength, tensile strength, and fracture toughness, it is necessary to consider other strength factors, such as rock texture and structure. These results demonstrate the application of nanoindentation experiment in rock materials and provide a reference for predicting macroscopic strength using micro mechanical parameters.
Keywords: indentation test; continuous stiffness measurement; hardness; micro mechanical parameters; marble

1 Introduction

Rock mass stability is a common issue in engineering constructions, such as tunnel excavation and slope support, and rock properties are crucial elements of addressing the related engineering problems. Currently, the rock properties are mostly examined in macro view, for instance, by uniaxial compression test[1], triaxial compression test[2], and split test[3]. The conventional mechanical tests have many limitations, for example, high-level specimens are required, coring quality is hardly guaranteed, and testing data are often collected with large dispersion in those tests. Moreover, it is difficult to obtain the mechanical parameters of minerals such as hardness and elastic modulus by the macro tests[4].

The rock is a type of multiphase material, and its macro mechanical properties are affected by its mineral compositions and the corresponding micro properties. Therefore, investigating the micro structure and mechanical properties of the rock is critical to illustrating the mechanism of related macro mechanical phenomena. As micro mechanical properties of materials are deeply investigated, nano-mechanical testing technology has become the primary means for testing main materials' mechanical properties[5–6]. The nano-mechanical testing technology has a long development history. In 1961, Stillwell et al.[7] proposed that the mechanical properties of materials could be measured by their elastic recovery after extrusion. In 1981, Pethica[8] first tested the mechanical properties of metal by injecting ion onto metal surfaces. In 1992, Oliver et al.[9] perfected the principle of indentation test, which laid the foundation of nanoindentation test.

Recently, the nano-mechanical testing technology has developed rapidly and been widely adopted to examine mechanical parameters of precision materials. In nano-mechanical tests, there are few requirements on the quality and size for rock specimens, and high testing efficiency can be achieved. Consequently, this testing technology

Received: 8 December 2022 Accepted: 7 February 2023
This work was supported by the National Natural Science Foundation for Young Scientists of China (41702314) and the General Program of National Natural Science Foundation of China (42177161).
First author: CHEN Xian-hui, male, born in 1999, Master's student, focusing on rock mechanical properties and micro fracture mechanisms. E-mail: xianhuiym@qq.com
Corresponding author: CHENG Yi, male, born in 1983, PhD, Associate Professor, research interests: rock fracture mechanics and engineering geology. E-mail: chengyi@cug.edu.cn

are expanding into the field of rock mechanics[10], for example, the nanoindentation tests have been broadly conducted on granite[11], shale[12], and marble[13] to explore mechanical parameters of mineral components within those rocks. At present, most studies are concentrated around elastic modulus[14−15], hardness[16−17], and fracture toughness[18−19] of minerals at nanoscale, while the mechanical parameters at nanoscale are rarely upgraded to parameters at macro scale[20]. The standard indentation mode is widely employed in nanoindentation tests on rocks, and the elastic modulus and hardness are obtained at the deepest indentation point in this mode. Recently, the continuous stiffness measurement (CSM) technique was utilized in the field of rock mechanics, and continuous measurement of micro hardness and elastic modulus can be realized by this technique, which avoids unreliable testing results caused by sudden changes of elastic modulus and hardness during indentation. However, the depth where the CSM results are collected is not yet unified when studying mechanics parameters of rocks. The indentation depth of 2 000 nm was set in the tests on shale by CSM[21−23], but Lei et al.[19] obtained reliable conclusions on granite when the indentation depth of 400 nm was set using CSM. Thus, various indentation depths can be used for different rocks, and a reasonable indentation depth helps reduce indentation time and save costs.

Nowadays, there is a lack of nanoindentation test results about minerals in marble, and there are few studies on predicting macro properties of rocks through nanoindentation tests. The correlation between micro parameters and macro properties of rocks should be further explored. As a result, four types of marble were selected for further deep investigation in present study, and the nanoindentation tests were carried out by CSM to determine an appropriate indentation depth for marble. Meanwhile, the consistency and correlation among micro mechanical parameters, theoretical results (obtained by Mori-Tanaka model), and real macro properties (obtained by laboratory physical tests) were discussed, and the results provide a theoretical basis for macro utilization of nanoindentation data.

2 Specimen selection and experiment scheme

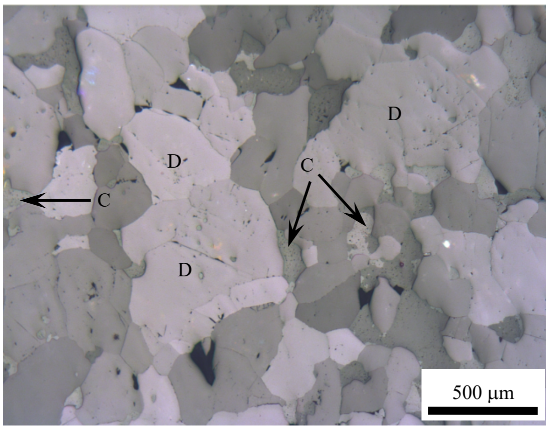
2.1 Experiment materials

Fangshan marble (Specimen No. 1), Chenzhou marble (Specimen No. 2), Hezhou marble (Specimen No. 3), and Carrara marble (Specimen No. 4) were selected for nanoindentation experiment. The mineral compositions of the four groups of marble were identified, and then the marble was made into thin slices for petrographic and mineralogical identification under a microscope. The average grain size of Fangshan marble is 0.24 mm, and the dolomite and calcite contents in this marble are 85% and 15%. The average grain size of Chenzhou marble is 0.52 mm, and this marble is composed of 80% dolomite, 15% tremolite, and a small amount of calcite and muscovite. Both Fangshan marble and Chenzhou marble belong to dolomite marble. The average grain size of Hezhou marble is 0.48 mm, and the calcite content is more than 95%. The average grain size of Carrara marble is 0.08 mm, and the calcite content reaches 99%. Both Hezhou marble and Carrara marble belong to pure calcite marble (Fig. 1).

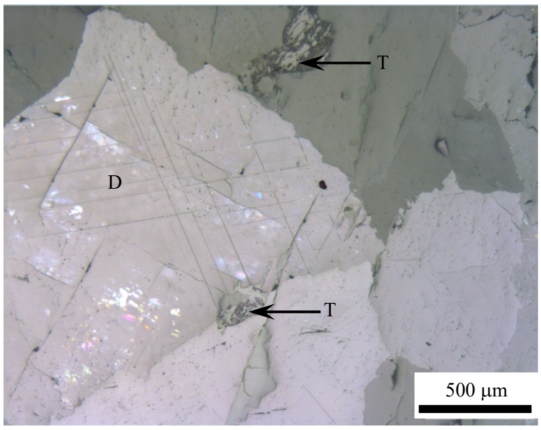
2.2 Experiment device and method

In the nanoindentation tests, the indenter tip contacted the specimen surface under a certain load, and the indentation depth as well as the corresponding load was recorded[16]. The Agilent G200 nanoindenter from School of Mechanical Science and Engineering, Huazhong University of Science and Technology (Fig. 2(a)) was used in the experiment, and the tests were performed using a Berkovich indenter. The maximum indentation load of the device is up to 500 mN, with a load resolution of 50 nN and a displacement resolution of 0.02 nm. During the experiment, the diamond indenter approached the specimen surface at a speed of 20 nm /s. In the CSM mode, the frequency was set to be 45 Hz, the displacement to be 2 nm, the strain rate to be 0.05 s^{-1}, and the maximum indentation depth to be 2 000−2 200 nm. The load was maintained for 10 s after the maximum load was reached. When using the device, the top and bottom surfaces of the specimens must be parallel, and a diameter of 30−32 mm and a height of 22−32 mm are required for specimens.

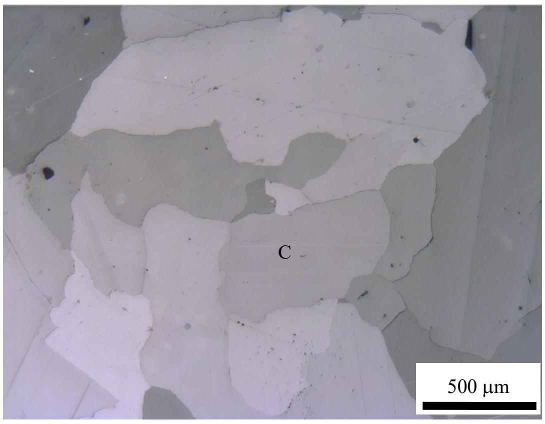
According to the requirements for the specimens used in the indentation experiment, the four types of marble specimens were cut and cast. After casting, the specimens were roughly burnished with P320, P600, and P1200 sandpaper successively, and then polished with 6 μm, 3 μm, 1 μm, and 0.05 μm polishing cloths in proper sequence. The completed specimens are displayed in Fig. 2(b). The points inside the grains and at the grain boundaries were chosen as the indentation testing points (Fig. 3). For Fangshan marble and Chenzhou marble, the testing points at the boundary were located between the dolomite grains. For Hezhou marble and Carrara marble, the testing points at the boundary were located between the calcite grains.



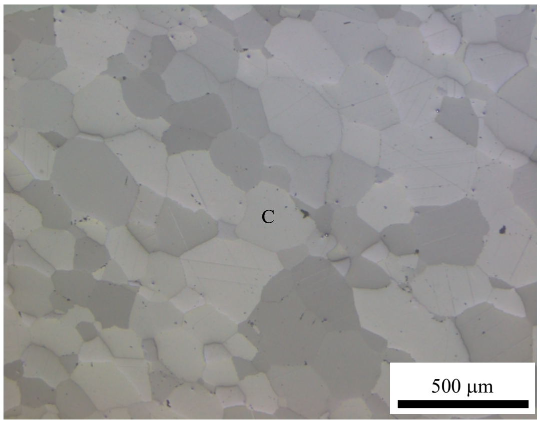
(a) Fangshan marble



(b) Chenzhou marble



(c) Hezhou marble



(d) Carrara marble

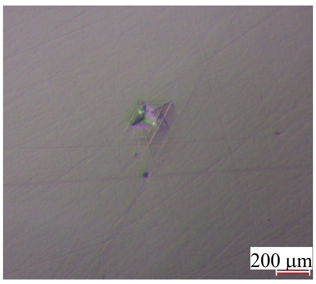
Note: the letters C, D, and T represent calcite, dolomite, and tremolite.

Fig. 1 Microscope images of micro marble structure

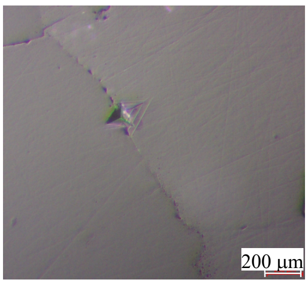
(a) G200 nanoindentation testing system

(b) Indentation test specimen from Fangshan marble

Fig. 2 Nanoindenter and test specimen



(a) Interior of grain



(b) Grain boundary

Fig. 3 Indentation testing points (Hezhou marble)

2.3 Nanoindentation principle

Oliver et al.[9] refined the principle of nanoindentation testing. In nanoindentation tests, the load–displacement curve can be plotted using obtained testing data, and the elastic modulus and hardness of the material can be calculated based on the curve. Also, the fracture toughness of the material can be got using the energy method. As illustrated in Fig. 4, the indentation stage of the load–displacement curve can be divided into the loading stage, the load–holding stage, and the unloading stage. U_e is the elastic energy corresponding to unloading rebound, U_p is the pure plastic energy, and h_f is the residual depth after unloading.

The hardness H of the tested material is calculated as follows[4, 24–25]:

$$H = \frac{F_{max}}{A_c} \tag{1}$$

where F_{max} is the maximum load of nanoindentation test, A_c is the projection area of the contact surfaces between the indenter and the material, and it is a function of the contact depth h_c.

$$A_c = 24.56 h_c^2 \tag{2}$$

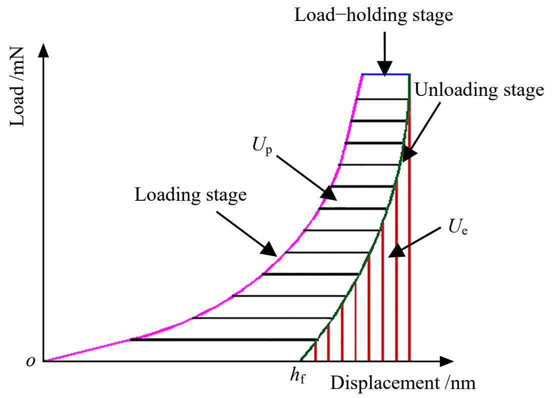


Fig. 4 Typical load−displacement curve

$$h_c = h_{max} - \varepsilon \frac{F_{max}}{S} \quad (3)$$

The reduced indentation modulus E_{re} of the tested material is

$$E_{re} = \frac{\sqrt{\pi} S}{2\beta \sqrt{A_c}} \quad (4)$$

The elastic modulus E_{IT} of the tested specimen is

$$E_{IT} = \frac{1-\nu^2}{\frac{1}{E_{re}} - \frac{1-\nu_i^2}{E_i}} \quad (5)$$

where h_{max} is the maximum indentation depth; ε is a constant related to the indenter shape, and $\varepsilon = 0.75$ for Berkovich indenter; S is the contact stiffness, which can be calculated with the load−displacement curve, and the points within the range from the initial unloading point to the upper 25%−50% of the unloading curve are selected as the calculation basis[26]; ν is the Poisson's ratio of sample; E_i is the elastic modulus of the diamond indenter, and E_i = 1 140 GPa; and ν_i is the Poisson's ratio of the diamond indenter, and $\nu_i = 0.07$.

Through the above method, only the elastic modulus and hardness at the maximum indentation depth can be acquired. For the viscoelastic-plastic materials with negative slopes of the unloading curve, the modulus and hardness of the material cannot be calculated. To address this problem, Pethica et al.[8] proposed a measurement method of continuously calculating the contact stiffness during the loading process. The continuous measurement of contact stiffness leads to the continuous measurement of hardness and elastic modulus with the change of indentation depth, and this method is called continuous stiffness measurement (CSM) method[6]. Li et al.[25] gave a detailed description of the CSM principle. In this experiment, the CSM method was employed for indentation tests.

3 Experimental results

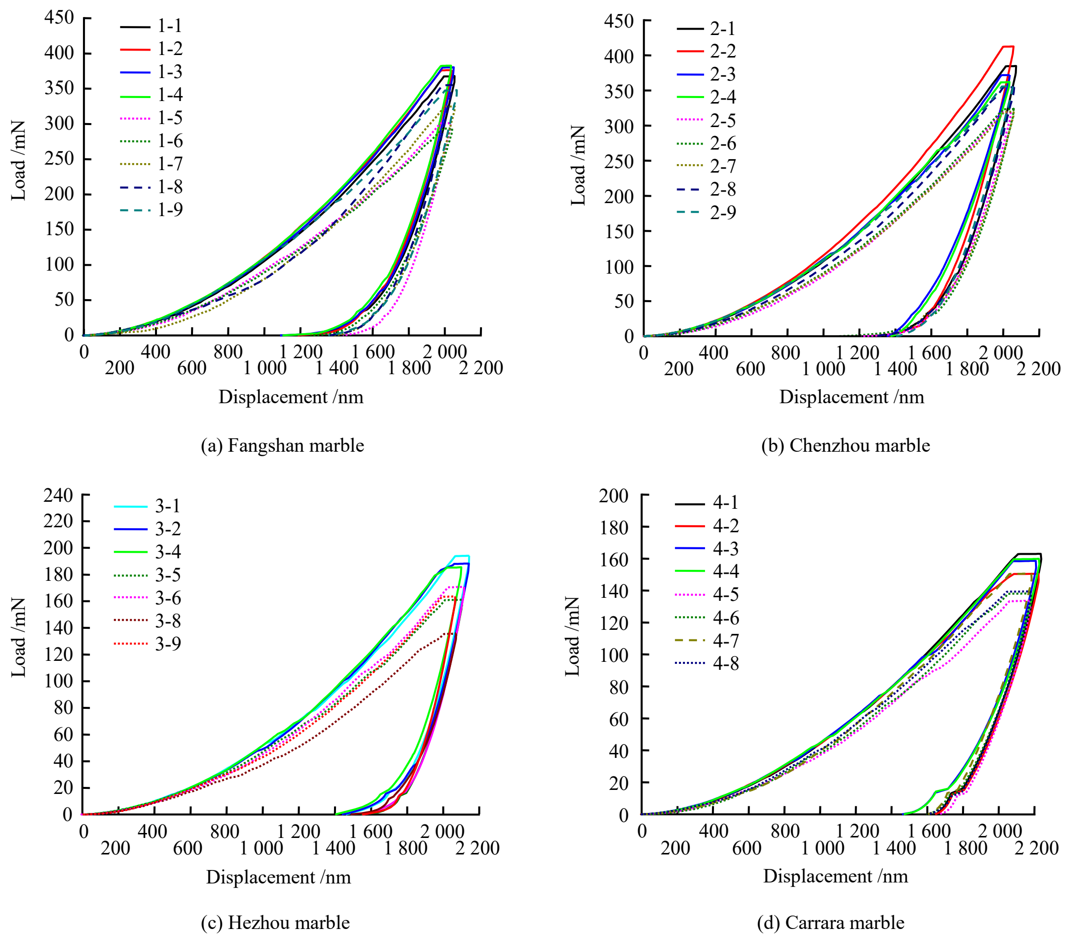
3.1 Load−displacement curve

In Fig. 5, the first number of the curve code represents the rock type (1 for Fangshan marble, 2 for Chenzhou marble, 3 for Hezhou marble, and 4 for Carrara marble), and the second one refers to the indentation point number (indentation points numbered 1−4 are points inside grains, and indentation points numbered 5−9 are points at grain boundaries). For example, the curve code "1-2" indicates the second indentation point for Fangshan marble, and the indentation point is inside the grain. Due to certain reasons about the device, the tests on specimen No. 3 failed at part of indentation points.

According to Fig. 5, the maximum indentation loads for Fangshan and Chenzhou marble reach about 400 mN, while those for Hezhou and Cararra marble are only about 200 mN, indicating that dolomite's strength is stronger than calcite's. Throughout the whole indentation process, the curve for dolomite is much smoother compared to that for calcite, and more concave and convex points are identified in the curve for calcite. At the load-holding stage, the creep of calcite is slightly larger than that of dolomite, demonstrating that the crystal structure surface in calcite is easier to slip under loading than that in dolomite, and the calcite has stronger plasticity. After unloading, the rebound of calcite is less than 1/3 of the overall deformation, and that of dolomite is slightly more than 1/3, showing that calcite is more prone to plastic deformation. Compared to the rebound of sapphire and mica of about 1/2 and feldspar and quartz of nearly 2/3[27−28], the rebound of calcite and dolomite is quite low, indicating stronger plasticity of the two minerals.

3.2 Micro parameter curves

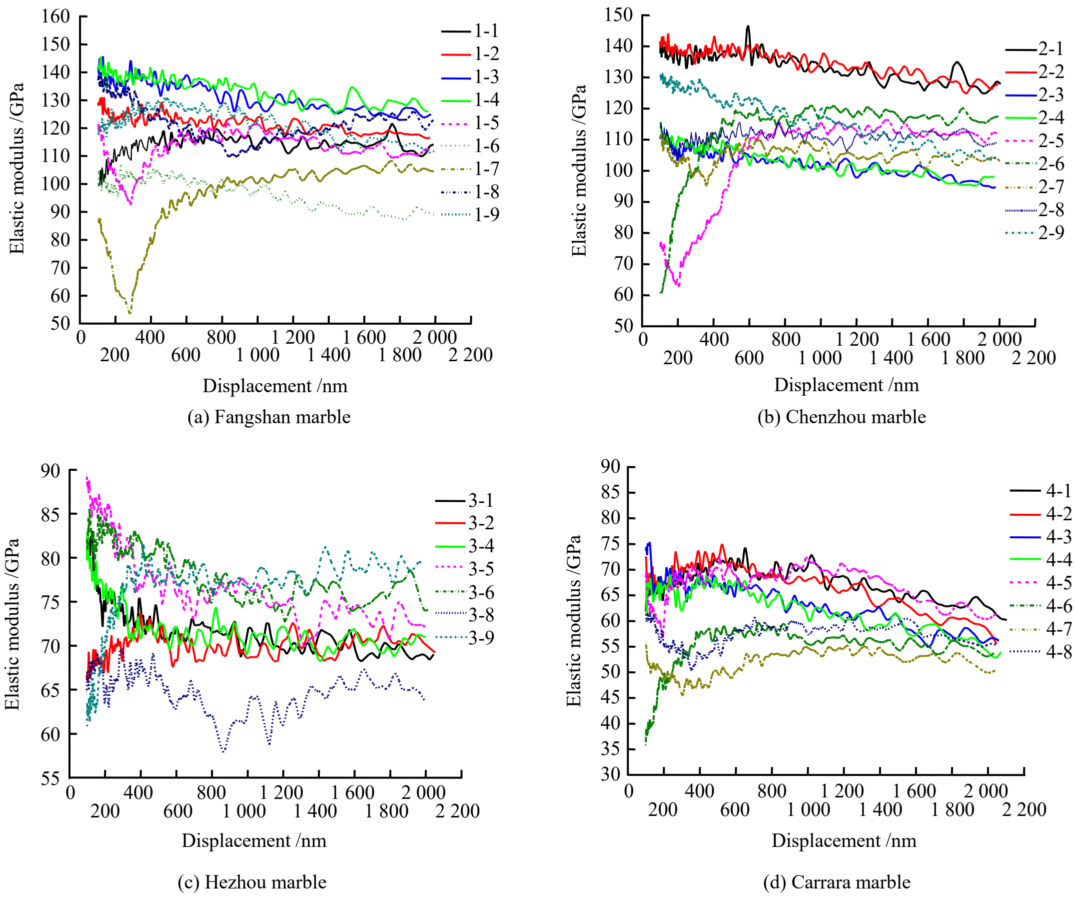
The data in Fig. 5 were processed to draw the elastic modulus−displacement curves and the hardness−displacement curves, as shown in Figs. 6 and 7, and three laws can be concluded from those curves. Firstly, similar change characteristics of micro elastic modulus and hardness are witnessed, and both micro elastic modulus and micro hardness slowly decrease with the increase of indentation depth, denoting that the micro elastic modulus and hardness of the material have a close correlation with each other. Secondly, no matter the elastic modulus curves or the hardness curves, the curves for the boundary points are located below those for the interior points, suggesting that the deformation and strength properties at the grain boundary are weaker than those inside the grain. Finally,



(a) Fangshan marble (b) Chenzhou marble

(c) Hezhou marble (d) Carrara marble

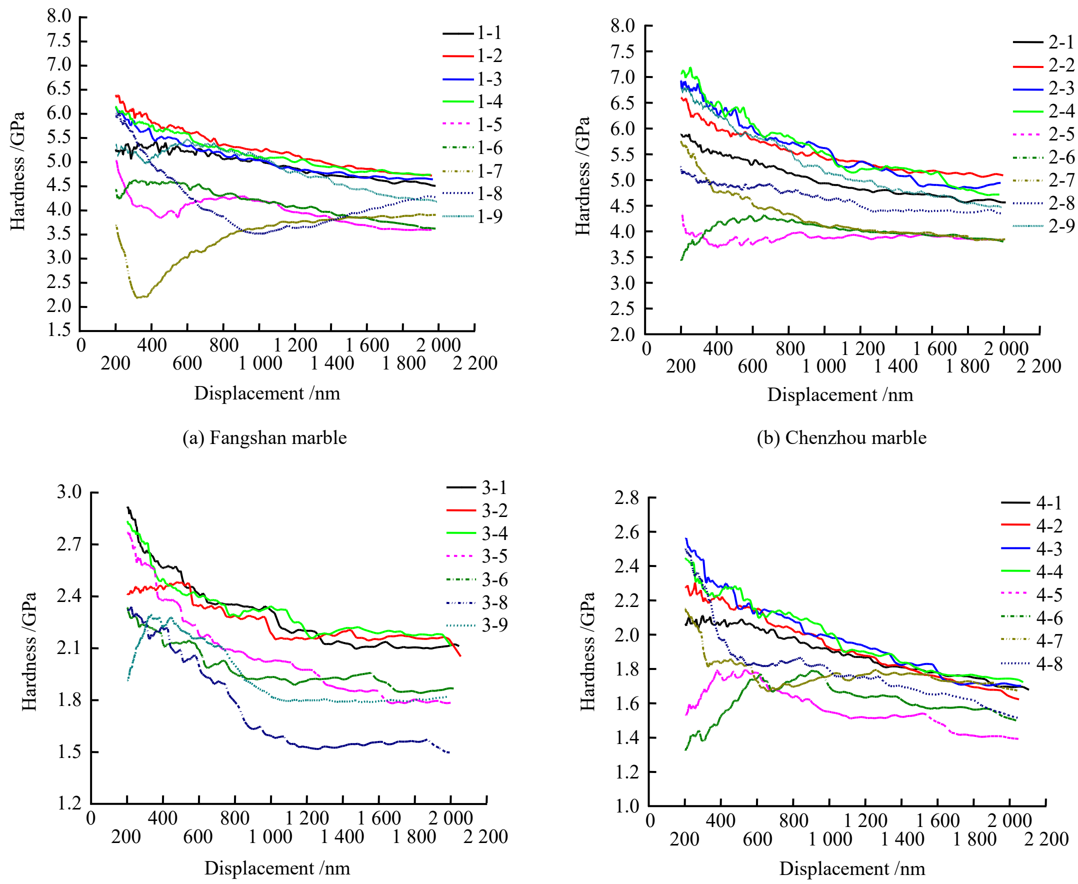
Fig. 5 Load−displacement curves



(a) Fangshan marble (b) Chenzhou marble

(c) Hezhou marble (d) Carrara marble

Fig. 6 Elastic modulus−displacement curves



(a) Fangshan marble (b) Chenzhou marble

(c) Hezhou marble (d) Carrara marble

Fig. 7 Hardness−displacement curves

the curves for the interior points are intensively distributed, while the curves for the boundary points are relatively dispersed with a slightly larger distribution range, which is more clearly shown in the hardness curves. As a result, the data at the boundary points are more discrete than those at the interior points, and the latter is more stable. The latter two laws are consistent with the structural properties of the crystalline rock mass, thus proving the validity and reliability of the indentation test results.

In the nanoindentation tests, large fluctuations of the testing data are observed across the specimen surface (around 300 nm), which may be caused by the large roughness of the specimen surface (poorly polishing) and defects on the specimen surface. For the calcite and dolomite in marble, the elastic modulus and hardness obtained by CSM start to be smoother at a depth of about 800 nm, so the recommended nanoindentation depth where elastic modulus and hardness are determined are beyond 800 nm. As the indentation depth increases, the developed cracks during the loading process result in a decrease in the elastic modulus and hardness[21]. Therefore, the greater the indentation depth, the larger the difference between the elastic modulus as well as hardness and the corresponding real values. So, an indentation depth of 800−900 nm was proposed for the experiment.

3.3 Data analysis

3.3.1 Micro elastic modulus and hardness

The data of elastic modulus and hardness corresponding to the displacement of 800−900 nm in Figs. 6 and 7 can be organized to obtain the results listed in Tables 1 and 2. In Table 1, the average micro elastic modulus of dolomite (Fangshan and Chenzhou marble) is 122.5 GPa, and that of calcite (Hezhou and Carrara marble) is 70.3 GPa, which is in good agreement with the existing results of 115 GPa and 74.5 GPa[29−31], further proving the rationality and reliability of the experimental results.

Table 1 Micro elastic modulus (800−900 nm)

Rock	Interior point /GPa		Boundary point /GPa		Reduction ratio /%
	Average	Standard deviation	Average	Standard deviation	
Fangshan marble	126.0	8.58	111.1	11.84	11.8
Chenzhou marble	118.9	19.05	112.9	3.90	5.0
Hezhou marble	71.1	1.47	72.6	8.52	−2.1
Carrara marble	69.4	2.22	57.3	8.70	17.4

Table 2 Micro hardness (800−900 nm)

Rock	Interior point /GPa		Boundary point /GPa		Reduction ratio /%
	Average	Standard deviation	Average	Standard deviation	
Fangshan marble	5.2	0.11	4.2	0.66	19.2
Chenzhou marble	5.5	0.30	4.5	0.56	18.2
Hezhou marble	2.3	0.03	1.9	0.15	17.4
Carrara marble	2.2	0.09	1.8	0.14	18.2

The conclusion in section 3.2 that the data dispersion at the boundary points is larger than that at the interior points is further quantified here. As displayed in Tables 1 and 2, the standard deviations of micro elastic modulus and hardness at the interior points are smaller than those at the boundary points, except for the standard deviation of elastic modulus of Chenzhou marble, which is a more intuitive indication that the data at the boundary points have a larger dispersion than those at the interior points.

In terms of data values (reduction ratio), the difference between the elastic modulus at the boundary point and that at the interior point is small and unstable, while the difference between the hardness at the boundary point and that at the interior point is large and stable, with a difference of about 18%. As a consequence, the hardness better reflects the defect effect of the boundary, which is similar to the results obtained by Brooks et al.[32].

3.3.2 Micro fracture toughness

The fracture toughness is the most important parameter to characterize the fracture resistance of a material. Existing studies are focused on the fracture toughness at the macro scale[33−34], and there are fewer studies on the micro fracture toughness of a mineral component. Therefore, the micro fracture toughness of minerals is calculated to enrich the corresponding micro data.

In nanoindentation tests, the fracture toughness can be determined by crack length-based method and energy analysis method[35]. Since crack length is difficult to measure, the energy analysis method was adopted to calculate the fracture toughness of the tested material. The total energy input during the test is U_t, the fracture energy is U_c, the elastic energy during unloading rebound is U_e, and the pure plastic energy is U_p, which consists of the fracture energy U_c and the plastic energy U_{pp}[19]. The relationships among these energies are as follows[4, 19]:

$$U_t = U_e + U_p = U_e + U_{pp} + U_c \tag{6}$$

$$\frac{U_{pp}}{U_t} = 1 - \left[\frac{1 - 3\left(\frac{h_f}{h_{max}}\right)^2 + 2\left(\frac{h_f}{h_{max}}\right)^3}{1 - \left(\frac{h_f}{h_{max}}\right)^2} \right] \tag{7}$$

The critical energy release rate for crack generation is

$$G_c = \frac{U_c}{A_{max}} \tag{8}$$

where A_{max} is the maximum projection area of the contact surface.

For the Berkovich indenter:

$$A_{max} = 24.56 h_{max}^2 \tag{9}$$

Then the fracture toughness of the tested material is

$$K_{IC} = \sqrt{G_c E_{re}} \tag{10}$$

The calculation results obtained using the above formula are given in Table 3. The difference in the calculated fracture toughness of the same mineral is large, with a difference of about 1 MPa·m$^{0.5}$ in various rock specimens. Except for Chenzhou marble specimen, the testing data at the interior points are larger than those at the boundary points, which reflects the defect effect of the boundary to a certain extent.

Table 3 Micro fracture toughness (MPa・m$^{0.5}$)

Rock	Fangshan marble	Chenzhou marble	Hezhou marble	Carrara marble
Average for interior points	9.58	6.85	4.41	2.97
Average for boundary points	6.87	7.58	3.66	2.54
Whole average	8.07	7.25	3.98	2.76

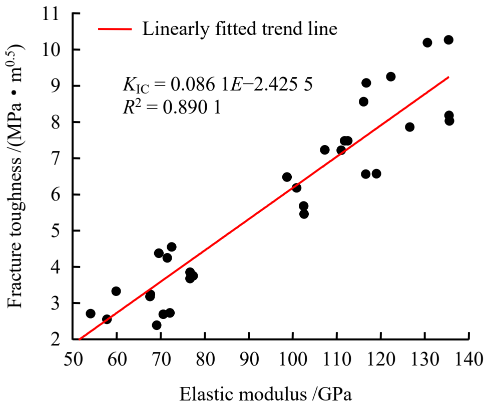
3.3.3 Relationship between mechanical parameters at nanoscale

The micros elastic modulus and hardness are usually easy to obtain, while the micro fracture toughness is difficult to measure[18]. Therefore, the relationship between fracture toughness and micro elastic modulus and hardness is explored, so that the fracture toughness can be predicted with the elastic modulus and hardness. The micro fracture toughness is shown in Table 3. Through linear regression analysis (Fig. 8), both the elastic modulus and hardness obtained from nanoindentation tests are found to have a good positive correlation with fracture toughness, with the correlation coefficients of 0.890 1 and 0.739 6, and the fitted relationship equations are

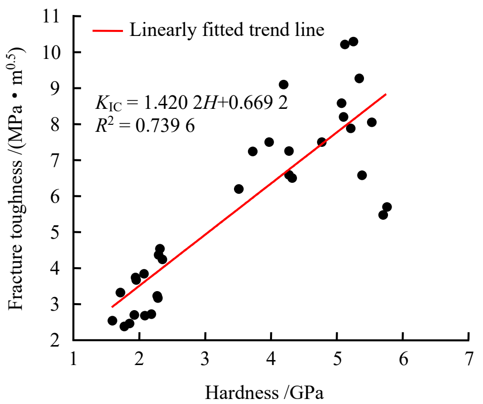
$$K_{IC} = 0.086\,1E - 2.425\,5 \tag{11}$$

$$K_{IC} = 1.420\,2H + 0.669\,2 \tag{12}$$

As the Young’s modulus increases, the ultimate fracture strength also increases[22], thus enhancing the fracture resistance of the material. Therefore, the elastic modulus is highly related with the fracture toughness.



(a) Relationship between fracture toughness and elastic modulus



(b) Relationship between fracture toughness and hardness

Fig. 8 Relationships between fracture toughness and elastic modulus, hardness

4 Correlation of macro properties with nanoindentation data

The mechanical parameters of individual mineral components in marble obtained by nanoindentation tests are not representative of the mechanical properties at the macro scale, therefore the upgrade of micro mechanical parameters to those at the macro scale was investigated[15, 22, 28, 36]. The elastic modulus and Poisson's ratio at nanoscale were transformed to macro mechanical parameters by Mori-Tanaka model. Macro mechanical tests were also conducted on four types of marble to obtain the macro elastic modulus, Poisson's ratio, and various types of strength indexes (Table 4). The homogenized mechanical parameters were compared with the macro mechanical parameters.

Table 4 Macro physical properties of four types of marble

Rock	Uniaxial compressive strength	Tensile strength /MPa	Macro elastic modulus	Macro fracture toughness /(MPa • m$^{0.5}$)	Macro Poisson's ratio
Fangshan marble	127.3	3.9	56.9	0.65	0.32
Chenzhou marble	151.7	6.7	67.2	1.14	0.27
Hezhou marble	49.3	3.5	36.0	0.60	0.21
Carrara marble	88.3	5.0	49.0	0.65	0.29

4.1 Elastic modulus and Poisson's ratio

The Mori-Tanaka method was used for homogenization of multiphase elastic materials. The marble was considered as a two-phase or three-phase composite, and its micro mechanical parameter were upgraded using the Mori-Tanaka method. The bulk modulus and shear modulus of each phase of the medium were calculated as follows[36–37]:

$$K_{\mathrm{M}}=\frac{\sum f_r\dfrac{k_r}{3k_r+4\mu_{\mathrm{low}}}}{\sum\dfrac{f_r}{3k_r+4\mu_{\mathrm{low}}}} \tag{13}$$

$$G_{\mathrm{M}}=\frac{\sum\dfrac{f_r\mu_r}{\mu_{\mathrm{low}}(9k_{\mathrm{low}}+8\mu_{\mathrm{low}})+6\mu_r(k_{\mathrm{low}}+2\mu_{\mathrm{low}})}}{\sum\dfrac{f_r}{\mu_{\mathrm{low}}(9k_{\mathrm{low}}+8\mu_{\mathrm{low}})+6\mu_r(k_{\mathrm{low}}+2\mu_{\mathrm{low}})}} \tag{14}$$

$$k_r=\frac{E_r}{3(1-2v_r)} \tag{15}$$

$$\mu_r=\frac{E_r}{2(1+v_r)} \tag{16}$$

where the subscripts r = 0, 1 for two-phase composites; r = 0, 1, 2 for three-phase composites; K_{M} is the shear modulus; G_{M} is the bulk modulus; E_r is the elastic modulus of mineral grain; f_r is the volume fraction of mineral; k_r is the bulk modulus of mineral; μ_r is the shear modulus of mineral grain; k_{low} is the equivalent bulk modulus of low-strength mineral; μ_{low} is the equivalent shear modulus of low-strength mineral; and v_r is the Poisson's ratio of mineral grain.

The mechanical properties of the pore structure and matrix minerals in the marble were homogenized into the mechanical properties of the matrix solid phase with pores[10]:

$$k_{\mathrm{low}}=\frac{4(1-\phi)k_{\mathrm{s}}\mu_{\mathrm{s}}}{4\mu_{\mathrm{s}}+3\phi k_{\mathrm{s}}} \tag{17}$$

$$\mu_{\mathrm{low}}=\frac{(1-\phi)\mu_{\mathrm{s}}}{1+6\phi\dfrac{k_{\mathrm{s}}+2\mu_{\mathrm{s}}}{9k_{\mathrm{s}}+8\mu_{\mathrm{s}}}} \tag{18}$$

where ϕ is the rock porosity; k_{s} is the bulk modulus of low-strength mineral; μ_{s} is the shear modulus of low-strength mineral.

After obtaining the equivalent mechanical parameters of each phase medium, the homogenized elastic modulus E_{hom} and Poisson's ratio v_{hom} of rock are calculated as:

$$E_{\mathrm{hom}}=\frac{9K_{\mathrm{M}}G_{\mathrm{M}}}{3K_{\mathrm{M}}+G_{\mathrm{M}}} \tag{19}$$

$$v_{\mathrm{hom}}=\frac{3K_{\mathrm{M}}-2G_{\mathrm{M}}}{6K_{\mathrm{M}}+2G_{\mathrm{M}}} \tag{20}$$

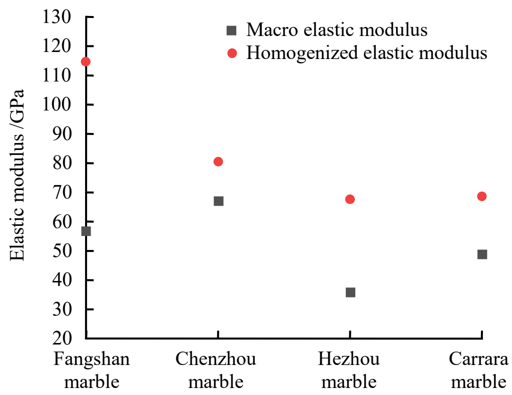
In this section, the indentation data inside the grain were collected for calculations. According to the illustration in Section 2.1, Fangshan marble is considered as a two-phase composite with calcite as a matrix mineral (content of 15%) and dolomite as an embedded mineral (content of 85%). Chenzhou marble is considered as a three-phase composite with mica as a matrix mineral (content of 5%) and dolomite (content of 80%) plus tremolite (content of 15%) as embedded minerals. Hezhou marble and Carrara marble are considered as two-phase composites, with mica as matrix mineral (contents of 5% and 1%) and calcite as embedded mineral (contents of 95% and 99%). The pores are assumed to be concentrated in the matrix minerals. According to the existing results[38−40], the elastic modulus of mica is 30.2 GPa, and the Poisson's ratio is 0.3. The elastic modulus of tremolite is 24.2 GPa, and the Poisson's ratio is 0.18. The Poisson's ratios of calcite and dolomite are 0.23 and 0.21, and the porosity is set as 0.33%[32].

For Fangshan marble specimen, $K_M = 66.74$, and $G_M = 47.34$. Then $E_{hom} = 114.9$ GPa and $\nu_{hom} = 0.21$ were obtained by Eqs. (19) and (20). (When the content of matrix mineral is changed by 5%, the homogenized elastic modulus is changed by about 2.5 GPa, and the homogenized Poisson's ratio is almost unchanged. Therefore, a small amount of change in mineral content has nearly no effect on the calculation results). The homogenized results of the remaining specimens were also obtained by the Mori-Tanaka method (Table 5), and they were compared with the macro testing results, as presented in Fig. 9. The homogenized elastic modulus of Fangshan marble and Hezhou marble deviates more from the macro testing results, while the deviations for Chenzhou marble and Carrara marble are smaller. The homogenized Poisson's ratio of the four types of specimens are roughly in line with the macro results.

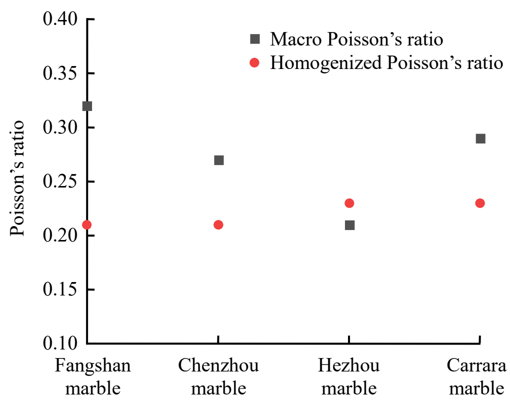
Table 5 Homogenized elastic modulus and Poisson's ratio

Rock	Homogenized elastic modulus /GPa	Homogenized Poisson's ratio
Fangshan marble	114.9	0.21
Chenzhou marble	80.6	0.21
Hezhou marble	67.7	0.23
Carrara marble	68.7	0.23

Because the Mori-Tanaka method is based on the isotropic hypothesis, the anisotropic characteristics cannot be reflected, resulting in the fact that the homogenized elastic modulus and Poisson's ratio of rock specimens obtained by this method deviates from the macro testing results. However, upgrading the nanoscale parameters to macro ones by the homogenization method has potential applications in engineering. For example, the homogenization analysis on small rock specimens can lead to the mechanical properties of surrounding rock, which solves the problem of difficulties in coring and high requirements for rock specimens.



(a) Elastic modulus



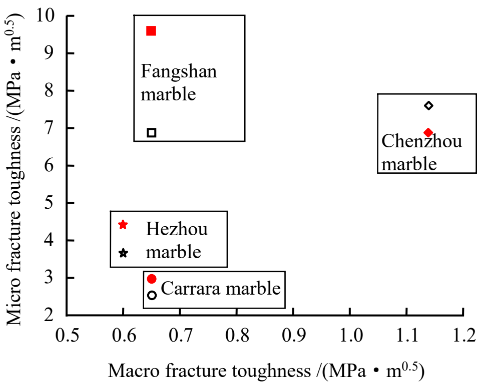
(b) Poisson's ratio

Fig. 9 Comparison of elastic modulus and Poisson's ratio obtained by homogenization method and macro test

4.2 Relationship between micro and macro fracture toughness

The macro fracture toughness was obtained from the three-point bending test (Table 4), and the micro fracture toughness was compared with the macro fracture toughness, as shown in Fig. 10.

The correlation between micro and macro fracture toughness is poor for the specimens with the same mineral compositions. The micro fracture toughness of calcite and Carrara marble is smaller than that of Hezhou marble, while the macro fracture toughness is larger than that of Hezhou marble. As regards the specimens with different mineral compositions, the macro fracture toughness of Fangshan marble and Carrara marble is the same, but the micro fracture toughness of Fangshan marble is much larger. The experimental results show that the correlation between micro and macro fracture toughness is very poor. The idea of predicting the macro fracture toughness through the micro fracture toughness calculated by nanoindentation method is not feasible at present, and there is still a lot of work needed to be done to realize this idea.



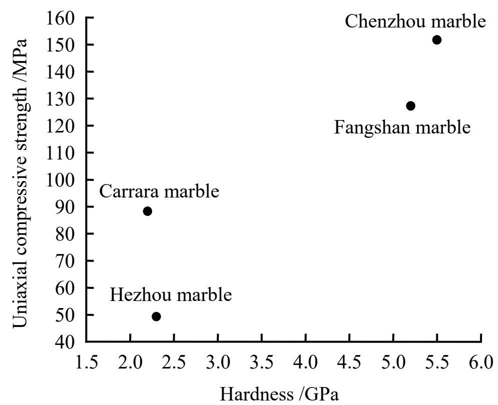
Note: solid points are interior points, and hollow points are boundary points.

Fig. 10 Relationship between micro and macro fracture toughness

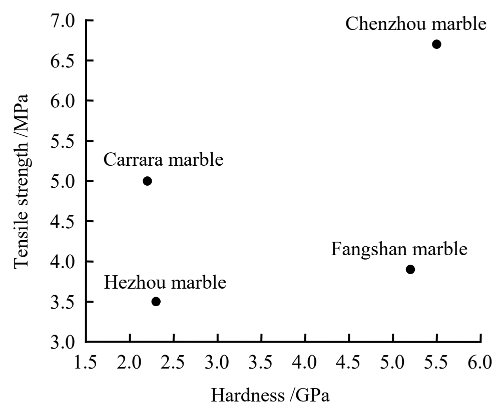
4.3 Compressive and tensile strengths

The strength indexes were obtained from the uniaxial compression tests and the Brazilian tests, and these indexes were compared with the micro properties, as shown in Fig. 11.

In Fig. 11(a), the hardness of Chenzhou marble and Fangshan marble is significantly greater than that of Carrara marble and Hezhou marble, so their corresponding macro compressive strengths are also significantly greater. However, when the relationships for the specimens with the same mineral composition are individually investigated, there are different results. For example, though both Hezhou marble and Carrara marble are calcite marble, the hardness of Hezhou marble is slightly higher than that of Carrara marble, while the uniaxial compressive strength is about 0.6 times that of Carrara marble. The reason for the existence of this seemingly paradoxical phenomenon is explained here. For the rocks with significantly different mineral compositions, the mineral plays a decisive role in the compressive strength though there are other affecting factors, and thus there is an overall correlation between mineral composition——micro hardness and the compressive strength. For the rocks composed of (approximately) the same mineral components, their mineral compositions and hardness are similar, and their strength differences are controlled by other factors (such as differences in grain size and cementation degree). When there is a big difference in these controlling factors, there will be the difference in compressive strength, causing the similar hardness but quietly different compressive strength of two kinds of rocks. As a result, the uniaxial compressive strength of rocks generally increases as the hardness increases, and this trend only reflects the influence of mineral composition. Because the compressive strength of rocks is also affected by other factors, so it is possible that the difference in hardness is small but that in the strength is significant.



(a) Relationship between hardness and uniaxial compressive strength



(b) Relationship between hardness and uniaxial tensile strength

Fig. 11 Relationships between hardness and uniaxial compressive strength, tensile strength

In Fig. 11(b), the mineral composition and hardnesses of Fangshan and Chenzhou marble are similar, but the tensile strength of Chenzhou marble is about 1.7 times that of Fangshan marble. The mineral compositions of Hezhou and Carrara marble are the same, but there is an inverse proportional relationship between tensile strength and hardness. For the marbles with different mineral compositions, the hardness of Fangshan marble is about 2.3 times that of Hezhou marble, but the tensile strength of Fangshan marble is only 1.1 times that of Hezhou marble. Although the tensile strength is affected by the mineral hardness, the influence of hardness is relatively smaller compared to other factors (internal defects and cementation degree). Therefore, there is no clear relationship between tensile strength and hardness of rocks, and the tensile strength cannot be predicted by hardness alone.

4.4 Discussion

The correlations between the nanoindentation results and various macro properties are complicated, which is further discussed in the following aspects.

Firstly, as far as the rock composition and texture are concerned, the indentation data can reflect the composition, but hardly reflect the structure. A large number of studies have shown that the mineral components that make up the rock has an important effect on the rock strength. For example, Cui et al.[41] and Meng et al.[42] concluded that the rock strength increases with the increase of quartz content in their experiment. Similarly, Wan[43] conducted the correlation analysis on the compressive strength of clay rock, and concluded that the clay mineral content of a rock is negatively correlated with both the uniaxial and triaxial compressive strengths, whereas the quartz, high-strength silicate, and calcium mineral content are positively correlated with the uniaxial and triaxial compressive strengths. By the nanoindentation tests, the elastic modulus and hardness of these different minerals can be determined, then the influence of the mineral composition (type) on the physical and mechanical properties of the rock can be fully investigated. However, the compressive strength of rock is also affected by structure factors (grain size and shape). For example, Liu et al.[44] and Singh[45] examined various kinds of sandstone with different grain sizes, and found that the uniaxial compressive strength of sandstone increases with decreasing grain size. Howarth et al.[46] found that the higher the rock structure coefficient, the higher the uniaxial compressive strength, indicating that the rock with rough grain boundaries has higher compressive strength than that with flat grain boundaries. The effects of these factors cannot be reflected by the micro elastic modulus and hardness, regardless of using the random indentation method or the indentation matrix method. Therefore, the micro hardness may show a poor correlation with the strength when these factors have a significant effect on the rock strength. For example, the indentation testing results of Hezhou marble and Carrara marble are close, but the strength of Carrara marble is about 1.8 times of that of Hezhou marble, which is probably due to the fact the average grain size in Carrara marble is only about 1/6 of that in Hezhou marble.

Secondly, as far as the rock structure is concerned, the indentation testing results can only reflect the properties at the indentation points, and even the indentation matrix results, homogenized statistical results from a large amount of data, mainly reflect the weighted average properties of the material’s constituents (including homogenized pores). The possible anisotropy and heterogeneity of the rock thus cannot be well reflected. In fact, the anisotropy and heterogeneity have a significant impact on rock properties, which can also lead to that the nanoindentation testing results are uncorrelated with the macro properties. For example, the strength and deformation properties of shale in the direction perpendicular to the bedding plane are significantly stronger than those in the direction along the bedding plane[47], but the average indentation results in the same plane and in different directions are the same. Therefore, future development of modeling methods that take into account the spatial distribution of indentation data is urgently needed, then the effects of anisotropy and heterogeneity can be presented by mapping the indentation testing results to their spatial locations.

Finally, there are many kinds of physical and mechanical parameters of rocks, and different parameters are influenced by different factors with varying influence degrees. As a result, the correlations between the nanoindentation testing results and different macro parameters should be different. For example, the macro elastic modulus and Poisson’s ratio are the parameters describing overall elastic deformation of rock, and they are greatly affected by mineral composition and rock porosity, while they are not sensitive to micro crack characteristics and macro rupture behavior. On the contrary, the indicators including macro tensile strength and fracture toughness are closely related to the micro crack characteristics and macro rupture behavior, and the factors such as cementation strength and grain boundary defects may have the same, or even a higher, influence weight compared to the mineral composition. Therefore, it is difficult to reliably predict such indicators by only relying on nanoindentation results that reflects the mineral properties.

Most of the current studies on nanoindentation tests are still limited to the elastic modulus and hardness at the nanoscale, and few studies have been conducted to upgrade the elastic modulus at the nanoscale to the macro deformation property through the homogenization model. However, some of the results are in good agreement with the actual ones[20, 29, 48–49], while some others are quite different from the actual ones[10, 15, 50]. Charlton et al.[14]

and Shi et al.[29] believed that the larger the scale of the macroscopic experiment, the more microcracks and micropores the specimen contains, resulting in smaller mechanical parameters obtained from the macro experiment. The Mori-Tanaka method was proposed based on the isotropic hypothesis, which cannot reflect the anisotropic characteristics, so the homogenization results are often in error.

There are few studies comparing nanoindentation results with macro mechanical strength properties. Ganneau et al.[51] conducted series of studies about glass materials, and proposed the method of predicting the cohesion and friction angle of the material using double indentation method. They obtained a positive correlation between the hardness ratio and the friction angle, but they ignored the roughness of the testing surface. More importantly, glass is a typical kind of homogeneous amorphous material at both macro and micro scales, and the structural and tectonic problems discussed earlier are naturally absent. Xu et al.[36] used the same method to calculate the cohesion and friction angle of shale, and the obtained results were in good agreement with the macroscopic testing results. But they assumed that the minerals such as feldspars and quartz do not have plasticity but only elasticity, and only the indentation data of clay minerals were taken into account instead of the indentation data of feldspars and quartz. Similarly, Palkovic et al.[52] used nanoindentation data to model the microstructure of hardened cement pastes, and the simulated uniaxial compressive strengths were in good agreement with the macro testing results. But they also assumed that the random inclusions such as silicate cement grains were only elastic and had no plasticity. The above discussions show that there are still limitations in relating the nanoindentation data to macro properties, and the method of using nanoindentation tests to study the macro rock properties still needs further improvement.

5 Conclusion

The nanoindentation tests on four different types of marble were conducted to obtain the micro mechanical parameters of dolomite and calcite, and related analysis and discussions were also performed. The following conclusions were drawn:

(1) The micro elastic modulus and hardness of dolomite are higher than those of calcite, which may result in the superior strength and deformation properties of dolomite marble over those of calcite marble when other factors (grain size and component) are similar.

(2) For the calcite and dolomite minerals in the marble, the elastic modulus and hardness obtained by CSM start to be stable at an indentation depth of about 800 nm, so the recommended indentation depth for testing the elastic modulus and hardness is beyond 800 nm.

(3) The data dispersion at the boundary points of the grains is larger than that at the interior points of the grains. The difference between the elastic modulus at the boundary point and that at the interior point is not obvious; while the difference of hardness is obvious. The hardness better reflects the defect effects of the boundary.

(4) The correlation between the micro elastic modulus of minerals and the micro fracture toughness is quite high, and the elastic modulus can be used to predict the fracture toughness.

(5) The homogenized elastic moduli of Fangshan marble and Hezhou marble deviate greatly from the macro testing results, while those of Chenzhou marble and Carrara marble deviate less. The homogenized Poisson’s ratios of the four types of specimens are in good agreement with the macro testing results. The method of upgrading nanoscale parameters to macro parameters by homogenization method has potential application values in engineering.

(6) The micro data from the nanoindentation tests of marble correlate slightly better with the macro elastic modulus and Poisson’s ratio, generally with the compressive strength, and not significantly with the tensile strength and fracture toughness. The current nanoindentation testing and analyzing method can only reflect the influence of the minerals (or pores) on the strength and deformation properties of rock components. If the accurate prediction of uniaxial compressive strength, tensile strength, and fracture toughness, rock texture, structure, and other factors affecting the strength must be comprehensively considered.

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