STUDY ON MECHANICAL PROPERTIES OF GRANITE MINERALS BASED ON NANOINDENTATION TEST TECHNOLOGY

by

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In this paper, the nanoscale mechanical properties of quartz, feldspar, and mica in granite are studied by the nanoindentation technique. Firstly, the surface morphology of each mineral composition in granite is obtained by a SEM. Secondly, the elastic modulus and hardness of three minerals in granite are calculated through the load-displacement curve obtained by the nanoindentation test. Based on the energy analysis method, the nanometer fracture toughness of three minerals in granite is obtained. Finally, the correlation between the elastic modulus, the hardness, and the fracture toughness are obtained by experimental data.

Key words: *granite, nanoindentation test, fracture toughness, mechanical property*

Introduction

Granite is widely used in hydropower stations, underground nuclear waste storage and tunnel construction because of its good mechanical properties, such as dense structure, high strength, unweatherable, stable structure and so on. The micro-structure of the granite and its mechanical properties were studied by the nanoindentation technique. It is of great significance to reveal the mechanism of the macroscopic mechanical phenomenon.

In recent years, researchers have studied the mechanical properties of rocks in various ways. Zhu *et al.* [1] described the mechanical properties of natural rocks on the nanoscale by the nanoindentation method. Zhang *et al.* [2] studied the microscopic mechanical properties of granite by nanoindentation test, which provided a reference for determining the macroscopic mechanical properties of rock from a microscopic point of view. Liu *et al.* [3] obtained the quantitative relationship between fracture toughness and elastic modulus of shale based on the energy analysis method. Although the nanoindentation technique can be used to study the mechanical properties of rock, many scholars regard rock as homogeneous isotropic material [1-5]. There are relatively few studies considering the multiphase and anisotropy of rock. It is important to reveal the mechanical properties of granite from the microscopic mechanical properties of minerals.

The indenter and penetration depth are in the nanoscale, which belong to the micro-scale. But the size of the mineral is of the order of a millimeter or even larger, which

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belongs to the macro-scale. Therefore, to study the mechanical properties of the granite under the nanoscale, the mechanical properties of the minerals in the granite must be studied. In this paper, the morphology and distribution characteristics of each mineral in granite at a microscopic scale are identified by SEM. The elastic modulus and hardness of the minerals in the granite are calculated by the small-load press-in test of the material in the elastic crack-free stage. Based on the energy analysis method, the fracture toughness of minerals in granite is obtained by a large load nanoindentation test at the stage of cracking and failure. The correlation among elastic modulus, hardness and fracture toughness of the three minerals is fitted by the least square method.

Nanoindentation equipment and theory

The nanoindentation is a technique that uses the tip of the indenter to contact the surface of the sample under a certain load, to record the depth of the indentation, and the load accordingly. Figure 1 is the Agilent Nano Indenter G200 nanoindentation tester in the USA. The Berkovich indenter is selected for this test.

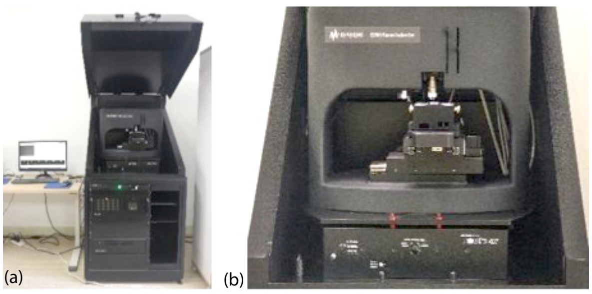

Figure 1. The G200 nanoindentation tester; (a) nanoindentation test system and (b) the G200 nanoindenter

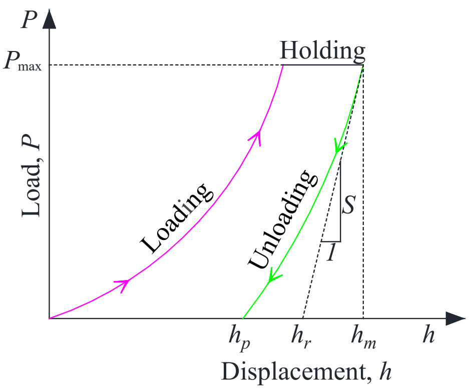

Figure 2. The typical single nanoindentation load-displacement curve

Figure 2 shows the typical three-stage process curve of single indentation loading, holding and unloading [4]. In the loading stage, the load increases with the increase of the indentation depth, which can be regarded as a combination of elastic deformation and plastic deformation. While in the unloading stage, only the elastic deformation can be recovered. It can be used to calculate the mechanical indexes such as elastic modulus and hardness of materials [3, 6].

The two physical quantities obtained by the nanoindentation test are the hardness, H, and the elastic modulus, E. To determine the hardness and modulus of elasticity of the material, the contact stiffness, S, is given:

$$S = \frac{\mathrm{d}P}{\mathrm{d}h}\Big|_{h=h_m} \tag{1}$$

where S is the contact stiffness, P is the load, and h is the displacement.

The reduced modulus is presented [7]:

$$E_r = \frac{\sqrt{\pi}}{2\beta}\frac{S}{\sqrt{A}} \tag{2}$$

where $\beta = 1.034$, h_c is the contact depth (nm), and A is the contact area.

For the Berkovich indenter, the contact depth is expressed as follows [8-10]:

$$h_c = h - \varepsilon\frac{P}{S} \tag{3}$$

where h_c is the contact depth and ε represents a constant associated with the shape of the indenter. The angle between the faceted pebble and the center line is $\psi = 65.3^\circ$, $\varepsilon = 0.75$. Figure 3 shows a schematic diagram of the loading and unloading process. Figure 4 is a schematic diagram of indentation.

The conversion method of fracture toughness based on Energy method. Fracture toughness is a characterization of the ability of materials [7]. Although the indentation test is a non-destructive test and most cases are used to evaluate the elastic properties of the material, the stress intensity factor of the crack tip reaches the critical value of cracking as the load increases [12, 13]. In other words, there are two cases in the process of the indentation. One is that the micro-crack is not initiated at all, and the whole process of the indentation is a pure elastic-plastic system. The other is that as the loading process proceeds, the crack grows and expands.

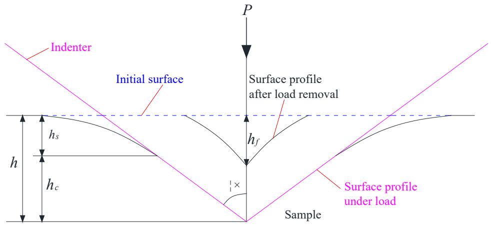

Figure 3. Schematic diagram of the loading and unloading process [11]

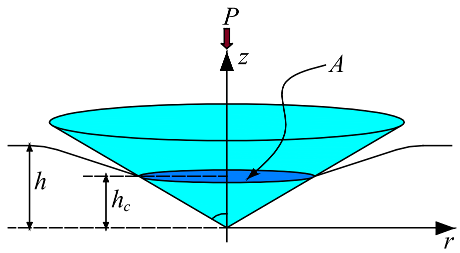

Figure 4. Indentation diagram [6]

The fracture toughness of the main minerals in granite is studied by energy analysis and nanoindentation fracture tests. According to the theory of Yang *et al.* [7], the total energy, U_t, input during the test is the sum of the fracture energy, U_c, the elastic energy, U_e, and the pure plastic energy, U_p, U_e is the elastic energy recovered from the unloading section. There is a relationship between the total energy, U_t, and the pure plastic energy, U_p:

$$\frac{U_p}{U_t} = 1 - \left[1 - 3\left(\frac{h_p}{h_\mathrm{m}}\right)^2 + 2\left(\frac{h_p}{h_\mathrm{m}}\right)^3\right] \frac{1}{1 - \left(\frac{h_p}{h_\mathrm{m}}\right)^2} \tag{4}$$

where U_t is the total energy, U_p – the pure plastic energy, h_m – the total displacement, and h_p – the irrecoverable displacement.

The indentation profile in the loading and unloading process is shown in fig. 5. Finally, the load-displacement curve of this test can be obtained. The hardness and elastic modulus of the sample can be calculated by the characteristic parameters on the curve [6, 8]:

– Firstly, the granite core is machined into the sample of 15 mm × 15 mm × 5 mm. Then it is polished with 100, 600, 1000, 2000, 5000, 7000 mesh of sandpaper in turn. Then ultrasonically clean the sample with absolute ethyl alcohol to make the surface of the sample free of any impurities. Finally, the sample was put into the oven at 50 °C for drying and the sample was dried.
– The treated sample was placed in the nanoindentation tester. The surface of the sample was observed using a 250× optical microscope to identify the minerals.
– The rock samples were analyzed by SEM after the indentation test. The morphology and distribution characteristics of various minerals at the micro-scale and the residual indentation and micro-cracks on different minerals are obtained.

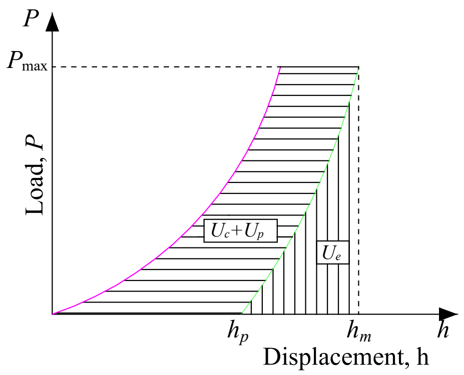

Figure 5. Schematic diagram of total work and unloading work

Results and discussion

Mineral micro-structure: the SEM test is carried out on the granite sample after the nanoindentation test is finished. Figure 6 is the three minerals observed under the SEM.

Analysis of Elastic Modulus and hardness of Minerals: The Poisson's ratios of quartz, feldspar and mica are 0.20, 0.25 and 0.3 [14]. Figure 7 shows the load-displacement curves of quartz, feldspar and mica. It can be seen from fig. 7 that the loading process can be divided into three stages. In the unloading stage, the elastic deformation is mainly restored. The elastic deformation of quartz is the largest, mica is the smallest, and the residual deformation law is the opposite. Figure 8 shows the elastic modulus-displacement curve and the hardness-displacement curve. It can be seen from fig. 8(a) that the elastic modulus of quartz decreases gradually with the increase of indentation depth, and the indentation depth tends to be stable at 250 nm. The hardness of quartz decreases and tends to be stable with the increase of indentation depth. Through statistical calculation, the elastic modulus of quartz is 108.71 ±7.77 GPa, the average value is 108.08 GPa, the standard deviation is 5.40, the hardness is 13.13 ±0.85 GPa, the average value is 13.19 GPa, the standard deviation is 0.60.

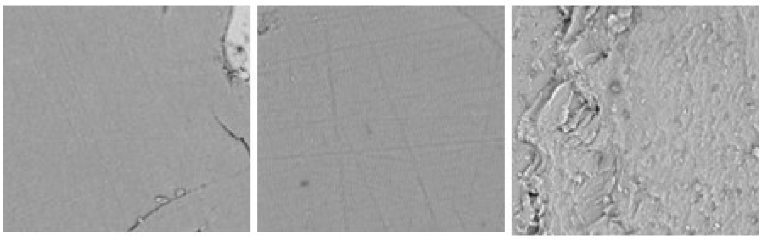

Figure 6. Quartz, feldspar and mica under SEM (magnification 8000×)

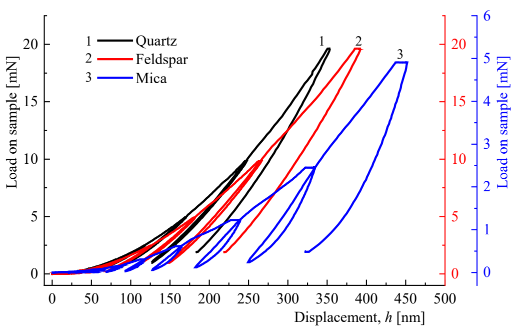

Figure 7. Load-displacement curves of quartz, feldspar, and mica

Figure 9 shows the variation of elastic modulus and hardness of feldspar with displacement. It can be seen from fig. 9 that the elastic modulus and the hardness gradually decrease and stabilize with the increase of the depth of the indentation. The elastic modulus of the feldspar is 87.99 ±9.06 GPa. The average value is 87.51 GPa. The standard deviation is 6.64. The hardness is 9.22 ±0.66 GPa. The mean value is 9.19 GPa and the standard deviation is 0.45.

Figure 10 shows the curve of elastic modulus and hardness of mica with displacement. The elastic modulus and the hardness of the mica are gradually stabilized with the increase of the depth of the indentation. The variation range is not large. When the depth of the indentation is more than 250 nm, the elastic modulus and hardness tend to be stable.

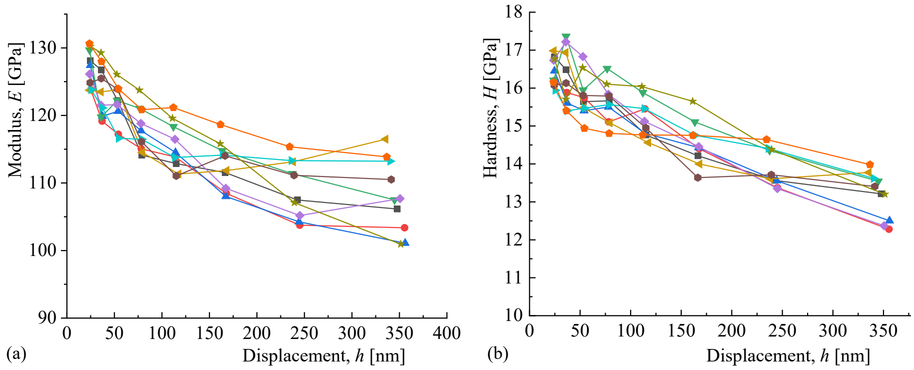

Figure 8. (a) Elastic modulus-displacement curve and (b) hardness-displacement curve

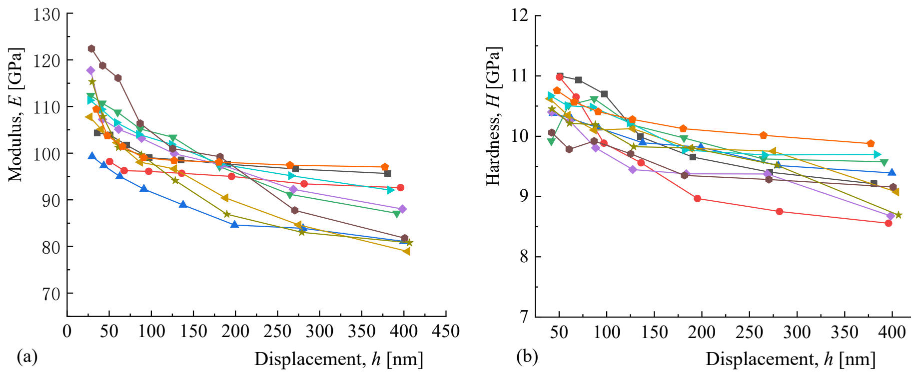

Figure 9. (a) Elastic modulus-displacement curve and (b) hardness-displacement curve

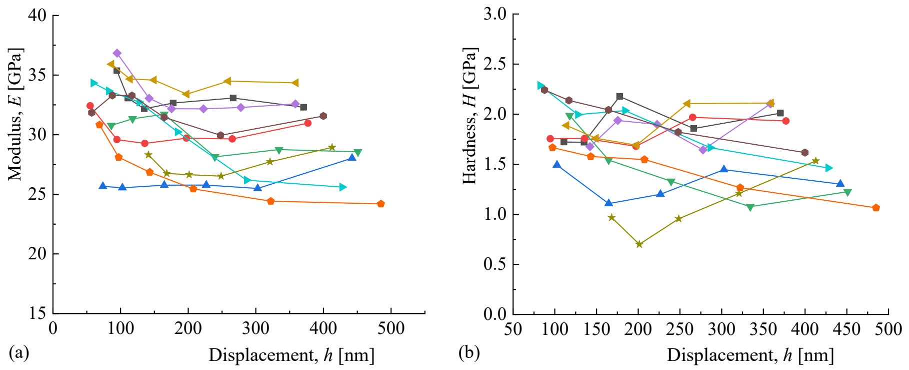

Figure 10. (a) Elastic modulus-displacement curve and (b) hardness-displacement curve

Analysis of fracture toughness: The fracture toughness of the mineral is studied at this stage when the load is greater than the critical load of the material cracking. Figure 11 is a single indentation load-displacement curve for three minerals. Figures 12(a)-12(c) are typical residual indentation images of the three minerals. There is a cracking in the red region in the quartz residual indentation. The cracking of the red area in the residual indentation of feldspar along the ra-

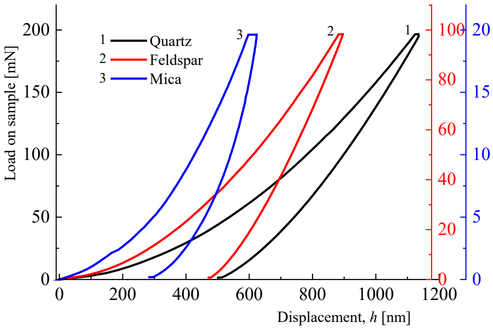

Figure 11. Load-displacement curves of quartz, feldspar, and mica

dial direction of the indentation can be observed from fig. 12(b). In fig. 12(c), as the mica is soft, the residual indentation of the mica has a radial crack and a slight peeling.

The fracture toughness of the three minerals is calculated through an indentation test load-displacement curve, as shown in fig. 13. The average fracture toughness of quartz, feldspar, and mica are 4.30 MPa·m$^{0.5}$, 3.70 MPa·m$^{0.5}$ and 2.12 MPa·m$^{0.5}$. The increase in the modulus of elasticity increases the ultimate breaking strength, thereby improving the resistance to the fracturing of the material.

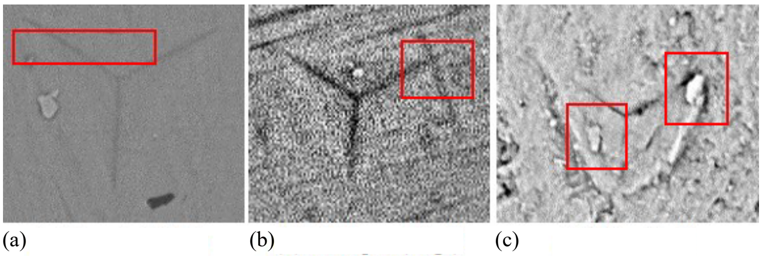

Figure 12. Typical residual indentation of the three minerals; (a) quartz, 20 gf, (b) feldspar, 10 gf, and (c) mica, 2 gf

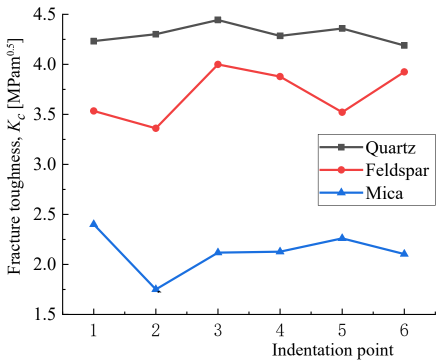

Figure 13. Fracture toughness of three minerals

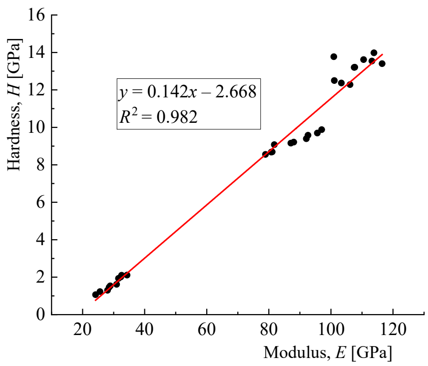

Figure 14. The relationship between *E* and *H*

The relationship between the mechanical properties of the mineral under the nanoscale

The relationships between elastic modulus, hardness and fracture toughness were analyzed. It is found that there is a good linear positive correlation between the elastic modulus and the hardness of the mineral, and the correlation coefficient, R^2, is 0.982, as shown in fig. 14.

Conclusion

At the present work, the elastic modulus and hardness of quartz and feldspar decrease gradually with the increase of indentation depth and tend to be stable, while the elastic modulus of mica has little change with the change of indentation depth. The fracture toughness value of the mineral with a large elastic modulus is relatively large. There is a good linear relationship between elastic modulus and hardness, elastic modulus and fracture toughness, hardness, and fracture toughness at the nanoscale. These results provide a good method for the analysis of rock mechanical properties by the nanoindentation test.

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Nomenclature

A – contact area, [m2]
E_r – reduced modulus, [GPa]
h_c – contact depth, [mm]
h_m – total displacement, [mm]

References

[1] Zhu, W. Z., *et al.*, Nanoindentation Mapping of Mechanical Properties of Cement Paste and Natural Rocks, *Materials Characterization*, 58 (2007), 11, pp. 1189-1198
[2] Zhang, F., *et al.*, Experimental Study on Micromechanical Properties of Granite, *Chinese Journal of Rock Mechanics and Engineering*, *36* (2017), 2, pp. 3864-3872
[3] Liu, K., *et al.*, Applications of NanoIndentation Methods to Estimate Nanoscale Mechanical Properties of Shale Reservoir Rocks, *Journal of Natural Gas Science and Engineering*, *35* (2016), Part A, pp. 1310-1319
[4] Hu, C., Li, Z., A Review on the Mechanical Properties of Cement-Based Materials Measured by Nanoindentations, *Construction and Building Material*, *90* (2015), 8, pp. 80-90
[5] Xue, Y., *et al.*, Productivity Analysis of Fractured Wells in Reservoir of Hydrogen and Carbon based on Dual-Porosity Medium Model, *International Journal of Hydrogen Energy*, 45 (2020), 39, pp. 20240-20249
[6] Hay, J., Introduction to Instrumented Indentation Testing, *Experimental Techniques*, *33* (2009), 6, pp. 66-72
[7] Yang, T. C., *et al.*, Scaling Relationships for Indention Measurements, *Philosophical Magazine A*, *82* (2002), 10, pp. 1821-1829
[8] King, R. B., Elastic Analysis of Some Punch Problems for a Layered Medium, *International Journal of Solids and Structures*, *23* (1987), 12, pp. 1657-1664
[9] Liu, J., *et al.*, Numerical Evaluation on Multiphase Flow and Heat Transfer during Thermal Stimulation Enhanced Shale Gas Recovery, *Applied Thermal Engineering*, *178* (2020), Sept., pp. 115554
[10] Oliver, W. C., Pharr, G. M., An Improved Technique for Determining Hardness and Elastic Modulus using Load and Displacement Sensing Indentation Experiments, *Journal of Materials Research*, *7* (1992), 6, pp. 1564-1583
[11] Oliver, W. C., Pharr, G. M., Measurement of Hardness and Elastic Modulus by Instrumented Indentation: Advances in Understanding and Refinements to Methodology, *Journal of Materials Research*, *19* (2004), 1, pp. 3-20
[12] Xue, Y., *et al.*, Analysis of Deformation, Permeability and Energy Evolution Characteristics of Coal Mass around Borehole after Excavation, *Natural Resources Research*, 29 (2020), 5, pp. 3159-3177
[13] Quinn, G. D., Bradt, R. C., On the Vickers Indentation Fracture Toughness Test, *Journal of the American Ceramic Society*, *90* (2007), 3, pp. 673-680
[14] Cai, M., *Rock Mechanics and Engineering*, Science Publishing House, Beijing, China, 2002

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